NM WRRI Student Water Research Grant

Progress Report

1. **Student Researcher:** Isuru Sachitra Abeysiriwardana Abeysiriwardana Arachchige
   Faculty Advisor: Dr. Nagamany Nirmalakhandan

2. **Project title:** Anaerobic Digester Supernatant Treatment Using Algal Systems

3. **Description of research problem and research objectives.**

Typical domestic wastewater treatment plants use the aerobic activated sludge (AS) process for removing dissolved organics to the discharge standards. One of the drawbacks of the AS process is its high energy demand of 0.3-1.89 kWh/m^3 of sewage treated [1]. As a means of offsetting a portion of this energy, anaerobic digestion (AD) is practiced in medium to large-sized plants to generate methane from the waste sludge from the AS process. According to Water Environment Federation database, 1286 of the 14,748 STPs in the US incorporate AD to convert waste sludge to methane [2]. Following the digestion process, the "stabilized" sludge is typically dewatered and the solid fraction is either incinerated, used as fertilizer or, disposed of in landfills [3]; the nitrogen-rich liquid fraction (AD supernatant) is often returned to headworks of the treatment plant [4,5].

The practice of retuning the AD supernatant has been a concern since it increases the nitrogen (N) load (up to 30%) of treatment plant. As a remedy to this problem, researchers have proposed systems such as Anammox-based processes to treat AD supernatant before recycling to the headworks [6]. But, most of these novel processes dissipate the valuable forms of N in the AD supernatant as gaseous products without any recovery. In this project, it is hypothesized that by utilizing the AD supernatant to cultivate algae, recycling to the headworks can be avoided while recovering a good portion of the nutrients incorporated into the algal biomass for beneficial use.

Our previous reports [7] have detailed the development of a novel algal-based wastewater treatment (WWT) system using a mixotrophic alga, *Galdieria sulphuraria*. Long-term stable operation of this system has been demonstrated at the Las Cruces Wastewater Treatment Plant (LC WWTP). Results from our previous field studies have confirmed that this system can reduce the Biochemical Oxygen Demand (BOD), Nitrogen (N), and Phosphorus (P) in primary effluent well below the discharge standards in a single step, within 2-3 days of operation. The recoveries of energy and nutrients from the produced algal biomass, respectively as biocrude and struvite, have also been demonstrated [8–10].

Expanding our scope, here, we tested the treatment of AD supernatant to remove its pollutant contents using the novel algal-based wastewater treatment system. Among many algal biomass-to-energy conversion processes reported in the literature, Feng et al. [8] have identified hydrothermal liquefaction (HTL) as the most energy-favorable process. As such, biomass produced from AD supernatant treatment was processed to extract energy under the optimum HTL conditions (350ºC, 20% solid content and 60 mins holding time) listed in our recent report [10]. Using data from our filed and ongoing studies, nutrient recoverability from byproducts (aqueous phase and biochar) of the HTL process as struvite was assessed. Finally, nutrient and energy flow through the proposed integrated algal system were analyzed to assess the impact of its implementation on the overall wastewater treatment plant performance.

4. Description of methodology employed

4.1 Algae cultivation

The proposed study was conducted in a 700-L photobioreactor deployed at the Las Cruces Wastewater Treatment Plant. The culture in the reactor was mixed and kept in suspension using a paddlewheel. The reactor was covered with a translucent plastic sheet to control evaporation/contamination and to enable the capture of solar heat to maintain above-ambient temperatures. Figure 1 demonstrates a schematic diagram of the 700L photobioreactor used in this study.

The system was operated in a fed-batch mode with a working volume of 700L comprising 400L of wastewater feed and 300L of preadapted culture. A mixture of primary effluent and AD supernatant served as the wastewater feed. In this study, four AD supernatant:primary effluent ratios were tested; 10:90; 15:85; 20:80; and, 25:75. Each fed-batch cycle was ended when reactor contents reached the discharge standards for all three primary pollutants: ammoniacal nitrogen- 10 mg/L; phosphate- 1 mg/L; and 5-day BOD – 30 mg/l. The paddlewheel was then turned off allowing the cultures to settle down for 24 hours. Thereafter, the supernatant was discharged (V_E) and the reactor was replenished with a fresh batch of 400L of feed to start the next cycle. Four such consecutive fed-batch cycles were completed (one for each ratio). After discharging the supernatant, reactor was mixed, and a portion of the algal biomass was harvested (V_H) such that $V_E + V_H$ = 400L. The harvested algal cultures were centrifuged and processed further as explained in section 4.2.

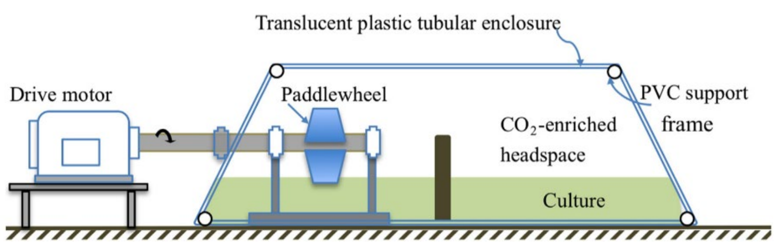

Figure 1: Schematic representation of the 700L photobioreactor deployed at the Las Cruces Wastewater Treatment Plant.

During this study, the biomass concentration in the reactor was measured daily as OD750 using a DR 6000 spectrophotometer (Hach, Colorado). Concentrations of phosphate, ammoniacal nitrogen, and nitrate-nitrogen were measured daily as well, following salicylate method 8155, phosver3 method 8048 and cadmium reduction method 8039, respectively; BOD_5 was measured once in 2 days following the standard method 5210B. The pH of the system was recorded daily using a Fisher Scientific® accumet® AE150 pH benchtop meter.

4.2 Hydrothermal liquefaction of algal slurry

Two HTL runs of the algal slurries (500ml) were performed in a 1.8L batch reactor (Parr Instrument Company, Moline, Illinois) under the following conditions: temperature of 350ºC, solid content of 20% (100g algae) and, holding time of 60 mins. The HTL process yields 4 products- energy-rich biocrude oil; N-rich aqueous phase; P-rich biochar; and off-gases. Procedures for processing the biocrude oil from the HTL products have been described elsewhere [8].

Samples of the aqueous phase (AP) resulting from the HTL step were analyzed for its N and P contents. Analyses of TN, NH_3-N, and PO_4^{3-}-P followed the TNTplus® 828- method 10208, salicylate method 10031, and Phosver3 Method 8048, respectively (Hach, Loveland, Colorado). To quantify the total phosphorus contents, char samples (0.25 g) were digested in 3 mL of 30% HCl, and 6 mL of 70% HNO_3 in a Multiwave 3000 microwave digestion system (Anton Paar, Ashland, VA). Digestates of biochar samples were diluted to 100 mL with deionized (DI) water. The total phosphorus content of these digestates and aqueous phase samples were measured using an Optima 4300 DV inductively coupled plasma optical emission spectrophotometer (ICP-OES) (PerkinElmer, Waltham, MA). Higher heating values (HHV) of the feedstocks, biocrude (heavy biocrude and light biocrude oil) and chars were measured using a model 6725 semi-micro bomb calorimeter (Parr Instrument Co., Moline, IL).

4.3 Mass and energy flow analysis

A mass flow diagram was developed considering the AD supernatant flow of the local wastewater treatment plant (95.38 m^3/d) to assess the fate of N and P. Operational data of the pilot-scale system, HTL and ongoing struvite precipitation experiments and, data from reports [9,11] were used to complete the mass balance for the proposed integrated algal system.

The raceway-type bioreactor in the pilot-scale algal system was fitted with off-the-shelf paddlewheel assemblies that are not optimized for the reactor volume. As such, energy input in the algal system was estimated assuming the power required to keep algal biomass in suspension (3.9 W/m^3) for full-scale high rate algal ponds (HRAP)[12]. The ambient temperature for energy calculations was assumed as 25ºC. Estimates of energy input for heating considered the initial energy required to raise the temperature of all elements of the process streams as well as the energy losses due to conduction. Heat loss from radiation was considered in the HTL process where the operating temperature is >300^{0}C. Convectional heat losses were assumed negligible. Heat recovery from HTL-products was assumed as 50% [8].

Since most of the phosphorus partitioned into biochar, a P-extraction process was developed using 0.5M sodium hydroxide at a solid content of 5%; temperature of 60ºC; and holding time of

3 days. Solar heating was assumed to serve the energy needs to raise the temperature for the P-extraction process. Wastewater flow through the plant was assumed to be under gravity except for internal recirculation and return activated sludge flow.

5. Description of results; include findings, conclusions, and recommendations for further research.

5.1 Anaerobic digester supernatant treatment

Characteristics of the AD supernatant and primary effluent used in this study are tabulated in Table 1. As can be noted, nutrient content in AD supernatant is much higher than that in primary effluent; BOD contents, however, were similar. It should be noted that the lack of biodegradable carbon in the AD supernatant impairs conventional heterotrophic bacteria-based treatment approaches without an external carbon source. This further emphasizes the need for using a mixotrophic algal species in AD supernatant treatment since it can adjust the carbon requirement by utilizing inorganic carbon from the atmosphere.

Table 1: Characteristics of AD supernatant and primary effluent used for the study

Property	Value		Unit
	AD supernatant	**Primary effluent**	
Ammonia nitrogen	265.01	23.14	mg/L
TN	315.33	31.00	mg/L
Nitrate	6.86	4.53	mg/L
Phosphate	33.33	6.88	mg/L
TP as phosphate	38.06	7.45	mg/L
BOD	34.17	34.25	mg/L
COD	272.67	127.33	mg/L
TOC	288.70	30.91	mg/L
pH	8.45	7.43	
Alkalinity	972.00	292.00	mg/L as CaCO3
Conductivity	4.11	1.55	mS/cm

Figure 2 shows the OD750 profile in the reactor during the study period. Here, concentration profile between days 0-5, 6-13, 14-21 and 22-29 corresponds to cycles having AD supernatant compositions of 10%, 15%, 20%, and 25%, respectively. As can be noted, consistent growth of algal biomass could be observed regardless of the AD supernatant composition.

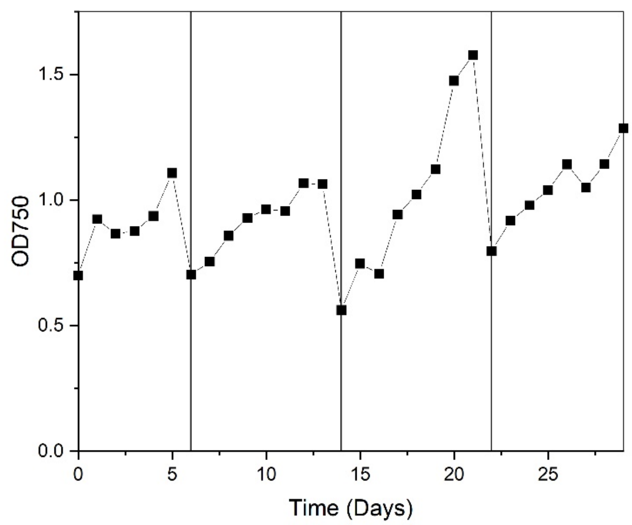

Figure 2: Temporal variation of OD750. AD supernatant compositions used were 10%, 15%, 20% and 25% respectively between 0-5, 6-13, 14-21 and 22-29 days.

Figure 3 presents the fate of the primary contaminants during these tests: a) ammoniacal nitrogen, b) nitrate-nitrogen, c) five-day biochemical oxygen demand (BOD) and, d) phosphate. As can be noted from Figure 3a, the algal system consistently reduced the ammoniacal nitrogen concentration below the discharge limit (10 mg/L) within 7 days of operation, except in the final cycle (25% AD supernatant). The average volumetric ammoniacal nitrogen removal rate calculated for AD supernatant and primary effluent mixed cycles (3.21±0.95 mg/L-d) was not significantly different compared to the control cycles (100% primary effluent) at a significance level of 0.05. Consequently, the higher ammonia load in AD supernatant cycles required a longer cycle time to reach the set standard, as opposed to the control cycles.

Since an acidic pH is maintained in the system, the possibility of removing ammonia through volatilization is minimal (P_{ka} of $NH_3 \rightleftharpoons NH_4^+ = 9.25$). Hence, a majority of ammoniacal nitrogen removal in the algal system could be attributed to algal uptake [13]. This hypothesis has also been validated through a mass balance approach in our recent report [10]. However, a consistent reduction of nitrate-nitrogen concentration could not be observed in this study (Figure 3b). As reported in the literature [14], this may be due to 1) the higher preference of algal for ammonia over nitrate and, 2) inhibition of nitrate uptake at high ammonia concentrations.

The algal system reached the discharge standard for phosphorus (1 mg/L) in all cycles (Figure 3d). The average volumetric removal rate of phosphorus (VVR_{PO4} = 1.55 ±0.48 mg/L-d) is significantly higher compared to control cycles at a significance level of 0.05. The removal of phosphorus by forming precipitates with cations such as calcium is minimal at the low pH in this algal system. Hence, the removal mechanism of phosphorus in the algal system can predominantly be due to algal uptake [13]. As before with ammoniacal nitrogen, this hypothesis has also been validated in our recently published work [10]. It should also be noted that relatively slower removal rates of both phosphorus and ammoniacal nitrogen contributed in determining the overall cycle time in the current study; in control cycles, however, relatively slower phosphorus removal rate alone determined the overall batch processing time [15].

As can be noted from Figure 3c, the algal system reached the discharge standard for BOD (30 mg/L) in under 2 days of operation. The average volumetric removal rate of BOD (VVR_{BOD}) (7.59±3.97 mg/L-d) during these experiments was significantly higher than that in control cycles (100% primary effluent) at a significance level of 0.05. The higher volumetric removal rate of BOD achieved in the current study may be due to the higher demand for carbon from to higher nutrient availability.

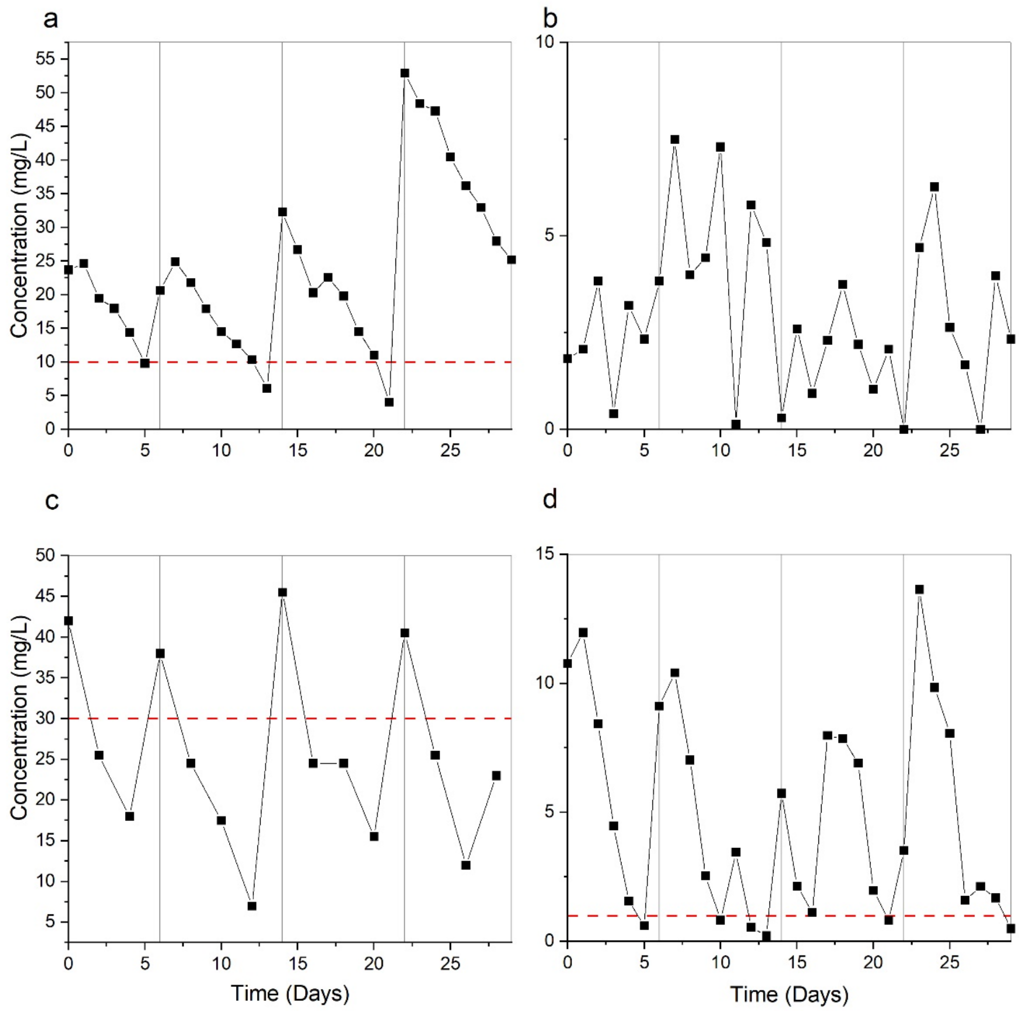

Figure 3: Concentration profiles of a) ammoniacal nitrogen, b) nitrate-nitrogen, c) BOD and d) phosphate in the reactor. The AD supernatant compositions used were 10%, 15%, 20% and 25% respectively between 0-5, 6-13, 14-21 and 22-29 days. The red dash-dot line represents the corresponding discharge standards.

5.2 Hydrothermal liquefaction

Hydrothermal liquefaction of algae yields four products: biocrude oils (light, LBO and heavy, HBO), biochar, gaseous products, and an aqueous phase. Duplicate runs in this study converted 7.75±1.30%, 10.8±2.53 and 22.16±7.71% of dry algae into light biocrude oil (LBO), heavy biocrude oil (HBO) and biochar, respectively. The oil yield in this study is somewhat lower compared to that reported by Li et al (2019) [11] under similar HTL conditions for *Galdieria sulphuraria* from 100% primary wastewater treatment (LBO+HBO=21.4%). Yet, the energy contents of both LBO (38.76 MJ/kg) and HBO (37.18 MJ/kg) in the current study were higher than those reported by Li et al (2019) [11] (34.90 MJ/kg).

Ammoniacal nitrogen (NH3-N), total nitrogen (TN), phosphate phosphorus (PO4-P) and total phosphorus (TP) contents of the aqueous phase were 11,303.3 mg/L, 12,952.3 mg/L, 4.5 mg/L and 9.6 mg/L, respectively. The NH3-N/TN ratio of 87% in this study, is much higher than 59% reported in the study by Li et al (2019) [11]. This warrants a higher N recovery from the aqueous phase in our parallel study using gas-permeable membranes. As seen in previous studies [8,11] most of the phosphorus in algae partitioned into biochar (36.5 g/kg).

5.3 Mass and energy flow

The LC WWTP handles a flow rate of 6.7 million gallons of sewage per day (25,329.3 m^3/d). With the current process layout, a return flow of AD supernatant at a flowrate of 95.4m^3/d is mixed with the sewage feed at headworks. Figure 4 demonstrates nutrient input to the secondary treatment process in this plant with and without the implementation of the proposed algal system for AD supernatant treatment.

As can be noted, the N and P load of the mainstream treatment process is reduced by 4.4% and 2.5% through the proposed side stream treatment process; and the mainstream flowrate is reduced by 1.9%. It should, however, be emphasized that the local wastewater treatment facility doesn't incorporate dedicated systems for nutrient removal. This leads to a lesser accumulation of nutrients in wasted sludge and therefore, lesser concentrations of nutrients in AD supernatant. Our previous study [10] substituting the prevailing secondary treatment system of the local wastewater treatment plant with an anaerobic/anoxic/aerobic (A2O) process identified that a dedicated nutrient removal process could increase nitrogen and phosphorus content of AD supernatant respectively to 7.4% and 13.2% of the total plant influent nutrient load. Consequently, implementing the proposed algal AD supernatant treatment approach on these conditions could reduce the N and P load of the mainstream treatment process at least by 7.4% and 13.2% respectively.

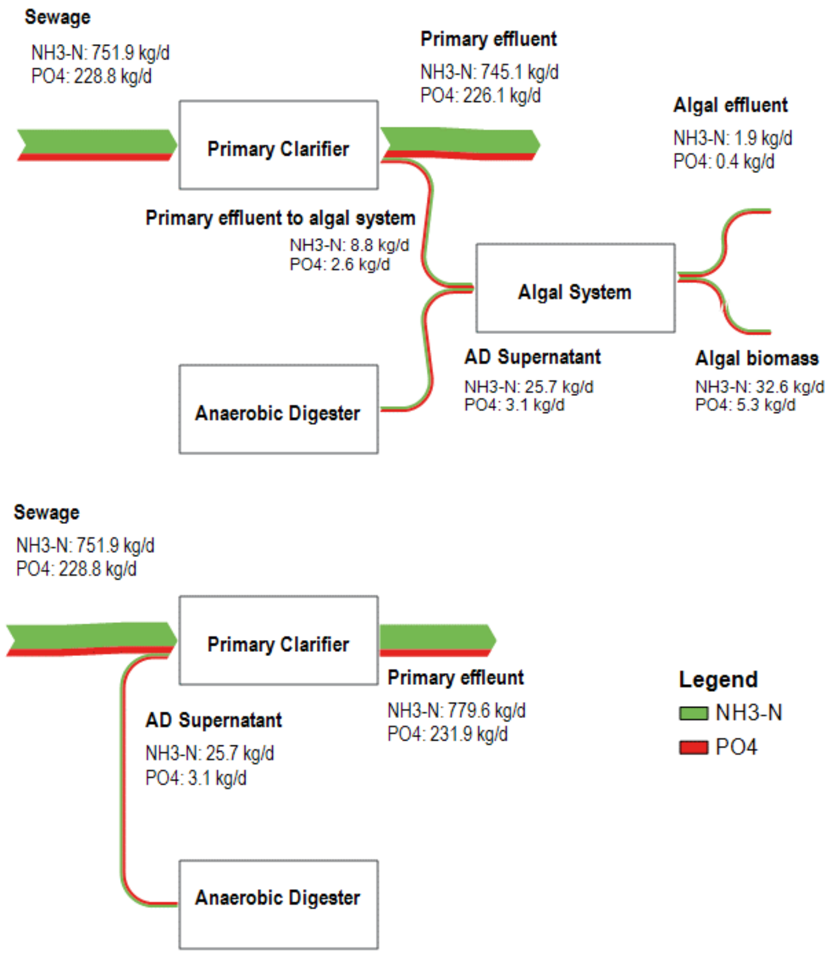

Figure 4: The nutrient load of mainstream wastewater treatment process **a)** with and, **b)** without the proposed algal system for AD supernatant treatment.

Figure 5 demonstrates the fate of N and P in the proposed algal system from AD supernatant treatment. As can be noted, the HTL process 61.9%, 16.6%, 10.1% and 11.4% of N respectively into the aqueous phase, biocrude, biochar, and off-gas. These results are similar to those reported in our previous study [10] where partitioning to each phase was reported as 57.2%, 19.3%, 11.2%, and 12.3%, respectively. Similar to our previously filed reports [9,10], almost all of the P (>99%) partitioned into biochar following the HTL process. Our ongoing nutrient recovery studies have shown the possibility to extract >85% P from biochar using 0.5M NaOH at respectively 5%, 60ºC and 3 days of solid content, temperature and holding time; the subsequent P recovery as struvite, on the other hand, was shown to be > 90%. For a conservative analysis, here, we assumed the extraction and precipitation efficiencies as 85% and 90%. Based on these considerations, it is estimated that 5.3% of N and 71.1% of P from the feed to the algal system could be recovered as struvite. This translates to a struvite yield of 6.75 kg per 100 m^3 of blended wastewater which is almost 3 times higher than that reported for control cycles (2.40 kg/m^3) [10].

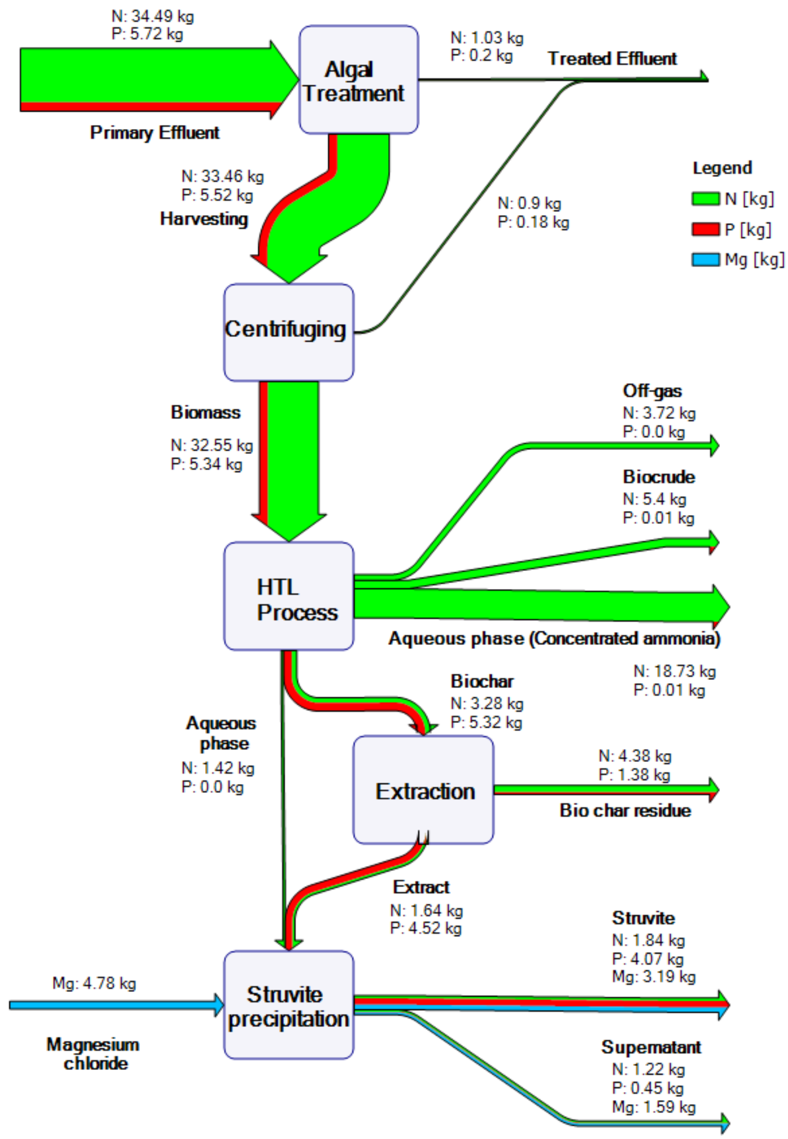

Figure 5: Fate of N and P in the proposed integrated algal system for a flow of 95.38 m^3/d of AD supernatant.

Figure 6 demonstrates the energy demands and recoveries of the algal system for an AD supernatant flow of 95.38 m^3/d. As can be noted, the algal system has an energy consumption of 1.15kWh/m^3 of blended (20% AD supernatant and 80% primary effluent) wastewater treated which is higher than that reported by Abeysiriwardana et al. (2020) [10] for 100% primary effluent treatment with the same system (0.49 kWh/m^3). Downstream processes to recover energy and nutrients in the STaRR system consume additional energy: 0.23 kWh/m^3 for separation of algae by centrifugation; 1.49 kWh/m^3 for heating in the HTL process; and 0.14 kWh/m^3 for heating in the P extraction process. Nevertheless, assuming 50% of the heat in the HTL process can be recovered [8], the energy generation from the HTL process as biocrude can be estimated as 1.83 kWh/m^3. Based on these considerations, the net energy consumption of wastewater treatment adds up to 1.18 kWh/m^3.

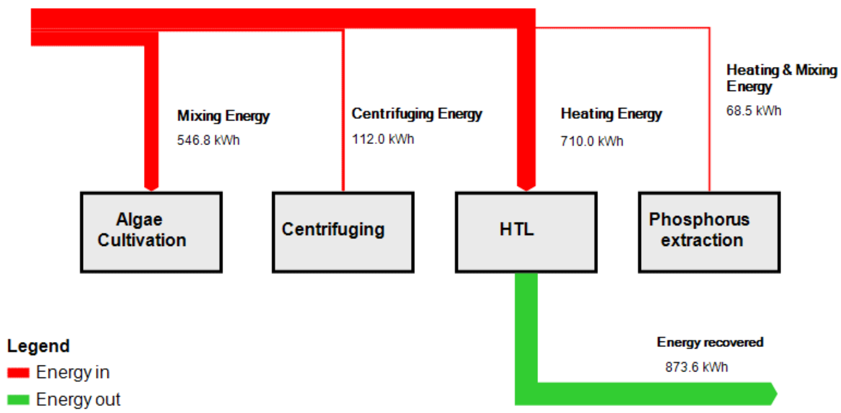

Figure 6: Energy demand and recovery of the integrated algal system for a flow of 95.38 m^3/d of AD supernatant.

Considering the mass of nutrient removal per unit energy ratios reported by Abeysiriwardana et al. (2020) [10] for full-scale wastewater treatment plants that use biological processes (81.1 g P/ kWh and 74.3 g N/ kWh), the net energy used by mainstream wastewater treatment to treat the same nutrient load is 1.06 kWh/m^3 wastewater. Although this is somewhat low compared to the algal system, it should be noted that the algal system also recovers nutrients as struvite at a yield of 6.75 kg/100 m^3 of blended wastewater. Hence, considering the energy consumption, environmental benefits and revenue from struvite, the algal system holds promise as a potentially greener alternative to the widely practiced AD supernatant recirculation to headworks.

6. **Provide a paragraph on who will benefit from your research results. Include any water agency that could use your results.**

The algal system investigated in this study is well suited for medium to large-sized municipal wastewater treatment plants in a semi-arid climate, such as the Las Cruces wastewater treatment plant in New Mexico. As identified through this preliminary study, the treatment of AD supernatant with algae has bright prospects. If this technology can be proven for AD supernatant treatment through long-term studies, wastewater treatment plants in cities like Las Cruces with anaerobic digestion will have the opportunity to lower their operational costs by introducing an algal system while also generating an extra income through nutrient recovery.

7. **Describe how you have spent your grant funds. Also provide your budget balance and how you will use any remaining funds. If you anticipate any funds remaining after May 31, 2020, please contact Carolina Mijares immediately. (575-646-7991); mijares@nmsu.edu)**

Grant funds were used in full to provide stipend support during summer.

8. **List presentations you have made related to the project.**

Poster presentation at New Mexico Water Conference, Hilton Santa Fe Buffalo Thunder Resort and Casino, Santa Fe, New Mexico; Title: *"Anaerobic Digester Supernatant Treatment Using Algal Systems"*

9. **List publications or reports, if any, that you are preparing. For all publications/reports and posters resulting from this award, please attribute the funding to NM WRRI and the New Mexico State Legislature by including the account number: NMWRRI-SG-2019.**

- Abeysiriwardana-Arachchigea, I.S.A., Chapmana, G., Rosalezb, R., Solizb, N., Cui, Z., Munasinghe-Arachchige, S.P., Delanka-Pedige, H.M.K., Brewer, C.E., Nirmalakhandan, N., n.d. Centrate Treatment and Resource Recovery in a Mixotrophic Algal System (manuscript under preparation).

10. List any other students or faculty members who have assisted you with your project.

Graham W. Chapman (grahamc@nmsu.edu)

11. Provide special recognition awards or notable achievements as a result of the research including any publicity such as newspaper articles, or similar.

As an article on me was published on New Mexico Water eNews in March 2020.

12. Provide information on degree completion and future career plans. Funding for student grants comes from the New Mexico Legislature and legislators are interested in whether recipients of these grants go on to complete academic degrees and work in a water-related field in New Mexico or elsewhere.

I am planning to complete my Ph.D. and graduate in the spring of 2021. If the opportunities would allow, I intend to complete a post-doctoral study immediately afterward. I am looking forward to joining a research-based career in the field of water and wastewater treatment.

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