



# Vacuum evaporation coupled with anaerobic digestion for process intensification and ammonia recovery: Model development, validation and scenario analysis

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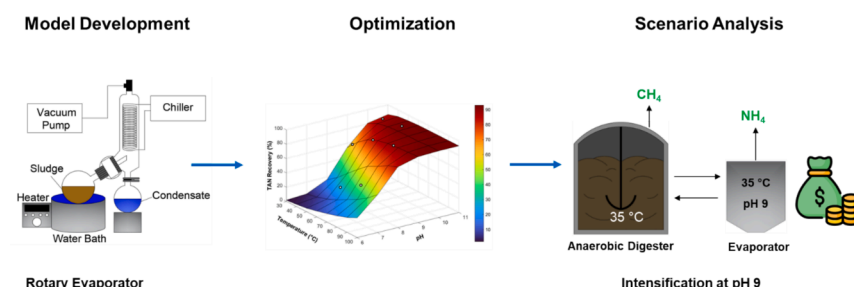
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## HIGHLIGHTS

- A mechanistic model was developed for vacuum evaporation.
- The model predicted ammonia recovery with an NRMSE of less than 10%.
- A pH increase is more effective for ammonia recovery than a temperature increase.
- Vacuum evaporation integration with anaerobic digestion improved process economics.

## GRAPHICAL ABSTRACT



## ARTICLE INFO

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## ABSTRACT

A mathematical model for vacuum evaporation process was developed, which was experimentally validated at different initial pHs and temperatures for ammonia removal from anaerobically digested sludge. Six scenarios were evaluated by combining vacuum evaporation process with anaerobic digestion using anaerobic digestion model 1. These scenarios included a control, a pretreatment by vacuum evaporation, a post-treatment by vacuum evaporation at pH 9, a post-treatment by conventional evaporation (100 °C), an intensification with vacuum-concentrated recycled digestate back to the digester, and a second intensification at pH 9. Results indicated that using the evaporator as post-treatment at pH 9 or for intensification at pH 9 were the most favorable options, recovering more than 76 % of the nitrogen present in influent sludge with no negative effect on methane production. An economic analysis showed that the intensification at pH 9 was cost-neutral, significantly higher than the net present value of the control scenario (−22 M\$).

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## 1. Introduction

A significant amount of sludge (biosolids) is produced in the treatment of wastewater. Based on the estimates of Environment Protection Agency (EPA) in 2022, approximately 3.76 million metric tons of dry sewage sludge was produced in the United States (USEPA, 2022). Anaerobic digestion (AD) is commonly applied for sludge stabilization to decrease sludge volume and minimize environmental risks associated with sludge disposal (Appels et al., 2008; Abdelrahman et al., 2023). In anaerobic digesters, ammonia toxicity at  $> 3000$  mg  $\text{NH}_4\text{-N/L}$  can limit the extent of AD intensification (Rajagopal et al., 2013). The anaerobic digestion model No. 1 (ADM1) which is commonly used for modeling AD of municipal wastewater treatment biosolids (Batstone et al. 2002) has been extended to include phosphorous, sulphur and iron transformations as well as different substrates such as food waste, agriculture waste, or other co-substrates with sewage sludge. The model has also been modified to simulate the performance of various anaerobic digesters design systems such as up-flow anaerobic sludge blanket reactor, anaerobic baffled reactors, two-phase AD, temperature-phase AD, and anaerobic membrane bioreactor (Batstone et al., 2015). It was also used to simulate intensification processes such as thermal hydrolysis pretreatment (THP) by adaptation of the disintegration rate in ADM1 from  $0.25$  to  $1.5$   $\text{d}^{-1}$  and conversion of endogenous products (Phothilangka et al., 2008).

Various ammonia recovery methods have been applied to reject water from digestate (Turker and Celen, 2007; Lorick et al., 2020) such as chemical precipitation forming struvite (Lorick et al., 2020), air and vacuum thermal stripping (Palakodeti et al., 2021), membrane distillation (Wen et al., 2021), adsorption/ion exchange (Manto et al., 2018), bioelectrochemical technologies (Zhang et al., 2021). However, many of these technologies have serious limitations such as extensive chemical addition (Ye et al., 2018), membrane fouling (Ye et al., 2018), and competing ions in ion exchange systems (Iddya et al., 2020). Ammonia stripping can recover ammonia in the form of pure ammonium sulfate, which can be an alternative to chemical fertilizer (Sobhi et al., 2020). Vacuum evaporation is a promising method to recover water and ammonia at low temperatures (Tao and Ukwuani, 2015). Under vacuum, the boiling point of water decreases, and thus water and  $> 50$  % of the digestate ammonia can be recovered without pH adjustment (Khadir et al., 2024).

Integration of vacuum evaporation with AD is a promising approach for recovery of water and ammonia, thickening sludge, and mitigating ammonia toxicity which enables high organic loading rates (OLR) (Han et al., 2022). Vacuum evaporation can be integrated in three different configurations including pretreatment to reduce digester volume (Zhang et al., 2012), post-treatment to recover ammonia and improve dewatering (Yellezuome et al., 2022), and side-stream treatment (Palakodeti et al., 2021). A side-stream configuration includes applying evaporation after digestion and recirculating the thickened sludge back to the digester to decouple solids retention time (SRT) from the hydraulic retention time (HRT), reducing digester size, mitigating ammonia toxicity (Han et al., 2022), and recovering water and ammonia.

The integration of vacuum evaporation with AD has been investigated in different studies (Ukwuani and Tao, 2016; Han et al., 2022; Okoye et al., 2022). Han et al. (2022) operated two mesophilic digesters, a control and a test digester coupled with vacuum evaporation under  $65$  °C for the treatment of a mixture of food waste and liquid dairy manure and reported that ammonia stripping improved the AD performance, allowing successful performance at an OLR of  $4.3$  g VS/(L·d) compared to  $2.5$  g VS/(L·d) for the conventional digester. Ukwuani and Tao (2016) applied vacuum evaporation to recover more than 93 % of the ammonia from a digester effluent followed by acid absorption. Okoye et al. (2022) operated a fermenter equipped with a vacuum stripper in a single treatment unit, IntensiCarb<sup>TM</sup>, at low SRT of 3 d and HRT of 1.5 d. It was reported that hydrolysis and volatile fatty acids (VFA) yields were higher compared to a control fermenter operated at

SRT/HRT of 3 d (56.4 % and 90.2 % increase, respectively). Khadir et al. (2024) used side stream vacuum evaporation for AD intensification and ammonia recovery, and reported similar volatile solids reduction ( $\sim 50$  %) at 3 times the OLR but without improvement in the methane yield, as well as 50 % recovery of the total influent nitrogen as ammonia in the condensate.

Development of a model for vacuum evaporation is important to optimize ammonia recovery and elucidate its effect on the AD process, and the economics of the wastewater treatment plant (WWTP). Previous studies have focused on modeling ammonia recovery in a vacuum evaporator from wastewater (Reza and Chen, 2021; Luqmani et al., 2024) or digestate after the AD (Ukwuani and Tao, 2016; Reza and Chen, 2022; Tao et al., 2024). Some studies only estimated the volumetric mass transfer coefficient ( $K_{La}$ ) of ammonia (Ukwuani and Tao, 2016; Luqmani et al., 2024; Tao et al., 2024) without a full process model capable of predicting pH and ammonia concentrations. However, this approach requires estimation of  $K_{La}$  for any change in pH or temperature. Furthermore, previous modeling studies have ignored carbon dioxide stripping, an essential aspect of the process leading to substantial pH increase, which then favors ammonia stripping by increasing free ammonia together. Other studies have focused on artificial intelligence models for vacuum evaporation of ammonia (Reza and Chen, 2021; Reza and Chen, 2022) which aim to optimize operational conditions for ammonia recovery from wastewater or digestate. However, these models were used primarily for modeling ammonia stripping without consideration of the impact of ammonia removal or pH change on AD performance. Although the artificial intelligence models can accurately predict the ammonia concentration and pH in the evaporated sludge, which can be useful for integration with digestion models, there are other aspects of vacuum digestion – beyond ammonia stripping that the existing models fail to consider, such as concentration of biomass, VFA production, and non-volatiles retention. Thus, there is a need for a mechanistic model to integrate the vacuum evaporation of ammonia with AD, because with evaporation, biomass, soluble chemical oxygen demand (SCOD), and VFAs are concentrated simultaneously with ammonia removal, and therefore with biomass recirculation for intensification, the potential for VFA inhibition of methanogenesis increases.

The aim of this study was to develop a mechanistic model for vacuum evaporation coupled with AD (a patent-pending process known as IntensiCarb<sup>TM</sup> process) to support scenario analysis, and model-based scale up studies, as well as a preliminary economic analysis for future full-scale applications.

## 2. Material and methods

### 2.1. Experimental methods

A laboratory-scale vacuum rotary evaporator unit (EcoChyll® X1, Ecodyst, North Carolina, USA) was used in this investigation (Fig. 1a). Anaerobic sludge obtained from a full-scale digester was placed in a 2 L flask, which was submerged in a temperature-controlled water bath. The vacuum pressure was controlled by adjusting the pressure of the vacuum pump. A chiller was used to cool extracted vapor, and the condensate was collected in a flask. Ten runs were tested under different operational conditions including ammonium concentrations (700, 1850 and 3000 mg/L), boiling points (35, 55 and 75 °C), and pH values (7.5, 9 and 10.5). For each run, 500 mL of anaerobic digested sludge was under vacuum evaporation for 2 h. Ammonium chloride and/or sodium hydroxide were added to manipulate the ammonium concentrations and pH, respectively. Selection of operational conditions was conducted using Design of Experiments (DOE) software (Stat-Ease®, Minneapolis, Minnesota, USA) (Table 1). The Student's *t*-test (two-sample assuming unequal variances) was performed to evaluate the statistical significance of differences in pH, volume reduction, and ammonia concentration between experimental test runs with a significance level (p-value) of 0.05 using Microsoft Excel 2019.

## 2.2. Model description

### 2.2.1. Anaerobic digestion model

The IWA anaerobic digestion model 1 (ADM1) was used to simulate biological reactions and production of gases during sludge treatment. The list of values of parameters were adopted from Batstone et al. (2002) (see [supplementary materials](#)). The inhibition constant for ammonia ( $K_i$ ) was considered as 0.0036 M, which is higher than the one reported in the study of Batstone et al. (2002), since the intensified AD system can handle high ammonia level more than the conventional systems.

### 2.2.2. Vacuum evaporation model

The vacuum evaporator model consists of a completely stirred tank reactor (CSTR) with a liquid volume and a sealed gas headspace, which is subjected to vacuum ([Fig. 1b](#)). ADM1 was used to define the biochemical reactions during evaporation. Temperature dependency for all equilibrium coefficients ( $K_a$ ,  $K_H$ ), was included via the first order form of the van't Hoff equation, with published enthalpy of formation values, as for the ADM1.

Similarly to ADM1, the acid-base rates of carbon dioxide ( $\text{CO}_2$ ) and ammonia ( $\text{NH}_3$ ) in the liquid phase are shown in Equations (1) and (2), respectively in a dynamic implementation:

$$\rho_{\text{CO}_2} = k_{A/B,\text{CO}_2} \cdot (S_{\text{HCO}_3} \cdot S_{\text{H}^+} - K_{a,\text{CO}_2} \cdot S_{\text{CO}_2}) \quad (1)$$

$$\rho_{\text{NH}_3} = k_{A/B,\text{NH}_3} \cdot (S_{\text{NH}_3} \cdot S_{\text{H}^+} - K_{a,\text{NH}_3} \cdot S_{\text{NH}_4^+}) \quad (2)$$

where  $k_{A/B,i}$  is the rate coefficient for the base to acid reaction ( $1/(\text{M} \cdot \text{d})$ ),  $K_{a,i}$  is the temperature dependent equilibrium coefficient (M),  $S_i$  is the concentration of the compound in the liquid (M). The increase in pH of the liquid was simulated by adding NaOH, the dissociation of which, is presented in Equation (3), where  $K_D$  is the dissociation coefficient (M):

$$S_{\text{Na}^+} - \frac{K_{D,\text{NaOH}} \cdot S_{\text{NaOH}}}{S_{\text{OH}^-}} = 0 \quad (3)$$

The rate of gas stripping including methane ( $\text{CH}_4$ ),  $\text{CO}_2$ , hydrogen ( $\text{H}_2$ ) and  $\text{NH}_3$  are given in Equations (4), (5), (6), and (7), respectively:

$$\rho_{\text{CH}_4} = K_L \cdot a \cdot (S_{\text{CH}_4} - 64 \cdot K_{H,\text{CH}_4} \cdot P_{\text{CH}_4}) \quad (4)$$

$$\rho_{\text{CO}_2} = K_L \cdot a \cdot (S_{\text{CO}_2} - K_{H,\text{CO}_2} \cdot P_{\text{CO}_2}) \quad (5)$$

**Table 1**

Operational conditions selected by DOE.

Runs	Initial ammonia concentration (mg/L)	Initial pH	Temperature (°C)	Pressure (mbar)
1	3000	9.0	75	395
2	700	9.0	75	395
3	1850	7.5	75	395
4	3000	10.5	55	158
5	1850	9.0	55	158
6	3000	7.5	55	158
7	700	10.5	55	158
8	700	9.0	35	58
9	3000	9.0	35	58
10	1850	10.5	35	58

$$\rho_{\text{H}_2} = K_L \cdot a \cdot (S_{\text{H}_2} - 16 \cdot K_{H,\text{H}_2} \cdot P_{\text{H}_2}) \quad (6)$$

$$\rho_{\text{NH}_3} = K_L \cdot a \cdot (S_{\text{NH}_3} - K_{H,\text{NH}_3} \cdot P_{\text{NH}_3}) \quad (7)$$

where  $\rho$  represents the rate of stripping,  $K_L$  is apparent mass-flux coefficient ( $\text{m/d}$ ),  $a$  is the area/volume ( $1/\text{m}$ ),  $K_{H,i}$  is the Henry's law equilibrium constant ( $\text{M/bar}$ ),  $P_i$  is the partial pressure of the gas (bar), and  $S_i$  is the concentration of the gas in the liquid (M).

The gas pressure ( $P_{\text{gas}}$ ) (bar) and flow rate ( $Q_{\text{gas}}$ ) ( $\text{m}^3/\text{d}$ ) were calculated as shown in Equation (8) and (9), respectively:

$$P_{\text{gas}} = P_{\text{CH}_4} + P_{\text{CO}_2} + P_{\text{H}_2} + P_{\text{NH}_3} + P_{\text{H}_2\text{O}} \quad (8)$$

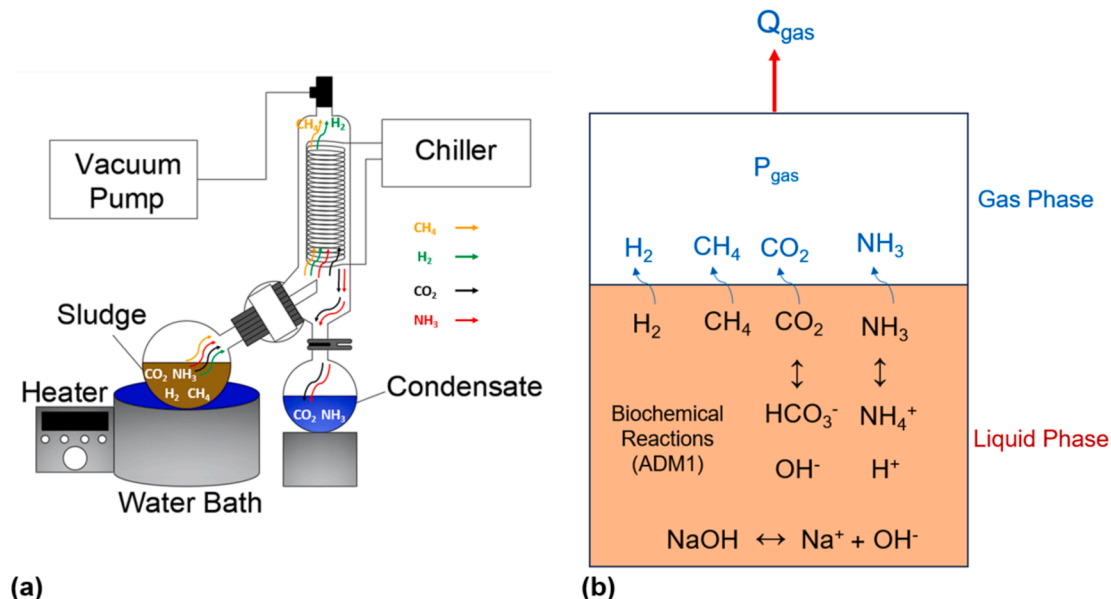
$$Q_{\text{gas}} = K_P \cdot (P_{\text{gas}} - P_{\text{vac}}) \quad (9)$$

where  $K_P$  the pipe resistance coefficient ( $\text{m}^3/(\text{bar} \cdot \text{d})$ ), and  $P_{\text{vac}}$  is the vacuum pressure used for evaporation. The values of all parameters used in this model are listed in the [supplementary materials](#) (see [supplementary materials](#)). In the evaporator, the biochemical reactions were the same as ADM1. It was assumed that there was no change in the kinetic parameters of ADM1 during the evaporation.

## 2.3. Simulation strategy

### 2.3.1. Vacuum modeling

The vacuum model was used to reproduce experimental data collected from the laboratory-scale testing. The initial pH was



**Fig. 1.** (a) Vacuum evaporator set-up; (b) Vacuum evaporation model.

manipulated by varying the NaOH concentration at the beginning of the simulation. The initial concentration of inorganic carbon was assumed as 0.21 kmole C/m<sup>3</sup>. The model, implemented in the AQUASIM v2.1, was calibrated by using only one run and validated by using the other nine runs. Ammonia stripping was validated when the normalized root mean square error (NRMSE) between the modeled and the laboratory data were less than 10 %. Following model validation, it was used to evaluate ammonia recovery under different pH (6–11) and temperatures (30–100 °C) after 2 h of stripping. The same kinetic parameters were used, while initial concentrations of total ammonia nitrogen (TAN) and inorganic carbon were assumed as 0.1 kmole N/m<sup>3</sup> and 0.21 kmole C/m<sup>3</sup>.

### 2.3.2. Scenario analysis

Six scenarios, which represented the use of the evaporator with anaerobic digester were modeled. These scenarios included:

1. Control: two conventional anaerobic digesters, in which this scenario was used as a base scenario.
2. Pretreatment: an evaporator was used as pretreatment unit before an anaerobic digester.
3. Post-treatment at 100 °C: an evaporator was used as a post-treatment unit after two digesters; the temperature in the evaporator was set as 100 °C.
4. Post-treatment at pH 9: an evaporator was used as a post-treatment unit after two digesters; 4.5 kmol NaOH/d was added to the evaporator to increase the pH to 9.
5. Intensification: An evaporator was coupled with an anaerobic digester, in which the concentrate was circulated from the evaporator back to the digester.
6. Intensification at pH 9: Same as the intensification scenario but the pH in the evaporator was increased to 9 by adding 4.5 kmol NaOH/d.

In all scenarios, the volume of each digester was 3400 m<sup>3</sup> with an influent flow of 340 m<sup>3</sup>/d. In the control and post-treatment scenarios, two digesters were used. The evaporator volume was 100 m<sup>3</sup> and the condensate flow rate was 170 m<sup>3</sup>/d in all scenarios that have an evaporator. Considering that sludge flow is about 1 % of wastewater flow (Brdjanovic et al., 2015), the influent sludge flow corresponds to a 34,000 m<sup>3</sup>/d plant. These scenarios were evaluated by using ADM1 and vacuum model in the AQUASIM v2.1 software. The model simulation used a 300-day period to achieve steady state. The total chemical oxygen demand (COD) of the influent sludge for all scenarios was ~ 57.0 kg COD/m<sup>3</sup>, distributed mainly as composite, carbohydrates, proteins, lipids and inerts (2.0, 7.5, 15, 7.5 and 25.0 kg COD/m<sup>3</sup>, respectively). The total nitrogen concentration is around 1.6 kg/m<sup>3</sup>. The ammonia and inorganic carbon concentrations in the influent sludge were 0.01 kmole N/m<sup>3</sup> and 0.04 kmole C/m<sup>3</sup>, respectively.

### 2.4. Economic analysis calculations

The daily total operational cost ( $C_T$ , in USD/d) was calculated based on energy consumption ( $E_c$ , in kWh/d), energy recovery from biogas ( $E_g$ , in kWh/d), sludge drying cost ( $C_{SD}$ , in USD/d), chemical cost ( $C_{ch}$ , in USD/d), nitrogen recovery revenue ( $R_N$ , in USD/d) and avoided costs of nitrogen removal ( $C_{NR}$ , in USD/d) as shown in Equation (10):

$$C_T = ((E_c - E_g) \cdot C_E) + C_{SD} + C_{ch} - R_N - C_{NR} \quad (10)$$

where  $C_E$  is the energy cost (0.1334 USD/kWh) (ENERGYBOT, 2023).

The  $E_c$  includes the energy required for heating the influent sludge, heat loss, mixing the digester and vacuum evaporation, as shown in Equation (11):

$$E_c = \left( \frac{Q_{\text{influent}} \cdot (T_{AD} - T_{\text{influent}}) \cdot \rho \cdot C_p}{1000 \cdot 3600} \right) + \left( \frac{A \cdot (T_{AD} - T_{\text{sur}}) \cdot U \cdot 24}{1000} \right) + (V \cdot \omega \cdot 24) + (Q_{\text{Cond}} \cdot E_{Ev}) \quad (11)$$

where  $Q_{\text{influent}}$  is the influent sludge flow rate (m<sup>3</sup>/d),  $T_{AD}$  is the digester temperature (35 °C),  $T_{\text{influent}}$  is the influent sludge temperature (20 °C),  $\rho$  is the sludge density (1050 kg/m<sup>3</sup>),  $C_p$  is specific heat capacity (4186 J/(kg·°C)),  $A$  is the digester and/or evaporator surface area (m<sup>2</sup>),  $T_{\text{sur}}$  is the surrounding temperature (20 °C),  $U$  is heat coefficient of heat transfer from walls (0.6 W/(m<sup>2</sup>·°C)),  $V$  is the volume of the digester (m<sup>3</sup>),  $\omega$  is specific power of the stirrer (0.005 kW/m<sup>3</sup>) (Tchobanoglous et al., 2014). The  $Q_{\text{Cond}}$  is the daily condensate flow rate (m<sup>3</sup>/d), and  $E_{Ev}$  is evaporator energy consumption per m<sup>3</sup> of condensate (50 kWh/m<sup>3</sup>, which was obtained from the manufacturer: <https://condorchem.com/en/blog/basis-vacuum-evaporation/>). It should be noted that the aforementioned energy consumption is for mechanical vapor recompression (MVP) evaporators which recycle the latent heat from vapor by compressing and reuse it within the system, unlike conventional evaporators.

The  $E_g$  includes the energy recovery from biogas by using a combined heat and power unit, which was calculated as shown in Equation (12):

$$E_g = Q_m \cdot CV_m \cdot E \quad (12)$$

Where  $Q_m$  is average methane daily production (m<sup>3</sup>/d),  $CV_m$  is calorific value of methane (9.7 kWh/m<sup>3</sup>),  $E$  is electricity and heat conversion efficiency (0.75) (Tchobanoglous et al., 2014).

The  $C_{SD}$  represents sludge dewatering using belt filter presses (6.16 USD/m<sup>3</sup>) (Bolzonella et al., 2018). The  $C_{ch}$  is the combined cost of sodium hydroxide (0.29 USD/kg) used for pH adjustment and sulfuric acid (0.14 USD/kg) used to recover ammonia.  $R_N$  is the revenue from the sale of the recovered ammonia as ammonium sulfate (0.24 USD/kg). The price of these chemicals was obtained from BUSINESSANALYTIC (2023). The  $C_{NR}$  represents avoided costs of removing nitrogen from the mainstream in the activated sludge system (5.16 USD/kg N) (Vineyard et al., 2020). It was assumed that the condensate was used to produce ammonium sulfate, which subject to further purification is marketable. Capital costs were calculated as the current costs of digesters (12 USD/gal) and evaporators (1.8 MUSD), which were obtained from the manufacturer (IWE Industrial Water Evaporators, Italy). Net present value (NPV, in USD) was calculated for project lifetime of 30 years as described in the study of Buller et al. (2022), and shown in Equation (11):

$$NPV = \sum_{t=1}^{t=30} \frac{-C_{t,y}}{(1+i)^t} - I_0 \quad (11)$$

where  $C_{t,y}$  is yearly total operational costs (USD),  $i$  is the interest rate (5 %),  $t$  is time period (years), and  $I_0$  represents the initial investment (USD). It should be noted that the costs for purification of ammonia and biogas treatment have not been included. The NPV was calculated for different WWTP configurations and market needs considering and ignoring the avoided costs of nitrogen removal ( $C_{NR}$ ) and nitrogen recovery benefits ( $R_N$ ) as form of sensitivity analysis.

Sensitivity analysis was conducted using the normalized sensitivity coefficient ( $S_{i,j}$ ) to define the influence of electricity price, digester cost, interest rate, ammonia revenue and sludge dewatering cost. The  $S_{i,j}$  was determined by using the method reported in USEPA (1987) as shown in Equation (12):

$$S_{i,j} = \left| \frac{\Delta Y_i / Y_i}{\Delta X_i / X_i} \right| \quad (12)$$

where  $S_{i,j}$  is the percent change in the output variable ( $Y_i$ ) to a 10 % change in the input variable ( $X_i$ ). The influence of each parameter was



interpreted as: (a) no significant influence ( $S_{i,j} < 0.25$ ), (b) influential ( $0.25 \leq S_{i,j} < 1$ ), (b) very influential ( $1 \leq S_{i,j} < 2$ ), and (d) extremely influential ( $S_{i,j} \geq 2$ ) (Eldyasti et al., 2012).

### 3. Results and Discussion

#### 3.1. Vacuum evaporation modeling

Fig. 2 shows experimental and simulated data for the remaining ammonia and pH profile during vacuum evaporation at different operational temperatures. The NRMSE for prediction of TAN concentrations and pH during vacuum evaporation was less than 10 % for all runs. The model well-predicted TAN concentrations with  $R^2$  of 0.98. The pH prediction ( $R^2$  of 0.86) was less accurate than TAN concentration estimates, which could be related to non-modeled interactions of different ions present in the sludge and/or VFA stripping. No significant

differences in pH, volume reduction, and ammonia concentration between the replicates (p-value  $> 0.05$ ) were observed. However, there was a significant difference between all test runs in pH and ammonia concentration (p-value  $< 0.05$ ), while there was no significant difference in volume reduction (p-value  $> 0.05$ ).

Fig. 3 shows the relationship between modelled TAN removal by stripping and free ammonia concentrations for all runs; the slope figure represents the volumetric liquid mass transfer coefficient ( $K_{La}$ ). It is apparent from Fig. 3 that one  $K_{La}$  describes all data regardless of temperature. While this appears to be contradictory to the widely known dependence of  $K_{La}$  in other mass transfer processes (Yellezuome et al., 2022), it should be noted that during vacuum stripping, boiling occurs at the ambient pressure, and water vapor drives ammonia stripping during evaporation (Tao et al., 2024); i.e., both the ammonia flux and water flux are impacted by pressure. Therefore, in this model,  $K_L$  was held constant (0.9 m/d) at different temperatures (boiling points).

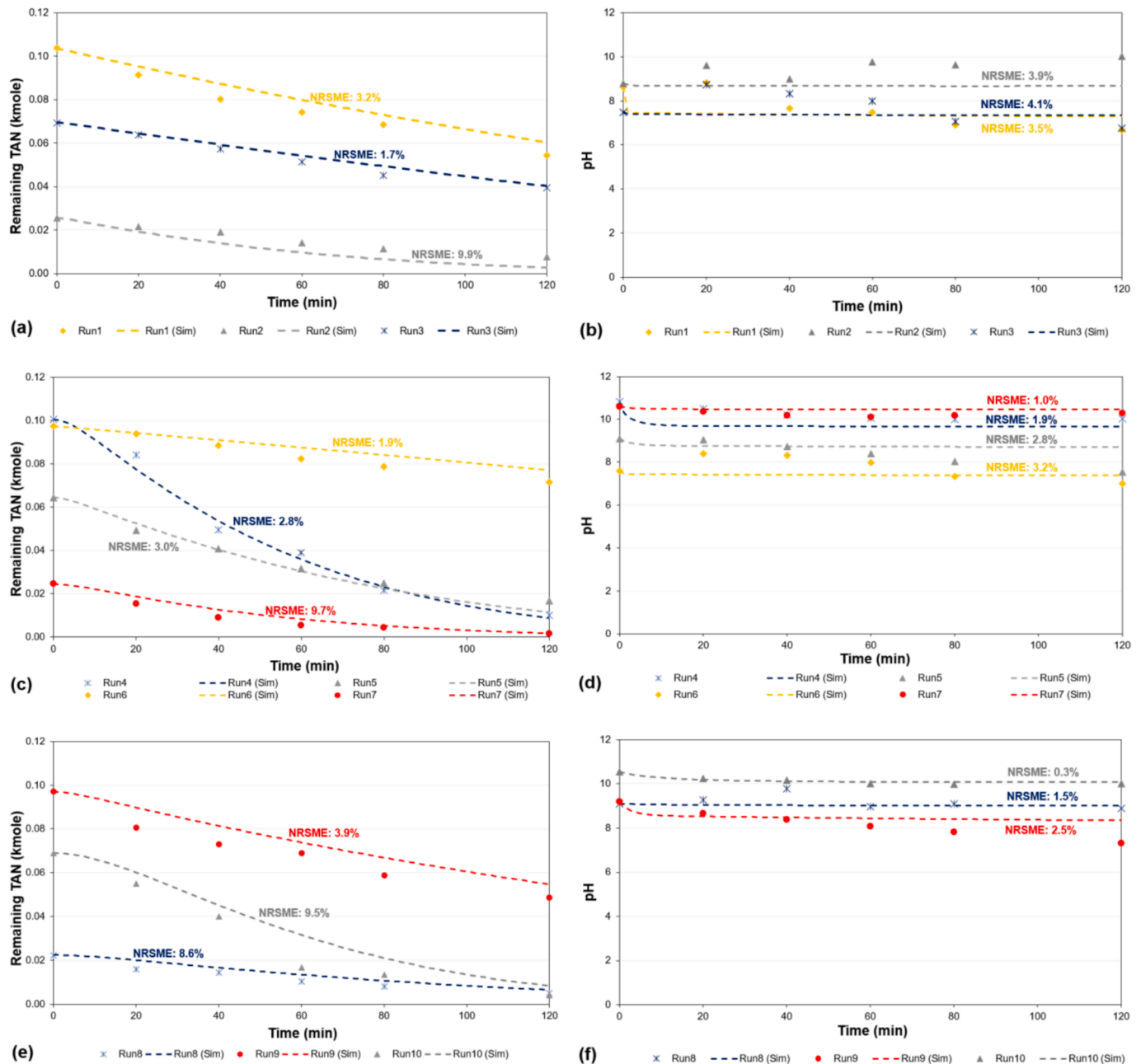


Fig. 2. Experimental and simulated data for remaining ammonia (a,c,e) and pH (b,d,f) profile during vacuum evaporation at different operational temperatures: (a) and (b) 75 °C, (c) and (d) 55 °C, (e) and (f) 35 °C.

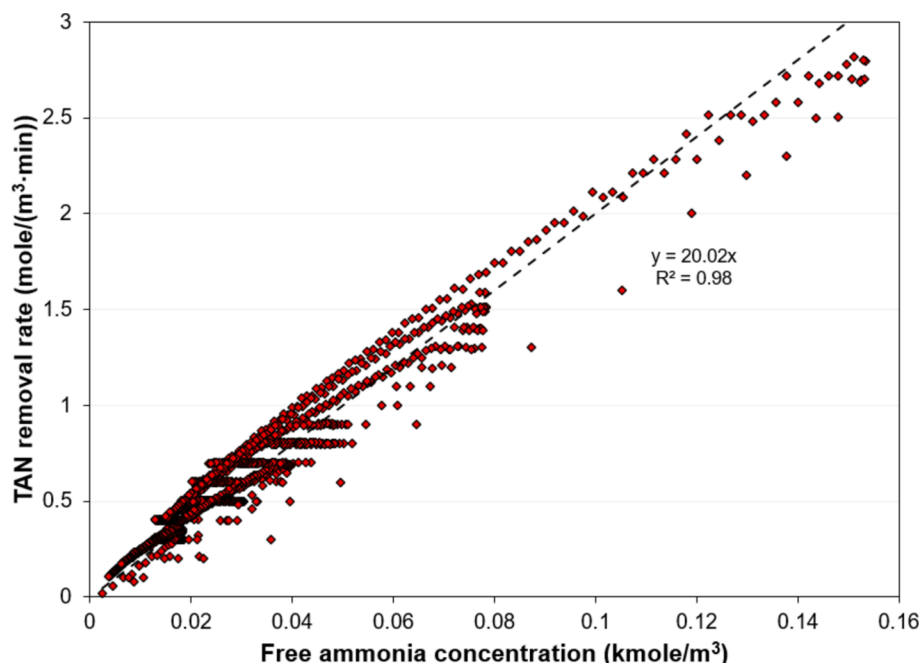


Fig. 3. Relationship between modeled TAN removal rate and modeled free ammonia concentration.

Due to the decrease in sludge volume during evaporation, the ratio of area to volume increased from 27 to 44 1/m, thus, the volumetric liquid mass transfer coefficient ( $K_{La}$ ) for all runs varied narrowly between 1.01 and 1.65 1/h. The value of  $K_L$  in this study (37.4 mm/h) was similar to those reported in the study of Ukwuani and Tao (2016) (21.8–37.3 mm/h), where the effect of increasing the pH to 9 and temperature from 50 to 100 °C was combined in the calculation of  $K_{La}$  by using the first-order rate equation only. Therefore, in this case, one  $K_L$  for ammonia is independent from temperature and pH, unlike the first-order equation. It is important to understand how ammonia can be stripped at pH of 7, even though almost all of TAN is in the form of ammonium ion ( $\text{NH}_4^+$ ) (Ho and Ho, 2012). Therefore, the model helped to fully describe the stripping mechanism. During the vacuum evaporation,  $\text{CO}_2$  was the first to be stripped since it has relatively low Henry's law constant compared to  $\text{NH}_3$ . The concentration of bicarbonate ( $\text{HCO}_3^-$ ) decreased since there is a balance between  $\text{HCO}_3^-$  and  $\text{CO}_2$  (see supplementary materials), as

described in Equation (1). Stripping  $\text{CO}_2$  increases the pH of the sludge instantaneously (see supplementary materials). This increase in pH converts  $\text{NH}_4^+$  to  $\text{NH}_3$  gas form, which can be stripped in the vacuum process (see supplementary materials). After 2 h of stripping, the pH increased from 7 to 7.3, which was the result of stripping of  $\text{CO}_2$  and  $\text{NH}_3$  gases. These modeling results are in agreement with experimental increase in pH reported by Li et al. (2016), after 15 min of vacuum evaporation.

Optimization of pH and stripping temperature is important to maximize TAN recovery. Fig. 4 shows the TAN recovery at different initial pH values and temperatures using the vacuum model at an initial concentration of inorganic carbon of 0.21 kmol C/m<sup>3</sup>. Increasing either pH or temperature led to increases in TAN recovery. At pH of 6 and 30 °C, almost no TAN recovery (1.8 %) was achieved. By increasing the temperature to 100 °C, only 28 % of TAN could be recovered at pH of 6, while increasing the pH from 6 to 11 at 30 °C led to recovery of 86 % of

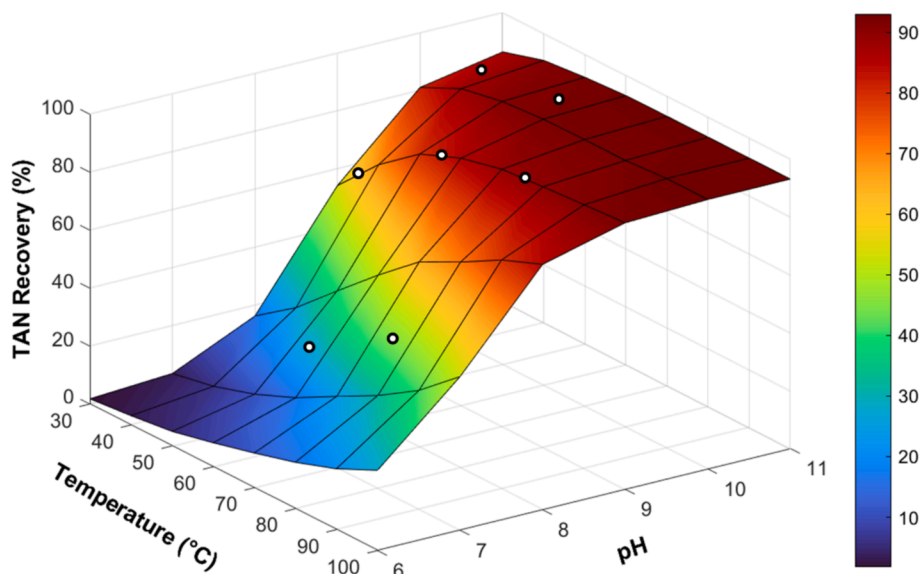


Fig. 4. Recovery of TAN at different temperatures and initial pH values. (○) represents experimental data collected in the laboratory-scale apparatus shown in Fig. 1.

TAN. Temperature change at pH of 8 had a significant effect on TAN recovery, in which increasing the temperature from 30 to 100 °C increased the TAN recovery from 16.3 % to 84.7 %. At pH of 9, more than 90 % of TAN could be recovered at temperatures of 70–100 °C, while pH had to be raised to 10 to recover more than 90 % of TAN at temperatures of 40–60 °C. Tao and Ukwuani (2015) no significant increase of TAN recovery by increasing the pH from 9 to 11 by applying thermal stripping at 100 °C, which is consistent with the model findings.

### 3.2. Coupling of vacuum evaporation with anaerobic digestion

Integration of vacuum with AD has great potential for ammonia recovery and water reuse. The mass balances of different configurations,

including pretreatment, post-treatment, and side-stream treatment (intensification), are shown in Fig. 5. The methane flow in the baseline (control) scenario was 1710 m<sup>3</sup>/d, which is a methane yield at 37 °C of 0.176 m<sup>3</sup> CH<sub>4</sub>/kg COD<sub>fed</sub>, corresponding to 44 % COD biodegradation. All the nitrogen in the influent sludge was retained in the waste sludge with no recovery (Fig. 5a). The vacuum pretreatment configuration aided in recovery of 2 kg of total nitrogen (TN) daily and concentrated the sludge prior to anaerobic digestion. Due to high total ammonia concentrations in the digester (2.7 kg N/m<sup>3</sup>), an inhibition effect is predicted which caused a significant decrease in methane yield (0.114 m<sup>3</sup> CH<sub>4</sub>/g COD<sub>fed</sub>) (Fig. 5b) and integration of vacuum unit as pretreatment step was not an efficient solution.

Application of vacuum unit as a post-treatment step showed a good

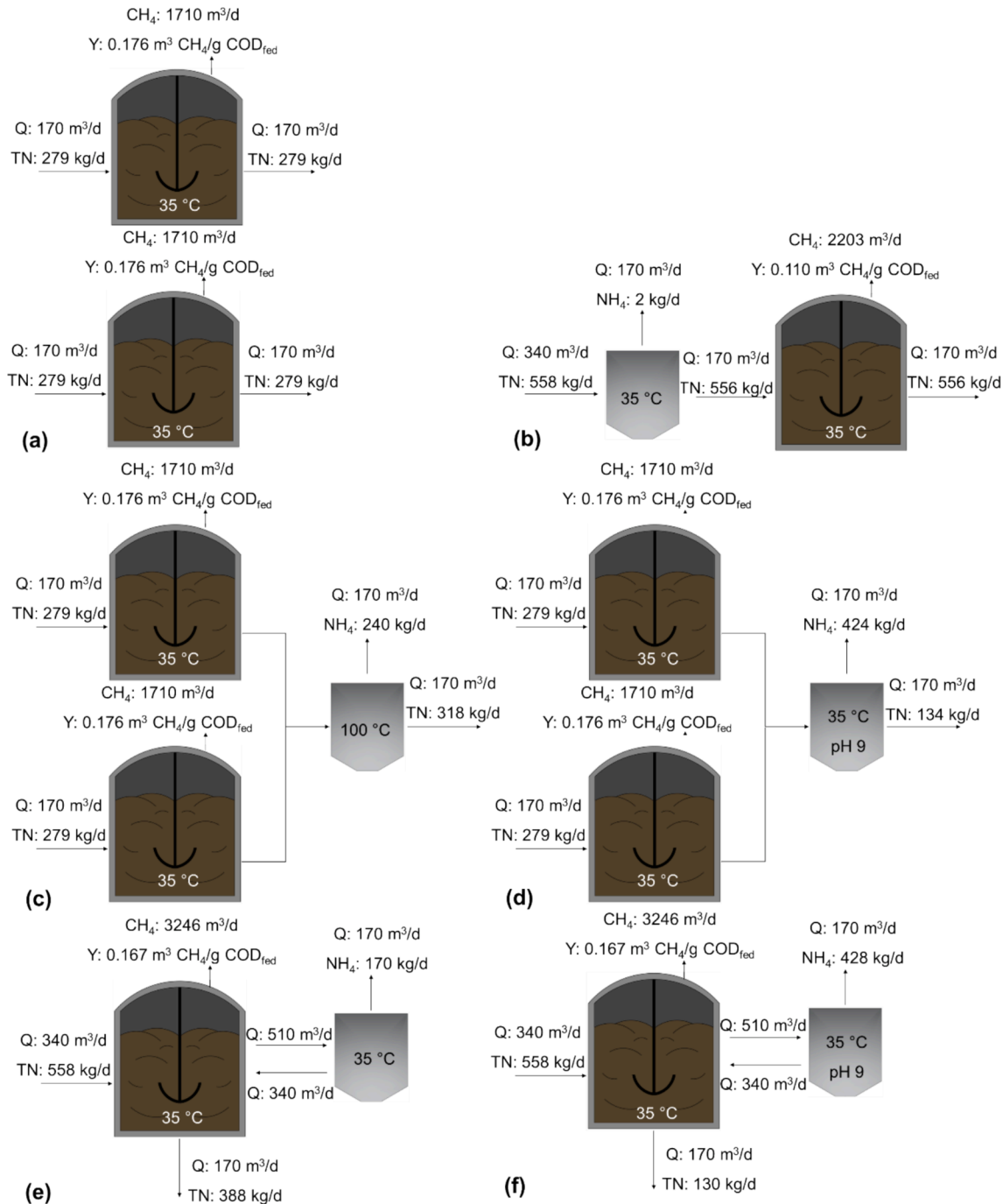


Fig. 5. Mass balance for each system: (a) control, (b) pretreatment, (c) post-treatment at 100 °C, (d) post-treatment at pH 9, (e) Intensification, (f) Intensification at pH 9.

potential for recovery of ammonia with no drawback on methane yield. Maximizing the ammonia recovery can be achieved by increasing the pH and/or temperature, as shown in section 3.1. Heating the vacuum to 100 °C recovered only 43 % of the influent nitrogen (Fig. 5c). While ~76 % of influent nitrogen was recovered when pH in the vacuum unit was adjusted to 9 (Fig. 5d), resulting in 99 % of the proteins being degraded in the digester. Increasing the pH appears to be more effective than increasing temperature. These results are consistent with work reported by Tao et al. (2018), in which increasing the pH of sludge to 9, yielded ammonia recovery in the range of 49–96 %.

A side-stream configuration includes recirculating sludge after vacuum treatment back into the digester, thus decoupling the HRT and SRT (a patent-pending process known as IntensiCarb™). In this scenario one digester was used versus two digesters in the control scenario for the same influent flow. The methane flow was 3246 m<sup>3</sup>/d, corresponding to a methane yield of 0.167 m<sup>3</sup> CH<sub>4</sub>/g COD<sub>fed</sub>. By coupling vacuum with digestion, ~30 % of the influent nitrogen could be recovered (Fig. 5e). To maximize nitrogen recovery from this configuration, pH of the vacuum unit was increased to 9, which led to an increase in nitrogen recovery (77 %). Ammonia concentrations in the digester decreased, reaching ~1.0 kg/m<sup>3</sup>, leading to a similar yield to 0.167 m<sup>3</sup> CH<sub>4</sub>/g COD<sub>fed</sub> (Fig. 5f). Moreover, the nitrogen recovery in this system was higher than the 47.5 % reported by Abdelrahman et al. (2022) using an

anaerobic membrane bioreactor (AnMBR) for primary sludge treatment. Additionally, the condensate would not have other impurities (ions) such as metals, as in the permeate of AnMBR (Ramírez et al., 2020). The stripped methane in the evaporator was less than 1 % of the flow in the digester in all scenarios. More information regarding concentrations of ammonia and VFA in each scenario is summarized in the supplementary materials.

Evaluating the economic feasibility of a vacuum system is important to informing engineering design and operational considerations for a full-scale integrated system. Capital costs and daily operational costs for each scenario are summarized in the supplementary materials. Fig. 6a shows the NPV of each scenario including the contribution of costs and revenues to the NPV considering ammonia recovery and removal benefits. The capital cost of the evaporator was 85 % lower than the digester cost. Thus, the capital costs in pretreatment and intensification scenarios (one digester) were 40 % lower than the capital costs of the control and post-treatment scenarios (two digesters). In all scenarios, the revenues from the recovered ammonia and chemical costs were the least contributors to total revenues and costs, respectively, accounting for 2 %–7% and 1 %–4%, respectively. The NPV of the control was –22 M\$, considering energy, sludge dewatering and capital costs were much higher than value of the recovered energy, denoted as energy revenue in Fig. 6a.

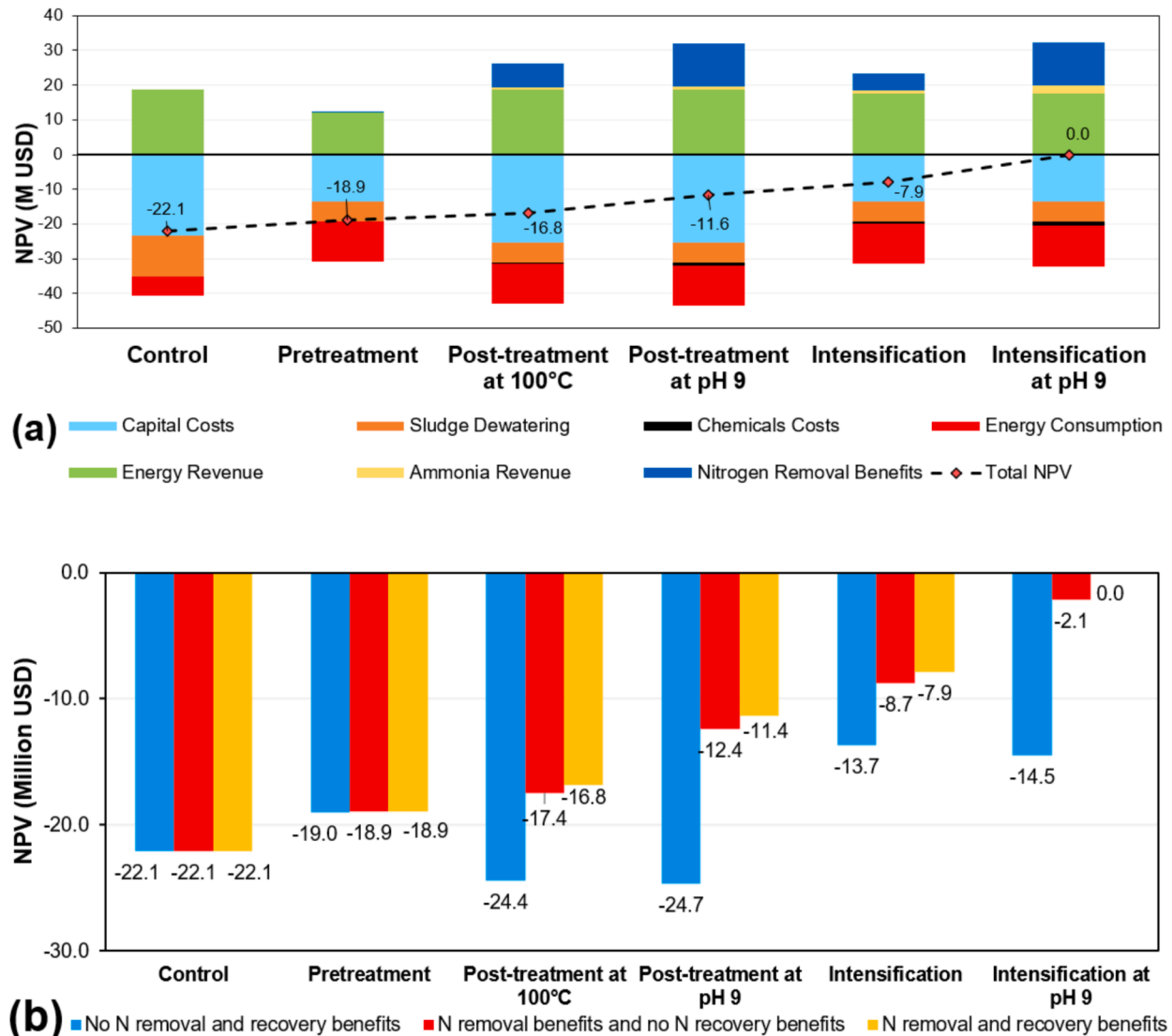


Fig. 6. (a) NPV of each scenario, (b) NPV of each scenario for different configurations.



Net energy revenues in all other scenarios were lower (46 %–54 %) than the control because of additional energy requirements of the vacuum unit. In addition, the lower digestate volume in scenarios with vacuum unit had lower sludge dewatering costs than the control. Although the pretreatment scenario had the lowest energy and nitrogen recovery, the NPV was less than the control. This improvement was because of lower capital costs, and reduced sludge volumes for dewatering. Due mainly to the benefits of diverting the nitrogen from the mainstream, the NPV of post-treatment scenarios were better than the control, achieving savings of 5.3–10.5\$M.

Using a vacuum unit as a post-treatment at pH of 9 had better NPV than 100 °C, because more ammonia was recovered at pH of 9. However, costs were higher than the revenues in both scenarios. The capital costs of intensification scenarios, where only one digester was required, were much lower than the control and post-treatment scenarios. These results show the value of this integration for cost reduction even at neutral pH. Intensification at pH of 9 was the only scenario that reached cost neutrality. Using vacuum seems to be the best option for intensification, compared to ammonia recovery by air stripping that may cause inhibition to the AD process due to oxygen. For plants, with sufficient digestion capacity, the NPV of intensification can be improved by co-digesting another substrate together with the sludge such as food waste (Koch et al., 2016), with no need for addition of another digester.

Three types of wastewater treatment plants were considered: a- a plant that has no ammonia nitrogen removal requirements; b- a plant that has ammonia nitrogen limits but uninterested in ammonia recovery, and c- a plant that has ammonia limits and interested in ammonia recovery. Fig. 6b shows the NPV of each scenario for the three different WWTP (a-c). Because there was almost no nitrogen removal (0.4 %) in the pretreatment scenarios, the NPV was the same for all configurations. Post-treatment scenarios seem to be not feasible if nitrogen removal avoided costs and recovery benefits were not considered, in which their NPV was higher than the NPV of the control. The NPV of intensification scenarios were lowest for all WWTP configurations due to lower capital costs in comparison with other scenarios. This is primarily due to the lower evaporator capital cost relative to the digester. Even for plant a, with no ammonia limits, and no consideration of ammonia recovery, an \$8M reduction in NPV can be achieved, relative to the control.

Electricity price, digester cost, interest rate, ammonia revenue and sludge dewatering cost are the most important parameters in the economic analysis. Thus, sensitivity analysis is important to understand the effect of each parameter on the NPV for each scenario. Digester cost was the most influential parameter, in which sensitivity coefficients in most of scenarios were > 1 (very to extremely influential) (see [supplementary materials](#)). For all scenarios except intensification at pH of 9, sensitivity coefficients of electricity price, interest rate, ammonia revenue and sludge dewatering cost were mainly less than 1 (no significant influence to influential). The sensitivity coefficient of all parameters for intensification at pH of 9 was more than 2 (extremely influential). This high value coefficient was due to the low NPV value of intensification at pH 9, in which a 10 % increase in the parameters' values changed the NPV significantly (–75 % to 200 %), thus, the sensitivity coefficient was more than 7 for most of the parameters. However, even considering the highest sensitivity coefficient for intensification at pH 9 i.e. 15.6 for digester cost, a 10 % increase in digester cost would change the NPV for plants a-c, from \$-14.5 M, \$-2.1 M, and \$0.0 M to \$-15.7 M, \$-3.3 M and \$-1.2 M, which for the worst-case scenario (plant a), would still reduce NPV by 34 % compared to the control.

#### Model limitations

The model developed herein provides a better mechanistic understanding of vacuum evaporation and its integration with AD. The behavior of ammonia stripping was well predicted with this model; however, pH prediction was not as accurate. This could be related to interactions of different ions present in the sludge and/or VFA stripping, which are not considered in the model. Therefore, accurate pH prediction requires measurement of ion concentrations in the sludge and

monitoring VFAs during stripping (Ren et al., 2012; Li et al., 2015). Vacuum evaporation may have an effect on hydrolysis of organics; Okoye et al. (2022) reported an increase in hydrolysis and VFA yields by coupling a fermenter and vacuum. Additionally, biomass activity may be affected by vacuum, which should be further investigated. Thus, it is important to simulate long-term operations of a digester coupled with vacuum unit. It is also noteworthy that this model was validated based on a laboratory-scale system, where ammonia behavior may differ from larger scale systems, with different surface-to-volume ratios.

## 4. Conclusions

A mathematical model for vacuum evaporation was developed, and experimentally validated for ammonia recovery from anaerobically digested sludge, with NRMSE of < 10 %. The model after integration with anaerobic digestion model 1 was used to economically evaluate pretreatment, post treatment, and anaerobic digestion intensification scenarios. Evaporation pretreatment was the worst for ammonia recovery and methane production while intensification at pH 9 was the best, with recovery of > 76 % of the influent nitrogen and no inhibition of methanogenesis. The integrated vacuum anaerobic digestion system showed significant economic improvement, with NPV of intensification at pH 9 significantly higher than the control.

### CRedit authorship contribution statement

**Amr Mustafa Abdelrahman:** Writing – original draft, Visualization, Software, Methodology, Investigation, Formal analysis, Conceptualization. **Ali Khadir:** Writing – original draft, Methodology, Investigation, Conceptualization. **Domenico Santoro:** Writing – review & editing, Supervision, Conceptualization. **Eunkyoung Jang:** Project administration. **Ahmed Al-Omari:** Writing – review & editing. **Chris Muller:** Writing – review & editing. **Katherine Y. Bell:** Writing – review & editing. **John Walton:** Writing – review & editing. **Damien Batstone:** Writing – review & editing. **George Nakhla:** Writing – review & editing, Supervision, Project administration, Methodology, Funding acquisition, Conceptualization.

### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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### Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.biortech.2024.131753>.

### Data availability

No data was used for the research described in the article.

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