*Desalination and Water Treatment*
www.deswater.com
doi: 10.5004/dwt.2020.25600

189 (2020) 98–107
June

# Static mixer continuous chemical coagulation–flocculation for cattle feedlot wastewater treatment

Santiago Nicolás Fleite[a,b], Ana Rosa García[b], Christian Nahuel De los Santos[b], Alicia Fabrizio de Iorio[b], Miryan Cassanello[a,c,*]

[a]*Laboratorio de Análisis de Reactores y Sistemas para la Industria, Departamento de Industrias, FCEyN, Universidad de Buenos Aires, Intendente Güiraldes 2620, Ciudad de Buenos Aires, Argentina, Tel. +54-11-52859029; emails: miryan@di.fcen.uba.ar (M. Cassanello), fleite@di.fcen.uba.ar (S. Nicolas Fleite)*

[b]*Cátedra de Química Inorgánica y Analítica (FAUBA), emails: agarcia@agro.uba.ar (A.R. García), delossantos@agro.uba.ar (C.N. De los Santos), aiorio@agro.uba.ar (A.F. de Iorio)*

[c]*Instituto de Tecnología de Alimentos y Procesos Químicos (UBA–CONICET)*

Received 16 August 2019; Accepted 3 February 2020

**ABSTRACT**

Feedlots generate a large amount of manure-contaminated runoff wastewater that is accumulated in reservoirs and should be treated promptly before disposal. A promising alternative to reduce the organic level while recovering the nutrients in a timely manner is a physicochemical treatment based on coagulation–flocculation. This work aims at optimizing the coagulation–flocculation stage of a feedlot wastewater treatment. First, batch experiments were carried out to define and optimize the dose of coagulants. Then, continuous coagulation was examined inside a horizontal tube, empty and provided with static mixers. Iron chloride, calcium hydroxide and magnesium hydroxide were tested as coagulants. The best strategy found was a combined addition of iron chloride followed by calcium hydroxide. Removal efficiencies of around 98% (phosphorus and organic matter) were achieved with a dose of 10 mM iron (III) plus the calcium hydroxide required for neutralization. The use of a Koflo type static mixer was found to be the best configuration, promoting the formation of flocs with excellent settling properties. Settler areas up to eight times smaller would be required if the flocculation was promoted in the static mixer rather than in a stirred tank. The proposed method would be convenient for both small and large feedlot establishments to self-treat wastewater shortly after a rainfall.

*Keywords:* Static mixer; Coagulation; Settling; Wastewater treatment; Beef cattle feedlot

## 1. Introduction

Livestock practices in humid climates give rise to large volumes of effluents with a very high organic load [1]. The excreta of animals in beef cattle feedlots constitute the main component of the effluents. Land spaces allotted for solid waste storage, lagoons, accumulation channels as well as adjacent agricultural fields with the addition of high doses of manure as fertilizer are prone to be contaminated. The biological degradation of manure releases a large amount of labile organic matter, nutrients (N and P) and salts, minor constituents such as metals (Cu, Zn, and Fe) and organic compounds (antibiotics, antiparasitics, hormones, and other ionophores), as well as pathogens (*Giardia*, *Escherichia coli*). These agents can be mobilized by rain and reach superficial or subsurface water sources, degrading their quality [2–4]. In countries with a long history of intensive livestock production, the collection and treatment of runoff include catchment areas and lagoon systems, as well as their subsequent reuse in agricultural fields [5]. Nevertheless, feedlot wastewater is still an unresolved problem, especially in regions with high rain precipitation levels where quick treatment procedures

* Corresponding author.

are required to cope with the contaminated water rapidly enough to avoid uncontrolled discharges. The irregular nature of the effluent generated by rains hinders the implementation of biological treatment technologies such as biogas production facilities. De Vuyst et al. [6] evidenced that biogas generation in beef cattle feedlots is not economically viable under current market conditions. Moreover, Caruana [7] recently showed that although anaerobic digestion and similar production systems could be applied for very large establishments, such systems are unprofitable for medium or small cattle feedlots due to the investment required for modifications in the production scheme.

An alternative to biological treatment is coagulation–flocculation/precipitation with chemical aids, followed by settling. This process is widely used for sewage treatment [8]. Iron salts or alums are used to remove colloids, nutrients and soluble organic matter from wastewater. The mechanism involves charge neutralization and coprecipitation [5,9] together with certain insoluble salts precipitation (e.g., $FePO_4$, $AlPO_4$). Phosphorus is a pollutant of main concern due to its disruptive effect on water bodies. Its removal and recovery for reuse have been the subject of intense research [10,11]. Sievers et al. [12] used iron chloride ($FeCl_3$) for poultry, cattle and swine wastewaters, finding removal efficiencies over 70%. The same authors used chitosan and some artificial synthetic polymers with similar results. They reported poor removal efficiencies with iron sulfate and magnesium chloride. Sherman et al. [13] used iron chloride ($FeCl_3$) for treating dairy wastewaters, achieving more than 80% removal of the phosphorous inorganic fractions. Laridi et al. [14] explored struvite ($MgNH_4PO_4$) precipitation for phosphorus and nitrogen removal from swine wastewater, using magnesium chloride and magnesium hydroxide as coagulants. They were able to remove 98% of the phosphorus and 17% of the ammonia. Nonetheless, this process needed a high ammonia concentration in the inlet flow, which is not common for most of the livestock wastewaters. Thapa et al. [15] used electro-coagulation with iron, aluminum and a mixed aluminum-iron alloy for treating feedlot wastewater. They reported an almost total phosphorus (TP) removal with varying results for nitrogen (approximately 40% on average) and organic matter (approximately 70% on average). This technique, which replaces the coagulant addition with an electrochemical reaction, is currently being investigated for several different wastewaters [15–18]. It involves managing electrical current within a highly conductive media (due to the high salinity of the wastewaters), and periodic replacement of the electrodes [15].

Flocculation is generally carried out in big tanks, which require large space and energy consumption. Several works have been devoted to optimizing the mixing for improving the flocs characteristics and settling efficiency [19]. The use of static mixers has been proposed for the flocculation stage applied to urban or mine wastewaters [20–22], thus avoiding the use of a stirred tank. Preventing the use of mobile parts is especially important for rural environments, where regular maintenance is difficult. Evidence of a positive effect of static mixers on settling has been reported. Demoz [20] used the settling curve analysis to determine the influence of the flow rate on flocculation promoted in static mixers for mineral wastewaters. Increased initial settling velocities were found, indicating improved settling behavior. The dewatering capability of flocculated wastewaters has been assessed using the capillary suction time (CST) test [23,24].

Even though previous works have demonstrated the ability of static mixers for reducing coagulant consumption for urban and mine wastewaters, the settling curves and nutrient removal efficiencies were usually not reported. Also, to our knowledge, there is no information regarding the use of static mixers for assisting flocculation of beef cattle feedlots wastewaters.

This work aimed to examine the coagulation–flocculation process for cattle feedlot wastewater treatment, focusing on nutrient and organic matter removal and recovery. Hence, the coagulants used were selected to allow for the future use of the sediment as fertilizer, after proper stabilization. Mixing conditions were particularly studied for optimizing the coagulant dose and the flocs settling properties. The suitability of promoting continuous flocculation using a static mixer instead of a mixing tank with mobile parts was assessed.

## 2. Materials and methods

Feedlot effluent was collected 24 h after a rain, from the main runoff collection pond of an establishment located in the Buenos Aires province of Argentina. It was characterized and used for flocculation and settling tests.

Electrical conductivity, pH, ammonia (NH4), total Kjeldahl nitrogen (TKN), total reactive phosphate (TRP), total phosphorus, total iron, chemical oxygen demand (COD), turbidity (NTU), volumetric solid fraction, hardness, and alkalinity were measured following the standard methods [23]. Organic fractions of nitrogen (N-Org) and phosphorus (P-Org) were obtained by difference with totals. Even if some features were not particularly affected by the studied treatment, they have been determined and reported for means of comparison with other wastewaters. Depending on the coagulant employed, hardness and alkalinity can be relevant for the coagulation process [8].

Effluent flocculation was tested in batch and continuous arrangements at a controlled temperature of 20°C. Experiments in the batch were carried out in 2 L flasks using a magnetic stirrer at 80 rpm, as recommended in the literature [19,25–27]. Iron (III) chloride hexahydrate, calcium hydroxide and magnesium hydroxide, which are not phytotoxic, were selected as coagulants considering the possibility of using the sediment as a fertilizer after proper stabilization. The feasibility of producing a solid fertilizer from coagulation treatments of different animal effluents has been already discussed and evidenced [11,13,14]. Flocculation was promoted using solutions of each coagulant at a time or combined, adding iron salt first and then, hydroxide. Examined treatments involved the addition of (i) iron chloride (Fe), (ii) calcium hydroxide (Ca), (iii) magnesium hydroxide (Mg), (iv) iron chloride followed by calcium hydroxide (Fe + Ca) and (v) iron chloride followed by magnesium hydroxide (Fe + Mg). Different concentrations were tested to determine the optimal dose.

For the continuous experiments, preliminary tests were carried out using the calcium carbonate formation as a model for the precipitation–coagulation–flocculation process [28]. Aqueous solutions prepared with anhydrous sodium carbonate and dihydrate calcium chloride were used to induce calcium carbonate precipitation. Iron chloride was used as the coagulant, and cationic polyacrylamide (105 g mol$^{-1}$) was added to the media for simulating the effect of organic matter aggregation (flocculation). This model fluid was used for testing different mixing conditions during the continuous flocculation process assisted with the addition of a coagulant at three flow rates: 2.0, 1.3, and 0.7 L min$^{-1}$. Once the best mixing configuration was determined using the model fluid, continuous flocculation of the effluent was assessed with the coagulation strategy and dose optimized in batch.

For both the batch and continuous experiments, stoichiometric relations were considered for determining the hydroxide dose in the Fe + Ca and Fe + Mg treatments Eqs. (1) and (2):

$$2FeCl_3 + 3Ca(OH)_2 \xrightarrow{H_2O} 3Ca^{2+} + 6Cl^- + 2Fe(OH)_3 \quad (1)$$

$$2FeCl_3 + 3Mg(OH)_2 \xrightarrow{H_2O} 3Mg^{2+} + 6Cl^- + 2Fe(OH)_3 \quad (2)$$

Hydroxides were added to attain a 2:3 molar ratio of iron chloride. Hydroxide prevents the acidification arising from iron addition and poor coagulation due to low effluent alkalinity. Treatment of low alkalinity wastewaters is difficult due to the drastic changes in pH induced by coagulants [8]. Hydroxide addition counteracts the effect of acid coagulants maintaining suitable pH conditions for phosphorus recovery.

Continuous flocculation was examined within an empty acrylic tube (ET), a mixer formed by an arrangement of three contraction-expansion stages (CE), and an eight-element Koflo type static mixer (SM) [29]. CE arrays are currently being studied for mixing operations [30], particularly for micromixing [31]. Testing their application in the flocculation process was considered, since the diffusion enhancement may improve the coagulants effect. The ET and the mixers used had a length of 0.3 m and an inner diameter of 1.9 cm (¾ inch). The CE mixer had contractions-expansions of 1.9 to 1.27 cm (¾ to ½ inch), and 1.27 to 1.9 cm (½ to ¾ inch), separated by 3.5 cm in between. Schemes of the tested static mixers are shown in Fig. 1a and b.

The effluent and the model fluid were driven from a 200 L reservoir using a centrifugal pump for the main flow, and two peristaltic pumps for the addition of the coagulant (Fig. 1c). The flow rates of the coagulant solutions were set as 5% of the effluent flow rate. The resultant coagulant concentrations were the ones optimized from the batch experiments. Stationary behavior was observed almost from the beginning. Hence, 10 min was allowed for stabilization before taking three samples successively, every 5 min, with the purpose of assessing the settling curves, and for further analytical determinations. As for the settling curves data, these were obtained directly by collecting the samples from the mixer or the ET outlet. The experiment with continuous flocculation of the effluent in the Koflo type static mixer with a flow rate of 2 L min$^{-1}$ was continued for one more hour to check for the process stability. Samples were collected at 10, 20, 30, and 60 min after the initial ones to measure the settling curves.

Settling curves were measured in 2 L standard graduated cylinders for both the batch and continuous experiments. Once a clear liquid–solid interface was developed, the time taken for it to reach different positions was recorded [23,32]. This single settling assay was carried out by triplicate. For a complete analysis of the whole settling curve, the limit solid flux (LSF) concept was considered since it is directly related to the final settler area required [8,33]. The solid flux is the rate of solid mass moving downward across a unit area in the settler, as defined by Tchobanoglous et al. [8]. The LSF is the minimum one required for a settler to handle a given organic load, and it is used to dimension the settlers. Such flux can be estimated from a single assay following the Kinch theory [8,33], assuming a model to describe the relationship between the settling velocity and the solids concentration; the Vesilind model was used as recommended in the literature [8,32,33]. A higher solid flux leads to better performance; thus, a lower settler area is required for the settling process.

The CST test [23,24] was used to compare the flocs strength generated in batch with the ones obtained while

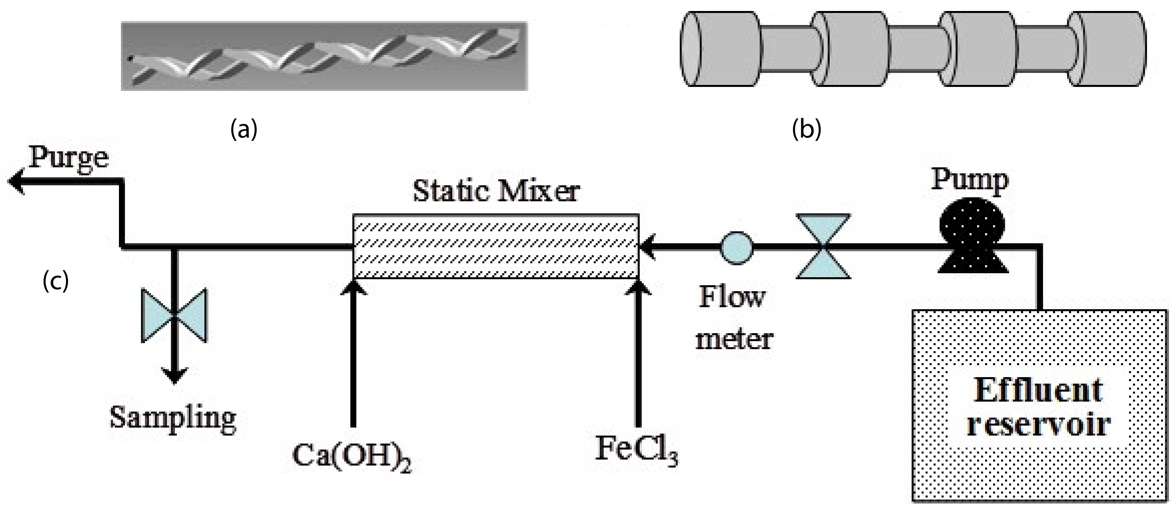


Fig. 1. Schemes of the static mixers, and flowsheet used for testing the continuous coagulation process: (a) Koflo type static mixer, (b) contraction-expansion static mixer, (C) schematics of the flowsheet.

promoting flocculation within the Koflo type static mixer. The CST test measures the filtration force generated by the capillary action of a chromatography paper (Whatman n°17 or equivalent) applied to a sludge sample. The lower the CST, the higher the floc's strength and dewatering capability.

## 3. Results and discussion

### *3.1. Feedlot effluent characterization and batch experiments*

Table 1 details the main features of the effluent collected sample. Low alkalinity was found, together with a high amount of nitrogen and phosphorus. The COD and nutrients were well above local discharge limits, which could be very dangerous for aquatic life preservation if disposed of without treatment [34]. The general characteristics of the feedlot effluent examined in this study were comparable to those reported in the literature for the feedlots of other countries [5,35,36].

Figs. 2 and 3 show the dose optimization curves for the flocculation experiments performed in batch using single coagulants.

Regarding removal efficiency, though strongly dependent on the dose, calcium hydroxide was effective in removing organic phosphorous, reaching values below 2 mg $L^{-1}$ with an optimum dose of 8 mM. As for nitrogen and TRP, the removal was less than 30% despite meeting conditions for calcium phosphates precipitation [37], which suggests a kinetic hindrance. Cao et al. [38] showed that humic acids generally inhibit calcium phosphates nucleation by adsorbing to the new nuclei and by complexation of calcium ions. These authors found more than 94% precipitation inhibition of calcium phosphates in the presence of humic acids. Magnesium hydroxide evidenced a similar behavior for organic nitrogen and the TRP. Organic phosphorous removal was less efficient and ammonia removal was almost negligible, despite the conditions were appropriate for struvite precipitation [39,40]. Again, the presence of organic matter could have inhibited precipitation. For both hydroxides, the attained COD and turbidity removal efficiencies were less than 20% whatever the dose used (Fig. 3), suggesting that pH changes and $Ca^{2+}$ or $Mg^{2+}$ were not effective to flocculate the existing organic matter.

Table 1
Raw effluent characterization

| Variable | Mean | VC %[a] | Unit |
|---|---|---|---|
| pH | 7.5 | 1.9 | |
| Electrical conductivity | 3 | 19.6 | mS $cm^{-1}$ |
| Hardness | 7.5 | 7.2 | mM |
| Alkalinity | 29.4 | 12.0 | mM |
| Turbidity | 1,161 | 7.8 | NTU |
| Ammonia ($NH_4$) | 51.1 | 16.7 | mg N $L^{-1}$ |
| Total reactive phosphate (TRP) | 91.3 | 26.0 | mg P $L^{-1}$ |
| Total Kjeldahl nitrogen (TKN) | 302.14 | 26.4 | mg N $L^{-1}$ |
| Total Phosphorus (TP) | 110.13 | 27.8 | mg P $L^{-1}$ |
| Total iron | 3.85 | 11.3 | mg Fe $L^{-1}$ |
| Chemical oxygen demand (COD) | 4,778.2 | 24.2 | mg $O_2$ $L^{-1}$ |

[a]Variation coefficient: standard deviation divided by the mean.

Iron chloride addition promoted excellent nitrogen and phosphorus removal efficiencies (Fig. 2), which is consistent with literature information for sewage, swine, dairy, cattle and poultry wastewaters [8,12,13,41]. Moreover, COD and turbidity decreased by more than 70% when high doses (above 25 mM) were used (Fig. 3). However, a bulking effect was found after 30 to 50 min when the iron dose was above 5 mM (Fig. 4). This effect could be related to the effluent low alkalinity (Table 1) and the acid character of iron (III) leading to carbon dioxide formation, saturation and evolution (detected qualitatively) after iron addition. The formation of micro-bubbles over the high specific surface flocs was likely responsible for the bulking. After adding iron chloride, the pH decreased from 5 to 1.5 when the dose was high (Fig. 2c). It is worth recalling that, when calcium or magnesium hydroxide was added, bulking did not occur, which is likely because the pH did not decrease (Fig. 2a and b). The resulting sewage contained a low solid fraction of around 30%, indicating the formation of low-density flocs with a high risk of bulking.

The effects of the combined treatments (iron chloride plus calcium or magnesium hydroxide: Fe + Ca or Fe + Mg) on removal efficiencies, alkalinity and pH are illustrated in Fig. 5 and can be compared with Fig. 2 to judge the advantages. Fig. 6 shows the combined treatments ability to eliminate

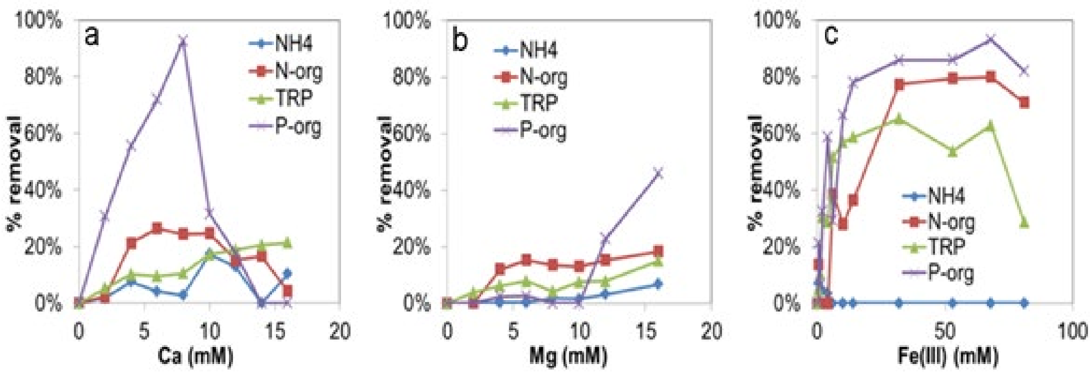


Fig. 2. Dose optimization for flocculation in a batch when using (a) calcium hydroxide, (b) magnesium hydroxide or (c) iron chloride as coagulants. $NH_4$: ammonia, N-Org: organic nitrogen fractions, TRP: total reactive phosphate, P-org: organic phosphorous fractions.

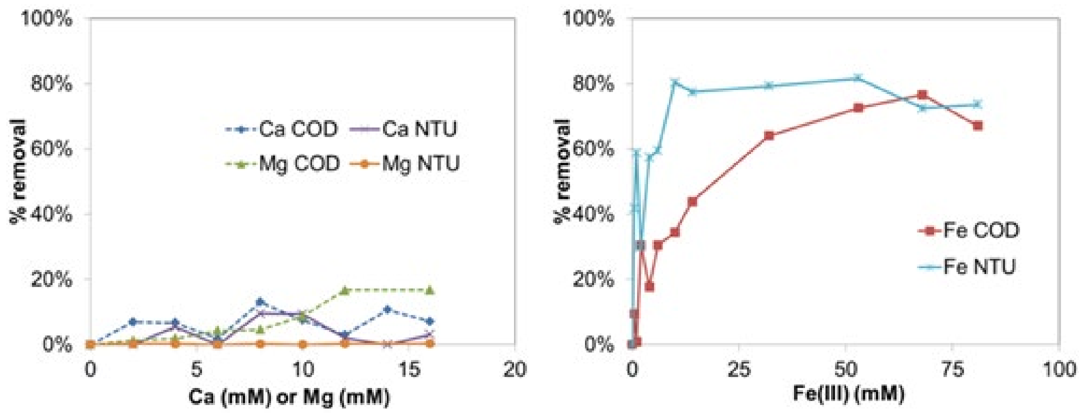


Fig. 3. Chemical oxygen demand (COD) and turbidity (NTU) removal efficiencies in batch, when using calcium hydroxide, magnesium hydroxide or iron chloride as coagulants.

Fig. 4. Bulking effect observed after flocculation promoted by using 10 mM iron chloride.

COD and turbidity, which should be compared with Fig. 3. The results point to the excellent performance of the combined treatments. Both treatments led to similar removal efficiencies, such as about 50% of the organic fractions of nitrogen and phosphorus, and almost all the TRP. Calcium or magnesium hydroxide addition was successful in preventing pH decrease induced by incorporating iron (III) to a low alkalinity raw effluent (Fig. 5).

Phosphates precipitation determined an almost complete elimination of the TRP. The ammonia removal efficiency was around 10%, which is probably related to volatilization given that the pH transiently increased after hydroxide addition, before attaining complete mixing and neutralization. COD removal efficiencies were higher than 80%, with almost total turbidity elimination for doses above 6 mM iron (Fig. 6).

Alkalinity was strongly modified when using iron chloride as a coagulant in high doses (Fig. 2), decreasing markedly due to the acid character of iron (III). Compensation by subsequent addition of calcium or magnesium hydroxide prevented an excessive decrease of the treated effluent alkalinity, keeping pH within dischargeable limits while promoting flocculation (Fig. 5).

### 3.2. Static mixer coagulation–flocculation optimization

The effect of using a static mixer instead of an agitated vessel was examined by inducing the coagulation–precipitation of calcium carbonate from the model system described in section 2. Calcium carbonate precipitation was significantly influenced by the mixing device used for assisting the flocculation/precipitation stage. Fig. 7 shows the measured settling curves. The Koflo type static mixer had the best performance, with the highest initial settling rate and the lowest compression effect. Only the ET at low and middle flow rates were able to attain similar behavior. The CE mixer promoted the formation of flocs with worse settling features than those arising from the other configurations. The ET and CE mixers led to similar results at the highest flow rate examined. Considering the profiles reported by Lü et al. [30], it is likely that mixing eddies around expansions and contractions produce good mixing but high shear stress. Hence, turbulent forces would prevent floc aggregation resulting in a detrimental effect on the settling curve.

The settling curves determined when promoting flocculation/precipitation of the model system within the Koflo type static mixer or the ET were both better than flocculation within an agitated vessel, in agreement with previous findings for other wastewaters [22]. The Koflo type static mixer led to flocs with better settling features and less dependent on the flow rate. Apparently, the Koflo type static mixer rapidly accomplished a good coagulant distribution, increasing the chances of collision and aggregation for incipient flocs. Therefore, a decrease in residence time at high flow rates caused a milder effect than in an ET.

The initial settling velocities, determined from the settling curves, are detailed in Table 2. When using the Koflo static mixer, the flow rate had low influence on the initial settling velocity and the opposite behavior was found with the ET, in agreement with previous findings for other wastewaters [22].

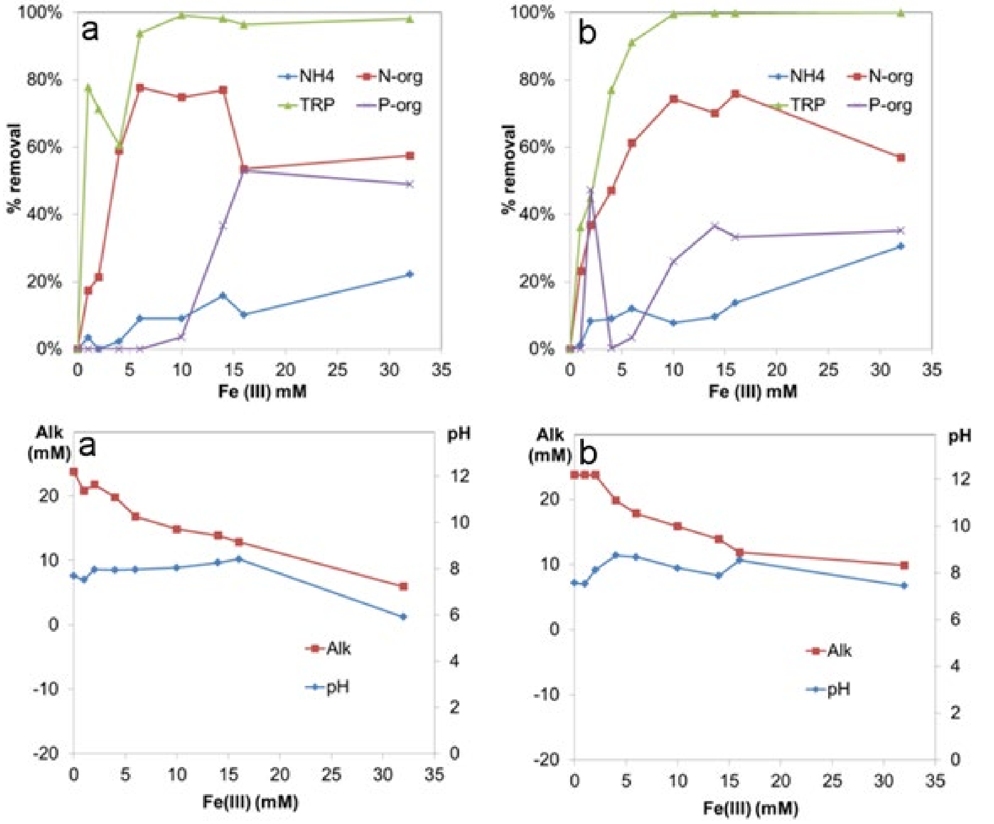


Fig. 5. Removal efficiencies attained when using the combined addition of iron chloride and subsequent calcium hydroxide (a) or magnesium hydroxide (b). NH4: ammonia, N-Org: organic nitrogen fractions, TRP: total reactive phosphate, P-org: organic phosphorous fractions, Alk: alkalinity.

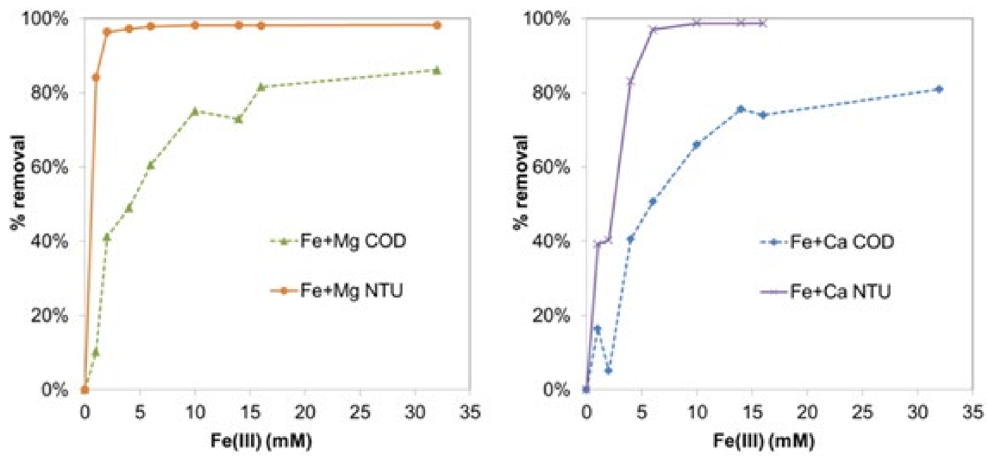


Fig. 6. Comparison of chemical oxygen demand (COD) and turbidity (NTU) removal efficiencies obtained by the combined addition of iron chloride and subsequent calcium (Fe + Ca) or magnesium (Fe + Mg) hydroxide.

Based on the results obtained for the model precipitation/flocculation/settling system, the Koflo type static mixer together with the combined addition of iron chloride and subsequent calcium hydroxide was selected for performing a continuous flocculation experiment with the effluent. Fig. 8 presents the settling curves measured for the flocculation promoted in the batch agitated vessel and within the Koflo type static mixer in the continuous system. In agreement with the results found when using the model system, the settling curve obtained when the flocculation was induced in the Koflo type static mixer showed a significantly better behavior for settling compared to the one obtained in the batch agitated vessel.

For the three flow rates, flocculation within the Koflo type static mixer led to higher initial settling velocities and

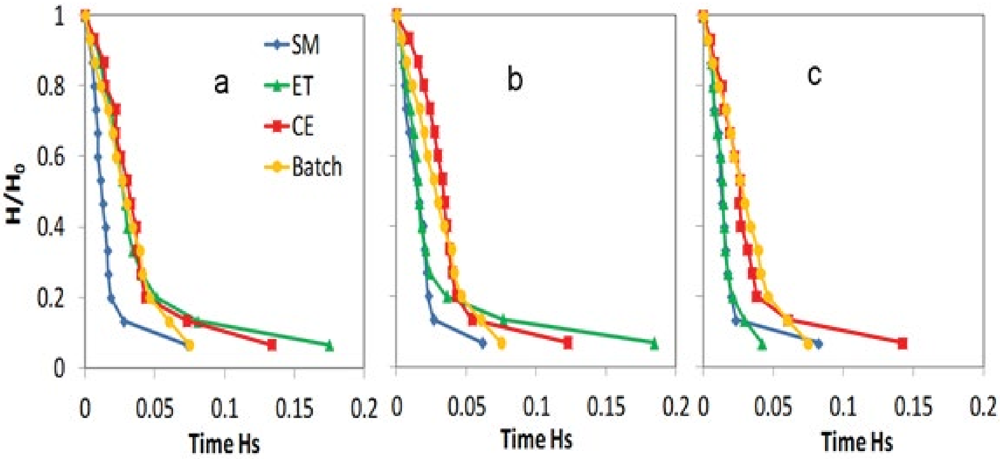


Fig. 7. Settling curves for the different mixing methods used during the flocculation stage with the model calcium carbonate precipitation/flocculation system at different flow rates: (a) 2.0 L min⁻¹, (b) 1.3 L min⁻¹, and (c) 0.7 L min⁻¹. SM: Koflo type static mixer, ET: empty tube, CE: contraction-expansion mixer, Batch: agitated vessel. The curve obtained for batch is included as a reference in all the figures.

Table 2
Initial velocities determined from the settling curves shown in Fig. 7

| Operation strategy/ mixer | Flow rate (L min$^{-1}$) | Initial velocity (cm s$^{-1}$) | (*) |
|---|---|---|---|
| Continuous-SM | 2.0 | 0.66 | *A* |
| Continuous-SM | 1.3 | 0.49 | *B* |
| Continuous-SM | 0.7 | 0.63 | *A* |
| Continuous-ET | 2.0 | 0.30 | *E* |
| Continuous-ET | 1.3 | 0.48 | *B* |
| Continuous-ET | 0.7 | 0.60 | *A* |
| Continuous-CE | 2.0 | 0.23 | *E* |
| Continuous-CE | 1.3 | 0.19 | *F* |
| Continuous-CE | 0.7 | 0.26 | *E* |
| Batch | – | 0.25 | *E* |

*Different letters indicate significant differences (alpha = 0.05).
SM: Koflo static mixer, ET: empty tube, CE: contraction-expansion mixer.

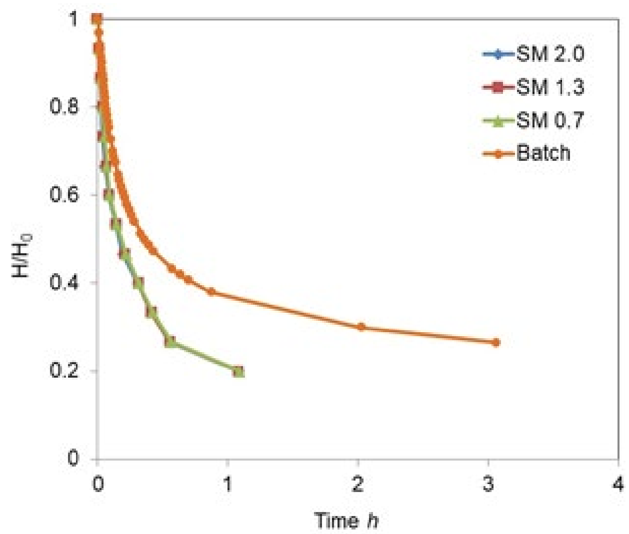


Fig. 8. Settling curves for the different mixing methods used during the flocculation stage when treating the real effluent. Numbers in the legend are the flow rates in L min$^{-1}$. $H/H_0$ is the interface position relative to the initial column height.

a smaller compressive behavior than the flocculation in the batch agitated vessel. Estimated initial settling velocities (Table 3) pointed to a slight increase in the settling efficiency with a higher flow rate. This behavior was coincident with the one reported by [20] for mining wastewater flocculated within a Koflo static mixer. The stability of the process was checked by prolonging the continuous flocculation for 1 h at 2 L min$^{-1}$. Initial settling velocities determined from the settling curves are also included in Table 3; the values found were almost invariable highlighting the stability of the process.

Characterization of the liquid obtained with the continuous treatment evidenced improvements of the removal efficiencies (Fig. 9) compared to the results obtained in the batch agitated vessel. TRP and turbidity were almost negligible in the clarified liquid. For nitrogen and phosphorous organic fractions (N-Org and P-Org), average removal efficiencies of around 80% were obtained. The decrease of the COD was larger than 90%.

The positive effect of the static mixer induced flocculation was particularly evident for the phosphorous organic fraction and the COD removal efficiencies. This result is likely related to the fast and good coagulant distribution accomplished with the Koflo static mixer. Rapid coagulant distribution would benefit the reaction between iron, phosphate and organic matter, preventing loss of iron as ferric hydroxide [22]. The slight increase in removal capability with smaller flow rates could be attributed to an increase in residence time. The ammonia removal efficiency was much higher than in batch conditions, likely arising from volatilization after hydroxide addition. Although pH levels

Table 3
Initial velocities estimated from the settling curves shown in Fig. 8. Operation at 2 L min$^{-1}$ was continued for 1 h, taking samples at 10, 20, 30, and 60 min

| Operation strategy/ mixer | Flow rate, (L min$^{-1}$) | Mean initial velocity, (cm s$^{-1}$) | (*) |
|---|---|---|---|
| Batch | | 0.026 | *A* |
| Continuous SM | 0.7 | 0.080 | *B* |
| Continuous SM | 1.3 | 0.086 | *B* |
| Continuous SM | 2.0 | 0.107 | *C* |
| Continuous SM-10 | 2.0 | 0.103 | *C* |
| Continuous SM-20 | 2.0 | 0.087 | *B* |
| Continuous SM-30 | 2.0 | 0.089 | *B* |
| Continuous SM-60 | 2.0 | 0.094 | *B* |

*Different letters indicate significant differences (alpha = 0.05).

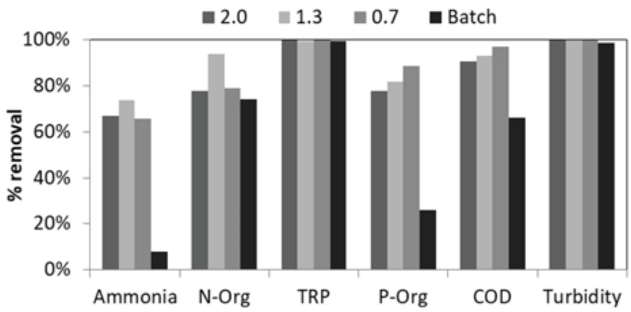


Fig. 9. Removal efficiencies attained by flocculation of the effluent in the Koflo static mixer at different flow rates and subsequent sedimentation; numbers in the legend indicate the flow rate in L min$^{-1}$. Results obtained in the batch agitated vessel are included for comparison. Both continuous and batch removal percentages are referred to the same optimum iron dose (10 mM Fe). N-org, P-org: nitrogen and phosphorous organic fractions, TRP: total reactive phosphate, COD: chemical oxygen demand.

measured in the clarified liquid several minutes after taking the sample from the mixer outflow were around 7 ± 0.5, local alkaline conditions (which can lead to ammonia volatilization) might be one reason for the strong ammonia concentration decrease due to a poor hydroxide instantaneous mixing. Hence, the addition of another static mixer after hydroxide addition could prevent volatilization by promoting fast distribution of the hydroxide.

A settling curve analysis was carried out to determine the LSF considering a solid concentration in the dense region 7.5 times higher than the initial one. When continuous flocculation was promoted within the Koflo static mixer, the LSF estimated from the settling curve was 714.0 kg m$^{-2}$ h$^{-1}$ for a flow rate of 2 L min$^{-1}$. The LSF determined from the settling curve in the agitated vessel under batch conditions was significantly lower, LSF = 102.6 ± 29.9 kg m$^{-2}$ h$^{-1}$. Hence, the area required for a settler would be considerably smaller if the continuous flocculation within a Koflo static mixer is used instead of a batch stirred tank.

The other examined flow rates led to limit solid fluxes of 809.9 and 823.0 kg m$^{-2}$ h$^{-1}$ for 1.3 L and 0.7 L min$^{-1}$, respectively.

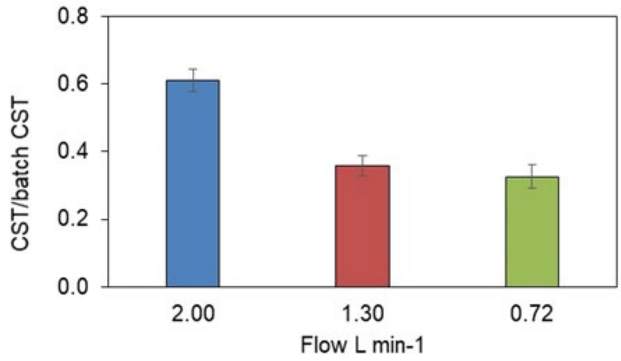


Fig. 10. Capillary suction time (CST) test determined for the flocs formed from the effluent in the Koflo type static mixer at different flow rates. The values refer to the one determined for the flocculation in the batch agitated vessel.

Settler areas up to eight times smaller would be required by inducing the flocculation in the Koflo static mixer. Therefore, the mixing process seriously affects the settling behavior of this type of effluent. Moreover, the use of a Koflo static mixer for the flocculation stage appears to be highly beneficial for minimizing installation and operation costs, while achieving improved removal efficiencies.

Finally, the flocs strength was examined using the CST technique, as proposed in [24]. Results (Fig. 10) indicate a significant increase of the flocs strength if formed in the Koflo static mixer instead of using a batch agitated vessel. The flocs strength further increased as the flow rate decreased. However, the relative difference between flow rates becomes smaller as they decrease. The flow rate will be a compromise considering the installation size and capacity.

## 4. Conclusion

The main conclusions arising from the performed study of coagulation/flocculation and subsequent sedimentation of a feedlot effluent are the following:

- Calcium hydroxide or magnesium hydroxide alone did not show good performance as coagulants of the tested effluent.
- Iron(III) chloride was efficient to promote the effluent flocculation/precipitation, with average removal efficiencies larger than 80% for the organic nitrogen and phosphorous fractions, and turbidity. However, such levels were achieved only at large doses, over 25 mM $Fe^{3+}$, inducing a pH decrease to less than 5 and triggering a bulking effect.
- The combined addition of iron chloride (10 mM $Fe^{3+}$ dose) and subsequent calcium or magnesium hydroxide to neutralize, led to almost quantitative elimination of the TRP and the turbidity of the examined effluent while decreasing the COD by around 70%.
- Calcium carbonate precipitation in a solution containing polyacrylamide was found to be an appropriate model system for optimizing the mixing strategies of the coagulation–flocculation operation. The use of this model

system deeply simplified the possibility of examining the influence of mixing procedures on the flocculation stage without using the actual effluent.
- Continuous flocculation in a Koflo type static mixer significantly improved the removal efficiency of the pollutants compared to flocculation in a batch agitated vessel. The most affected variables were the phosphorous organic fraction that moved from ~20% to ~90%, and the COD that moved from ~60% to ~90%. Moreover, the formed flocs showed improved settling characteristics: >3 times larger initial settling velocities and >7 times larger limit solid fluxes. Therefore, settler areas up to 8 times smaller than the ones used in the case of a conventional stirred-tank – thickener arrangement would be needed, with the resultant cost reduction. Hence, continuous flocculation in a Koflo type static mixer with a subsequent settler to achieve the solid–liquid separation constitutes a promising strategy for a time-effective treatment of the feedlot runoff effluent contained in accumulation ponds.

### Acknowledgments

Financial support from FONCyT (PICT2014-0704) and Universidad de Buenos Aires (UBACyT 20020130100544BA and UBACyT 20020150100156BA) are gratefully acknowledged. We would also like to thank Chem. Eng. Stella Piovano and PhD. Gabriel Salierno for their help and support.

### Symbols and abbreviations

| | | |
|---|---|---|
| Alk | — | Alkalinity, mM |
| Ca | — | Calcium hydroxide treatment, – |
| CE | — | Contraction-expansion continuous mixer, – |
| COD | — | Chemical oxygen demand, mg $L^{-1}$ |
| CST | — | Capillary suction time, s |
| ET | — | Empty tube continuous mixer, – |
| Fe | — | Iron (III) chloride treatment, – |
| Fe + Ca | — | Iron (III) chloride - calcium hydroxide treatment, – |
| Fe + Mg | — | Iron (III) chloride - magnesium hydroxide treatment, – |
| $H$ | — | Interface height, cm |
| $H_0$ | — | Initial interface height, cm |
| LSF | — | Limit solid flux, kg $m^{-2}$ $h^{-1}$ |
| Mg | — | Magnesium hydroxide treatment, – |
| $NH_4$ | — | Ammonia nitrogen, mg $L^{-1}$ |
| N-Org | — | Organic nitrogen, mg $L^{-1}$ |
| NTU | — | Turbidity, nephelometric turbidity unit |
| P-Org | — | Organic phosphorous, mg $L^{-1}$ |
| rpm | — | Revolutions per minute, $min^{-1}$ |
| SM | — | Koflo static mixer, – |
| TKN | — | Total Kjeldahl nitrogen, mg $L^{-1}$ |
| TP | — | Total phosphorus, mg $L^{-1}$ |
| TRP | — | Total reactive phosphate, mg $L^{-1}$ |
| mM | — | Milli molarity, mmol $L^{-1}$ |

### References

[1] A.R. García, S.N. Fleite, D. Vazquez Pugliese, A.F. de Iorio, Feedlots and pollution—a growing threat to water resources of agro-production zone in Argentina, Environ. Sci. Technol., 47 (2013) 11932–11933.
[2] A.R. García, R. Maisonnave, M.J. Massobrio, A.R. Fabrizio de Iorio, Field-Scale evaluation of water fluxes and manure solution leaching in feedlot pen soils, J. Environ. Qual., 41 (2012) 1591–1599.
[3] A.R. García, A.F. de Iorio, Phosphorus distribution in sediments of morales stream (a tributary of the Matanza-Riachuelo River, Argentina), the influence of organic point source contamination, Hydrobiologia, 492 (2003) 129–138.
[4] B. Eghball, J.E. Gilley, Phosphorus and nitrogen in runoff following beef cattle manure or compost application, J. Environ. Qual., 28 (1999) 1201.
[5] M. Hjorth, K.V. Christensen, M.L. Christensen, S.G. Sommer, Solid–Liquid Separation of Animal Slurry in Theory and Practice, E. Lichtfouse, M. Hamelin, M. Navarrete, P. Debaeke, Eds., Sustainable Agriculture, Volume 2, Springer, Netherlands, 2011, pp. 953–986.
[6] E.A. De Vuyst, S.W. Pryor, G. Lardy, W. Eide, R. Wiederholt, Cattle, ethanol, and biogas: does closing the loop make economic sense?, Agric. Syst., 104 (2011) 609–614.
[7] M.E.C. Caruana, Organizational and economic modeling of an anaerobic digestion system to treat cattle manure and produce electrical energy in Argentina's feedlot sector, J. Cleaner Prod., 208 (2019) 1613–1621.
[8] G. Tchobanoglous, H. Stensel, R. Tsuchihashi, F. Burton, M. Abu-Orf, G. Bowden, W. Pfrang, Wastewater Engineering: Treatment, Reuse, and Recovery, Metcalf and Eddy Inc., 5th ed., McGraw Hill, USA, 2014.
[9] A. Amirtharajah, C. O'melia, Eds., Coagulation Processes: Destabilization, Mixing, and Flocculation, McGraw-Hill, Inc., USA, 1990.
[10] H. Huang, P. Zhang, Z. Zhang, J. Liu, J. Xiao, F. Gao, Simultaneous removal of ammonia nitrogen and recovery of phosphate from swine wastewater by struvite electrochemical precipitation and recycling technology, J. Cleaner Prod., 127 (2016) 302–310.
[11] L.E. de-Bashan, Y. Bashan, Recent advances in removing phosphorus from wastewater and its future use as fertilizer (1997–2003), Water Res., 38 (2004) 4222–4246.
[12] D.M. Sievers, M.W. Jenner, M. Hanna, Treatment of dilute manure wastewaters by chemical coagulation, Trans. ASABE, 37 (1994) 597–601.
[13] J.J. Sherman, H.H. Van Horn, R.A. Nordstedt, Use of flocculants in dairy wastewaters to remove phosphorus, Appl. Eng. Agric., 16 (2000) 445–452.
[14] R. Laridi, J.-C. Auclair, H. Benmoussa, Laboratory, and pilot-scale phosphate and ammonium removal by controlled struvite precipitation following coagulation and flocculation of swine wastewater, Environ. Technol., 26 (2005) 525–536.
[15] A. Thapa, S. Rahman, M.S. Borhan, Remediation of feedlot nutrients runoff by electrocoagulation process, Am. J. Environ. Sci., 11 (2015) 366–379.
[16] E. Butler, R.E. De Otte, C.F.M. Clewett, O. Mulamba, N. Spaar, Y.-T. Hung, Treatment of beef cattle feedlot wastewater by electrocoagulation technology, Desal. Water Treat., 101 (2018) 77–85.
[17] V. Khandegar, S. Acharya, A.K. Jain, Data on treatment of sewage wastewater by electrocoagulation using a punched aluminum electrode and characterization of generated sludge, Data Brief, 18 (2018) 1229–1238.
[18] K. Bensadok, N. El Hanafi, F. Lapicque, Electrochemical treatment of dairy effluent using combined Al and Ti/Pt electrodes system, Desalination, 280 (2011) 244–251.
[19] D. Fitria, M. Scholz, G.M. Swift, Impact of different shapes and types of mixers on sludge dewaterability, Environ. Technol., 34 (2013) 931–936.
[20] A. Demoz, Scaling inline static mixers for flocculation of oil sand mature fine tailings, AIChE J., 61 (2015) 4402–4411.
[21] A. Ghanem, T. Lemenand, D. Della Valle, H. Peerhossaini, Static mixers: mechanisms, applications, and characterization methods–a review, Chem. Eng. Res. Des., 92 (2014) 205–228.
[22] A. Amirtharajah, S.C. Jones, Mixing for Coagulation: Organic Polymers, Static Mixers, and Modeling, H. Hahn, E. Hoffmann,

H. Odegaard, Eds., Chemical Water and Wastewater Treatment IV, Springer, Berlin, Heidelberg, 1996, pp. 3–15.
[23] B. Rodger, L. Bridgewater, Standard Methods for the Examination of Water and Wastewater, 23rd ed., American Public Health Association, Washington, D.C., 2017.
[24] O. Sawalha, M. Scholz, Impact of temperature on sludge dewatering properties assessed by the capillary suction time, Ind. Eng. Chem. Res., 51 (2012) 2782–2788.
[25] B. Gao, B. Liu, T. Chen, Q. Yue, Effect of an aging period on the characteristics and coagulation behavior of polyferric chloride and polyferric chloride–polyamine composite coagulant for synthetic dyeing wastewater treatment, J. Hazard. Mater., 187 (2011) 413–420.
[26] P.T. Spicer, W. Keller, S.E. Pratsinis, The effect of impeller type on floc size and structure during shear-induced flocculation, J. Colloid Interface Sci., 184 (1996) 112–122.
[27] V.A. Mhaisalkar, R. Paramasivam, A.G. Bhole, Optimizing physical parameters of rapid mix design for coagulation–flocculation of turbid waters, Water Res., 25 (1991) 43–52.
[28] D.D. Harris, A Kinetic Study of Aqueous Calcium Carbonate, Master Thesis, Brigham Young University, Provo, USA, 2013.
[29] Y.F. Maa, C. Hsu, Liquid–liquid emulsification by static mixers for use in microencapsulation, J. Microencapsulation, 13 (1996) 419–433.
[30] Y. Lü, S. Zhu, K. Wang, G. Luo, Simulation of the mixing process in a straight tube with sudden changed cross-section, Chin. J. Chem. Eng., 24 (2016) 711–718.
[31] E. Yıldırım, Analysis, and testing of a contraction-and-expansion micromixer for micro-milled microfluidics, Microsyst. Technol., 23 (2017) 4797–4804.
[32] L.K. Wang, Y.T. Hung, K.K. Shammas, Eds., Handbook of Environmental Engineering, Volume 3, Physicochemical Treatment Processes, The Humana Press Inc., Totowa, NJ, 2005.
[33] J.C. Crittenden, R.R. Trussell, D.W. Hand, K.J. Howe, G. Tchobanoglous, MWH's Water Treatment: Principles and Design, John Wiley & Sons, Hoboken, New Jersey, 2012.
[34] http://ceqg-rcqe.ccme.ca/en/index.html (Accessed on March 05, 2019)
[35] L.M. Pepple, D.S. Andersen, R.T. Burns, L.B. Moody, Physical and chemical properties of runoff effluent from beef feedlots in Iowa, Trans. ASABE, 54 (2011) 1079–1084.
[36] J.L. Hatfield, B.A. Stewart, Animal Waste Utilization: Effective Use of Manure as a Soil Resource, CRC Press, Boca Raton, Florida, USA, 1997.
[37] W.A. House, The physico-chemical conditions for the precipitation of phosphate with calcium, Environ. Technol., 20 (1999) 727–733.
[38] X. Cao, W.G. Harris, M.S. Josan, V.D. Nair, Inhibition of calcium phosphate precipitation under environmentally-relevant conditions, Sci. Total Environ., 383 (2007) 205–215.
[39] S. Kataki, H. West, M. Clarke, D.C. Baruah, Phosphorus recovery as struvite: recent concerns for use of seed, alternative Mg source, nitrogen conservation, and fertilizer potential, Resour. Conserv. Recycl., 107 (2016) 142–156.
[40] I. Çelen, J.R. Buchanan, R.T. Burns, R. Bruce Robinson, D. Raj Raman, Using a chemical equilibrium model to predict amendments required to precipitate phosphorus as struvite in liquid swine manure, Water Res., 41 (2007) 1689–1696.
[41] K. Fytianos, E. Voudrias, N. Raikos, Modeling of phosphorus removal from aqueous and wastewater samples using ferric iron, Environ. Pollut., 101 (1998) 123–130.