Experimental and Modeling of a Hybrid Algal Photosynthesis and Ion-Exchange (HAPIX) Process for Side Stream Wastewater Treatment

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ABSTRACT

A hybrid ion-exchange and algae photosynthesis (HAPIX) process was used for treatment of side stream centrate from an anaerobic digester treating waste activated sludge. Although the high NH_4^+ -N concentration of the centrate (~1180 mg/L) inhibited the algae growth in unamended controls, addition of 150 g/L of zeolite reduced the ammonia toxicity due to its ion exchange capacity. NH_4^+ -N was reduced from 1,180 mg/L to 107 mg/L within 24 hours by ion exchange. Na^+ was the major cation exchanged with NH_4^+. The addition of algae further reduced the NH_4^+ -N concentration to 10.5 mg/L after 8 days of operation. Zeolite that was saturated with NH_4^+ can be bioregenerated by the algae growth so that the zeolite can adsorb more NH_4^+ in the wastewater. The mathematical model that combined ion-exchange and algal photosynthesis processes predicted the aqueous NH_4^+ -N concentration well. The HAPIX process is feasible to treat high NH_4^+ -N side stream wastewater.

KEYWORDS

Ion-exchange, algal photosynthesis, side stream, mathematical modeling, NH_4^+ removal.

INTRODUCTION

Management of the nitrogen (N) cycle was identified by the National Academy of Engineering (NAE) as one of the grand challenges of the 21st century. In particular, high NH_4^+ strength wastewaters, such as industrial wastewaters (e.g. food processing, fertilizer, plastic industries), landfill leachate, source separated urine and centrate from anaerobic digestion, are challenging and expensive to treat (Kjeldsen et al., 2002; Udert et al., 2003). Anaerobic digestion centrate is of particular interest since anaerobic digestion technology is being promoted for stabilization and bioenergy recovery from waste resources (Cantrell et al., 2008). The high NH_4^+ strength centrate is often recycled back to the head of the wastewater treatment plant, resulting in high irregular nutrient loads that can upset mainstream biological N removal (BNR) processes and increase costs (Fux et al., 2006; Wett & Alex, 2003).

Although a number of advanced BNR processes, such as shortcut nitrification-denitrification and anaerobic ammonium oxidation (anammox), have been developed to reduce the energy and chemical costs of high NH_4^+ strength wastewater treatment (Ciudad et al., 2005;; Kumar & Lin, 2010; Ruiz et al., 2006), utilization of algae for treatment of these wastewater presents an opportunity for co-production of biofuels, high value chemicals and animal feeds (Li et al., 2011; Park et al., 2010; Rusten & Sahu, 2011; Wang & Park, 2015; Wang et al., 2015). The high nutrient concentrations in these wastewaters also have the potential to support higher algal biomass densities than low-strength wastewaters, resulting in lower downstream costs for thickening and dewatering (Halfhide et al., 2015). A challenge with this approach is that NH_4^+-N concentrations greater than 200 to 300 mg/L are known to inhibit algae growth due to the uncoupling effect of free NH_3 on photosynthetic processes in chloroplasts (Crofts, 1966; Park et al., 2010). Prior studies have addressed this issue by either dilution with fresh water or low strength wastewater (Yuan et al., 2012) or using reactor configurations that reduce the NH_3 concentrations that algae are exposed to (Halfhide et al., 2015).

Zeolites are natural hydrated aluminosilicate materials with a high affinity for NH_4^+ ions (Malovanyy *et al.*, 2013; Rožić *et al.*, 2000). Common forms of natural zeolite include clinoptilolite, mordenite, phillipsite, chabazite, stilbite, analcime and laumontite (Wang & Peng, 2010). Clinoptilolite is the most abundant and lowest cost natural zeolite material; however, its NH_4^+ capacity is low compared with chabazite (Aponte-Morales, 2015), which was used in this study. Prior studies of biological treatment of high NH_4^+ strength wastewaters have successfully used the ion exchange (IX) capacity of natural zeolites to reduce the toxicity of free NH_3 to nitrifying prokaryotes (Aponte-Morales et al., 2016; Martins et al., 2017; Tada et al., 2005); however, this approach has not been previously used with algae. In this study, we propose a novel hybrid algal photosynthesis and ion-exchange (HAPIX) process to recover nutrients from high NH_4^+-N strength wastewater. It is hypothesized that NH_4^+ in the wastewater will be absorbed by the zeolite and exchanged with cations such as Na^+ and K^+. Adsorption of NH_4^+ will reduce free NH_3 concentrations in the liquid phase to below inhibitory levels for algae growth. As algae utilize NH_4^+ in solution, NH_4^+ stored in the zeolite will be slowly released, allowing for additional algal biomass production while regenerating the IX capacity of the zeolite.

This study evaluated the feasibility of a HAPIX process for algae cultivation and treatment of high NH_4^+ strength wastewater. A mathematical model was also developed to predict the HAPIX process including temporal evolution of the NH_4^+ concentration.

METHODOLOGY

Characteristics of anaerobic digester centrate

Centrate used in this study was obtained from a pilot anaerobic digester treating waste activated sludge (WAS) from a local enhanced phosphorus removal treatment facility. The anaerobic digester had a working volume of 18 L and was operated at 45 °C and a solids residence time of 21 days for over 100 days. Effluent from the digester was centrifuged at 4000 rpm for 15 mins. The supernatant from this initial solid-liquid separation step was further filtrated through 1.5 μm filter paper (934-AH, Whatman). The filtrate was collected as centrate for this study. The characteristics of the centrate are summarized in Table 1.

Table 1. Characteristics of centrate from anaerobic digestion of sewage sludge

Parameters	**Unit**	**Value**
NH_4^+-N	mg L^{-1}	1,180
TP	mg L^{-1}	265±4.2
VFA	mg L^{-1}	822±4.9
COD	mg L^{-1}	2340±0
Alkalinity	mg L^{-1} as $CaCO_3$	2,585±304
pH	-	8.0±0.1

HAPIX reactors setup

Wild-type algae *Chlorella* originally harvested from a local wastewater treatment plant were enriched in diluted sludge centrate for this study. Algae were settled for 24 hours, and the settled algae were collected as the inoculum for HAPIX reactors. The chlorophyll a and dry weight of algal inoculum was 81.1±2.5 mg L^{-1} and 5412 ± 159 mg L^{-1}, respectively. The algal inoculum was washed with DI water for three times to remove residuals from the diluted sludge centrate before seeding the HAPIX reactors.

Chabazite (a type of zeolite, product code: AZLB-Ca) from St. Cloud, New Mexico was sieved to obtained particle size between 1.0-1.4 mm. Chabazite was washed with DI to remove the dust residual and dried at 100°C for the study. The zeolite dosages evaluated was 150 g L^{-1}, based on isotherm and kinetics study. 500 mL of centrate was added in 1L Erlenmeyer flasks with desired amount of zeolite. Reactors were set up in the 21±2°C constant temperature room in duplicate. A shaking table set at 100 ± 10 rpm provided mixing for the reactors. After 24 hours when the IX reaction reached equilibrium, 40 mL of pre-washed algae inoculum were added into each reactor, which was recorded as time zero. The reactors were illuminated at 121±6 µmoles $m^{-2}s^{-1}$ continuously. Zeolite was always resident at the bottom of reactors due to their high density. The reactors were continuously operated until a constant NH_4^+-N concentration was observed. Algae was harvested when the total suspended solids (TSS) concentration of the algal biomass reached stationary state. Reactors with solely zeolite or algal biomass were also set up as controls.

Analytical methods

Biomass dry weight (total suspended solids [TSS]) and alkalinity were measured according to *Standard Methods* (APHA et al., 2012). COD was measured according to *Standard Methods* (5200B) using Orbeco-Hellige mid-range (0-1500 mg/L) COD kits. Total P was measured using

Hach TNT plus 845 test kits. Volatile fatty acid (VFA) concentrations were measured by the esterification method using Hach TNT plus 872 test kits. The results were reported as the equivalent concentration of acetic acid. Cation including NH_4^+, Na^+, K^+, Ca^{2+}, and Mg^{2+} concentrations were measured using a Metrohm Peak 850 Professional AnCat ion chromatography (IC) system (Metrohm Inc., Switzerland).

MATHEMATICAL MODEL DEVELOPMENT

The mathematical model developed accounts for ion exchange kinetics between co-ionic species NH_4^+ and Na^+ by surface diffusion (Auerbach et al., 2003) and algal growth under multiplicative limitation (Lee and Zhang, 2016). A conceptual model of the process is shown in Figure 1.

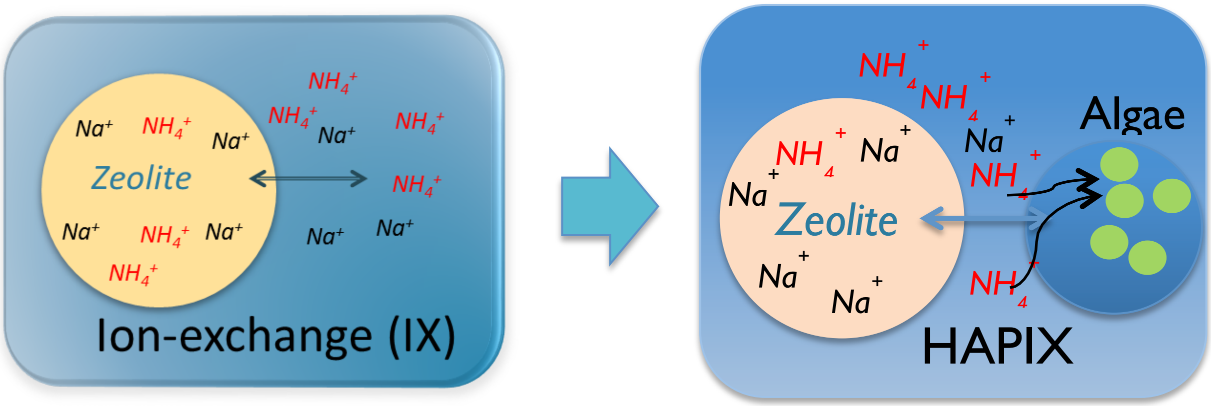

Figure 1: Schematic of the HAPIX process showing the combination of IX and algal photosynthesis.

In the HAPIX system the *main modeling assumptions* are given as follows:

1. The chabazite particles are spherical and homogenous, and the limiting diffusion process is an intraparticle surface diffusion process.
2. The IX process is such that the exchange of co-ions in the bulk liquid and solid phase is between NH_4^+ and Na^+, and there is no competition for IX sites by other cations.
3. Ion-ion and ion-solid interactions within the chabazite are ignored and Fickian diffusion is the driving mechanism for ionic migration.
4. There is dual limitation to algal growth due to light intensity and the nutrient NH_4^+.

In the aqueous phase, the NH_4^+ concentration is influenced by IX and utilization by algae, which leads to the following equation for the mass balance of the batch system:

$$\frac{dC_{NH_4^+}}{dt} = \frac{J_{NH_4^+} A}{V} - r_{NH_4^+} \tag{1}$$

where A is the chabazite particle interfacial area, V is the volume of liquid in the reactor, $C_{NH_4^+}$ is the NH_4^+ concentration in the aqueous phase, $J_{NH_4^+}$ is the mass flux of NH_4^+ ions between the bulk liquid and solid phase, and $r_{NH_4^+}$ is the rate of substrate utilization.

Several approaches have been adopted to model surface diffusion in zeolitic materials, including semi-empirical, Fickian-based, Nernst-Planck ,and Maxwell-Stefan models (Lito et al., 2012). The homogeneous surface diffusion model (HSDM) is a Fickian-based model that has been used successfully in previous studies to model ion exchange kinetics in microporous materials. The main model assumptions are that the material is homogeneous, isotropic, spherical, and that the surface diffusive flux is controlled by Fick's first law of diffusion leading to:

$$J_{NH_4^+} = -D_s \left. \frac{\partial q_{NH_4^+}}{\partial r} \right|_{r=R} \tag{2}$$

where D_S is the surface diffusivity coefficient, r is the radial coordinate in the chabazite particle, $q_{NH_4^+}$ is the NH_4^+ concentration in the solid phase, and R is the radius of chabazite particles. The mass balance for the chabazite phase based on the HSDM is:

$$\frac{\partial q_{NH_4^+}}{\partial t} = \frac{D_s}{r^2} \frac{\partial}{\partial r} \left(r^2 \frac{\partial q_{NH_4^+}}{\partial r} \right) \tag{3}$$

The initial and boundary conditions were assigned to reflect the experimental conditions. During the first phase of the experiment (24 hours), no NH_4^+ was assumed to be in the zeolite and the strictly IX occurred. In the second phase of the experimental study, both IX and NH_4^+ utilization by algae were present. The end of the first phase provided the starting point for the simulation of the second phase of the simulation period. The following equations are the initial and boundary conditions used:

$$\left\{ \begin{array}{l} q_{NH_4^+}(0 \le t \le 24hr, r) = 0 \\ q_{NH_4^+}(t > 24hr, r) = q_{hpx} \\ \dfrac{\partial q_{NH_4^+}}{\partial r} = 0, r = 0, t > 0 \\ q^{eq} = \dfrac{QKC_{NH_4^+}}{C_{Na^+} + KC_{NH_4^+}} \end{array} \right\} \tag{4}$$

where C_{Na^+} is the aqueous phase concentration of Na^+, q_{hpx} is solid phase concentration during the HAPIX phase, and Q and K are constants in the IX isotherm related to maximum adsorption capacity and affinity for the exchanger, respectively. The first equation is the initial condition, which states that initially there is no NH_4^+ ions present in the zeolite. The second equation imposes a Neumann boundary condition at the center of the chabazite particles, and the third is an IX isotherm which provides the equilibrium relationship between the NH_4^+ concentration at the zeolite surface, and the NH_4^+ concentration in the bulk liquid.

Algae have been shown to be inhibited by high NH_3-N concentrations as high as 150 mg/L. Light is one of the limiting factors to algal growth due to light attenuation in the reactor, caused by self-shading by algal biomass. We follow an approach whereby a multiplicative model is used to describe microbial kinetics when algae are exposed to an inhibitory substrate and limited light intensity conditions (Lee and Zhang, 2016). The multiplicative theory in this research assumes the algal growth rate is a function of the nutrient and light intensity, given by the Andrew's equation combined with the Chalker model:

$$\left\{ \begin{array}{l} r_{NH_4^+} = \dfrac{\mu_{\max,NH_4^+} C_{NH_4^+} X_A}{Y_A \left(\dfrac{C_{NH_4^+}{}^2}{K_{I,NH_4^+}} + K_{S,NH_4^+} + C_{NH_4^+} \right)} * \theta^{(T-20)} \tanh(I_{av} / I_k) \\ I_{av} = \dfrac{I_0}{kdX_A} 1 - \exp(-kdX_A) \end{array} \right\} \qquad (5)$$

where $\mu_{\max,NH_4^+}$ (t^{-1}) is the maximum specific growth rate, X_A (M L^{-1}) is the phototrophic biomass concentration, Y_A (dimensionless) is the yield coefficient, K_{S,NH_4^+} (M L^{-1}) is the half saturation constant, $K_{I,AOB}$ is the inhibition coefficient (M L^{-1}), θ is the Arrhenius growth coefficient (dimensionless), I_{av} (M L^{-2} T^{-1}) is the average light intensity, I_k is the light half saturation coefficient (M), k (L^2 M^{-1}) is the light attenuation rate, d is the reactor diameter, and T is temperature (T). Substitution of the rate expression (Eq. 5) into the mass balance (Eq. 1) yields the following equations for the NH_4^+-N concentration and phototrophic biomass growth rate:

$$\left\{ \begin{array}{l} \dfrac{dC_{NH_4^+}}{dt} = -\dfrac{3M}{RV} D_s \left. \dfrac{\partial q_{NH_4^+}}{\partial r} \right|_{r=R} - \dfrac{\mu_{\max,NH_4^+} C_{NH_4^+} X_A}{Y_A \left(\dfrac{C_{NH_4^+}{}^2}{K_{I,NH_4^+}} + K_{S,NH_4^+} + C_{NH_4^+} \right)} * \theta^{(T-20)} \tanh(I_{av} / I_k) \\ \dfrac{dX_A}{dt} = \dfrac{\mu_{\max,NH_4^+} C_{NH_4^+} X_A}{Y_A \left(\dfrac{C_{NH_4^+}{}^2}{K_{I,NH_4^+}} + K_{S,NH_4^+} + C_{NH_4^+} \right)} * \theta^{(T-20)} \tanh(I_{av} / I_k) Y_A \end{array} \right\} \qquad (6)$$

RESULTS

HAPIX performance

Over 95% of IX activity between NH_4^+ and Na^+ occurred within 24 hours after the addition of chabazite. The decrease in NH_4^+ concentrations eliminated the ammonia toxicity to the algae. NH_4^+-N concentration in the HAPIX reactor were reduced from 84 meq/L (1,180 mg/L) to 7.7 meq/L (107 mg/L) after adsorption for 24 hours. The addition of algae further reduced the NH_4^+-N concentration. The growth of algae after 9 days in the HAPIX reactor reduced the NH_4^+-N concentrations to 0.7 meq/L (10.5 mg/L. Figure 2a). The NH_4^+-N adsorbed by the chabazite was released to the liquid phase when aqueous NH_4^+-N was taken up by the algae. The saturated chapazite can be regenerated along with algae growth. However, the reactor without algae (IX-Control) reached steady state and no further removal of NH_4^+-N was observed after 2 days. The NH_4^+-N concentration of the IX-control was still as high as 50 mg/L, which is much higher than that of HAPIX reactor (10.5 mg/L).

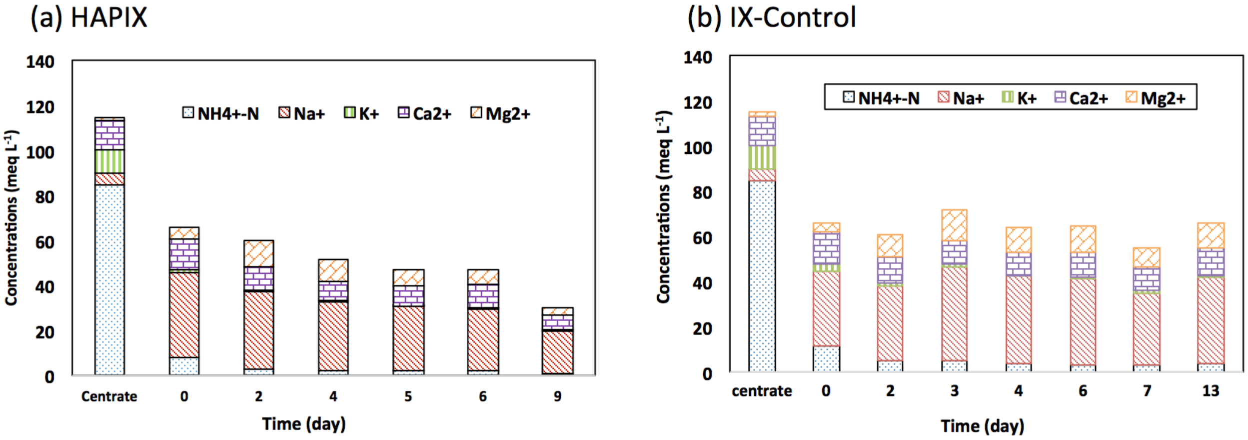

Figure 2. Cations along with time. (a) HAPIX reactor, and (b) IX-control.

Algae biomass in the HAPIX reactor increased from 700 mg/L to over 2000 mg/L after 8 days. However, the control reactor without the addition of chabazite showed an inhibition of algae growth. The HAPIX process was feasible for the treatment of high strength wastewater and produced biomass that could be used for the production of proteins, animal feed or biofuels (data not shown).

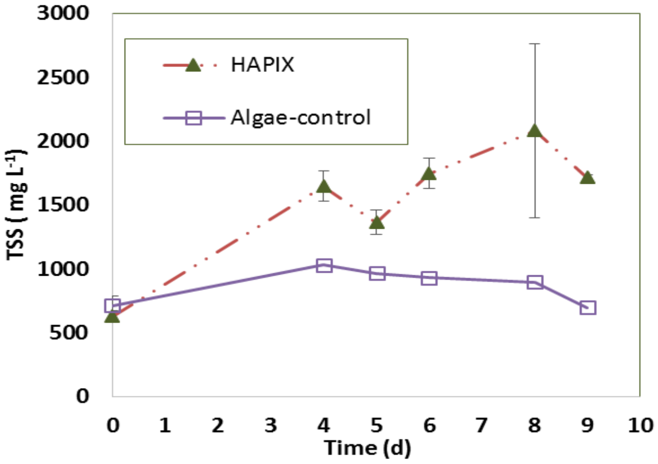

Figure 3. The dry weight of biomass of HAPIX reactor and algae-control reactor without zeolite.

IX kinetics modeling

The parameter values used in the HAPIX model were determined either by calibration or from prior studies of algal growth utilizing nutrients from wastewater, and are provided in Table 2. Figure 4 shows the IX kinetic model fit to the experimental kinetic data. The IX capacity, *Q*, and selectivity coefficient, *K*, were determined from the isotherm model in Eq 4. The IX kinetic model provides an accurate representation of the reduction in the NH_4^+ concentration showing most of the removal occurring within the first five hours of the experiment. The model prediction of Na^+ ion release from the zeolitic material is shown in the dotted red line and exhibits a faster rate of liberation than the rate suggested by the experimental data (blue squares). A likely explanation for this discrepancy between the model estimated Na^+ concentration and experimental results is the presence of other ions such as Ca^{2+}, Mg^{2+}, and K^+ that would interact with the zeolitic material; a competitive IX model was not considered but would possibly yield improved predictions.

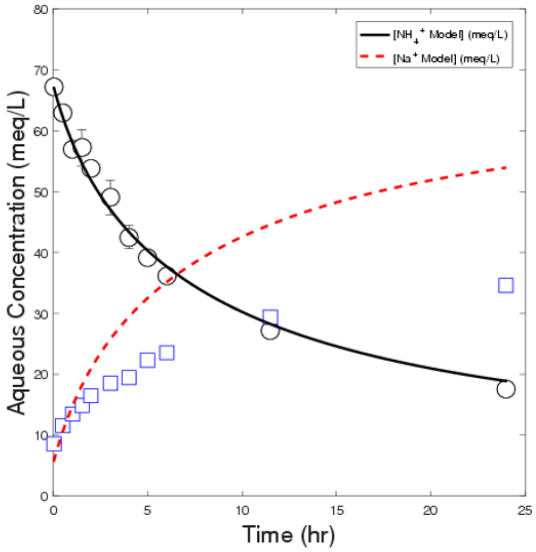

Figure 4: IX kinetics for NH_4^+ uptake onto chabazite and release of counterion Na^+.

Table 2. Table of parameters used in numerical simulation of concentration profiles

Parameter	**Definition**	**Value**
IX Model Parameters		
D_s	Surface diffusion coefficient	$5.6\text{x}10^{-13}\text{m}^2\ \text{s}^{-1}$ (fitted to surface diffusion model)
K	Selectivity coefficient	2.9 (dimensionless) (fitted to IX isotherm)
Q	Maximum ion exchange capacity	2.8 meq g^{-1} (fitted to IX isotherm)
Algal Photosynthesis Parameters		
K_I	Inhibition coefficient	364 mg-N L^{-1} (Lee and Zhang, 2016)
K_s	Half saturation coefficient	5.0 mg-N L^{-1} (Lee and Zhang, 2016)
$\hat{q}$	maximum specific rate of substrate utilization	0.89 mg-N g-VSS hr^{-1} (fitted to Andrew's equation) (Lee and Zhang, 2016)
k	Attenuation rate	0.2 m^2g^{-1} (Juneja et al., 2013)
θ	Arrhenius growth coefficient	1.35 (dimensionless) (Lee and Zhang, 2016)
I_0	Incident light intensity	100 $\mu mol\ photon\ m^{-2}\ s^{-1}$
I_k	Light saturation point	16.98 $\mu mol\ photon$ (Lee and Zhang, 2016)

HAPIX Process modeling

We used the parameters from the IX component of the process together with parameters from the algal growth model given in Table 2 and simulated the HAPIX system represented by Eq.6. The simulated temporal variation of the NH_4^+-N concentration is shown in Figure 5. The model is in good agreement with the observed concentrations, showing that most of the removal occurs within the first 100 hours of the experiment. The model provides a quantitatively accurate description of the experimental data, even though mass transfer resistance due to algal biomass growth was considered to be negligible.

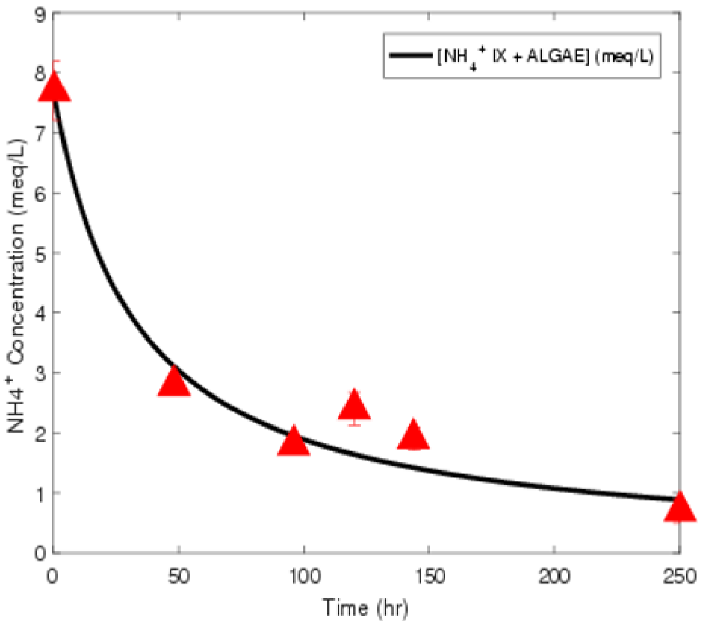

Figure 5. NH_4^+ removal due to IX and algal processes.

CONCLUSIONS

This research presents the feasibility of a HAPIX process for high NH_4^+ -N side stream treatment. A mathematical model describing the HAPIX process has been developed that accurately predicts the fate of NH_4^+ -N and algal biomass growth over time. The HAPIX process serves a two-fold purpose for removal of high strength NH_4^+ -N wastewater and the potential for biofuel, and commercial biopolymer production. Future research will focus on simulating various impacts of zeolite dose on system performance, and accounting for the impact of algal growth on the kinetics of the process.

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