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ALTERNATIVE TREATMENT TECHNOLOGIES FOR LOW-COST INDUSTRIAL  
AND MUNICIPAL WASTEWATER MANAGEMENT

by

Alan Hodges

A thesis submitted in partial fulfillment  
of the requirements for the degree

of

MASTER OF SCIENCE

in

Biological Engineering

Approved:

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Charles D. Miller, Ph.D.  
Major Professor

---

Judith L. Sims, M.S.  
Committee Member

---

Ronald C. Sims, Ph.D.  
Committee Member

---

Mark R. McLellan, Ph.D.  
Vice President for Research and  
Dean of the School of Graduate Studies

UTAH STATE UNIVERSITY  
Logan, Utah

2017

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## ABSTRACT

Alternative Treatment Technologies for Low-Cost Industrial  
and Municipal Wastewater Management

by

Alan Hodges, Master of Science

Utah State University, 2017

Major Professor: Dr. Charles D. Miller  
Department: Biological Engineering

Municipal and Industrial Wastewater production in North America alone is estimated at 85 cubic kilometers per year. This wastewater is subsequently treated by a variety of biological and physical processes. This thesis examines two novel treatment processes, one biological and one physical in nature, namely, Rotating Algae Biofilm Reactor treatment and expanded shale augmented coagulation-flocculation.

Rotating algae biofilm reactors (RABRs) were compared with suspended-growth open pond lagoon reactors for removal of nutrients and suspended solids in petrochemical wastewater. RABR treatment demonstrated a statistically significant increase in removal of nutrients and suspended solids and an increase in biomass productivity, compared to the open pond lagoon treatment. These trends translate to a greater potential for the production of biomass-based fuels, feed, and fertilizer as value-added products. This study is the first demonstration of the cultivation of mixed culture biofilm microalgae on petroleum refining wastewater for the dual purposes of treatment and biomass production.

A blend of expanded shale, a low cost ceramic derived from the concrete industry and an industry standard coagulant, ferric sulfate, was demonstrated as a feasible, novel, low cost coagulant. The optimum blend concentration for turbidity removal, 0.1 g/L expanded shale and 0.01 g/ L ferric sulfate, removed 84.7% and 91% of turbidity and suspended solids, respectively. This blend outperformed both the ferric sulfate only treatment and the expanded shale only treatment, demonstrating expanded shale as an effective additive to traditional coagulation-flocculation systems.

(51 pages)

## PUBLIC ABSTRACT

### Alternative Treatment Technologies for Low-Cost Industrial and Municipal Wastewater Management

Alan Hodges

Roughly the same volume of water that rushes over the Niagara Falls is produced as wastewater in North America. This wastewater is treated through a variety of means to ensure that it can be safely returned to the natural ecosystem. This thesis examines two novel means for this treatment, one biological and one physical-chemical in nature, namely, Rotating Algae Biofilm Reactor treatment and expanded shale augmented coagulation-flocculation.

Rotating algae biofilm reactors (RABRs) support biofilm algae growth, and in turn, the algae take up harmful contaminants from the wastewater. This system was tested in wastewater from petroleum refining operations. The efficacy of the RABR system was compared with a traditional method of wastewater treatment, open pond lagoons, where wastewater is open to sunlight and algae growth occurs in suspension as compared to the biofilm formed by the RABR system. The RABR treatment demonstrated a statistically significant increase in removal of three constituents in wastewater that are harmful to the environment: nitrogen, phosphorus, and suspended solids. Additionally, the RABR treatment demonstrated increased biomass production. This biomass can be converted into a variety of bioproducts including biofuels, agricultural feed, and nutraceuticals. This study is the first demonstration of this system in petroleum refining wastewater.

Currently, many wastewater treatment facilities use coagulation-flocculation to remove suspended solids from the wastewater. To achieve this removal, coagulants are added to the wastewater, which removes surface charges of the suspended particles, allowing particles in solution to coalesce and settle by gravity out of solution. One common coagulant added to wastewater is ferric sulfate. This study demonstrated that the addition of a new compound, expanded shale, to ferric sulfate could greatly improve the efficacy of the existing ferric sulfate coagulation system.

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Alan Hodges

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## CHAPTER I

### INTRODUCTION

Municipal and industrial wastewater is produced in large quantities worldwide in both industrial countries such as the United States (79.6 km<sup>3</sup>) and less developed countries such as Egypt (8.5 km<sup>3</sup>) making wastewater treatment an important global issue (Sato et al., 2013). Wastewater treatment is an economical issue as well, as wastewater treatment can often be a significant portion of an industry's or municipality's budget (Engin and Demir, 2006; Loehman et al., 1979). Therefore, there is a need to develop low cost effective wastewater treatment alternatives.

Current wastewater treatment processes can be grouped into three major categories: physical, biological, and chemical (EPA 2004). Physical processes include removal of bulk debris through screening and settling. Biological processes use microorganisms to reduce the concentration of organic wastewater constituents and include a variety of suspended and attached growth systems. Chemical treatment processes utilize chemicals to aid in wastewater treatment and includes coagulation-flocculation, ozonolysis, and chlorination among others. These treatment processes are used individually and in tandem to treat wastewater and mitigate the environmental impact of releasing the effluent into the environment (EPA, 2004).

This thesis describes the efficacy of two novel and potentially economical wastewater treatment strategies. 1) The Rotating Algae Biofilm Reactor (RABR), a biological treatment strategy, which produces algal biomass that can remove wastewater constituents as it grows. This biomass can be utilized to produce a variety of valuable

bioproducts that can offset wastewater treatment costs. 2) Coagulation-flocculation, a physical-chemical process, utilizing expanded shale as a low cost additive to improve treatment efficiency of conventional ferric sulfate systems.

Chapter II addresses the application of RABR treatment to petroleum refining wastewater. RABR treatment has been demonstrated in both industrial and municipal treatment systems to effectively treat wastewater and produce value-added products such as protein for animal feed, nutraceuticals, biocrude and other valuable products (Christensen and Sims, 2011; Satish et al., 2015). RABR treatment however has not previously been demonstrated for the dual purposes of treatment and biomass production in petroleum refining wastewater.

Petroleum refining wastewater contains nutrients, solids, and organic compounds, providing for a waste that is difficult and costly to treat using traditional wastewater treatment methods (Knight, 1999). Discharge of nutrients in wastewater including nitrogen and phosphorus, can cause eutrophication, or over-fertilization of receiving waters. Eutrophication is toxic to aquatic organisms, promotes excessive plant growth, depletes or reduces available oxygen, harms spawning grounds, alters habitats, and can greatly reduce biodiversity (Correll, 1998). In addition, high levels of total suspended solids (TSS) and chemical oxygen demand (COD) can reduce dissolved oxygen through the inhibition of photosynthesis due to turbidity and the stimulation of bacterial activity, respectively. This decreased dissolved oxygen caused by the wastewater can be detrimental to fish and other aquatic life. Therefore, the development of a wastewater treatment strategy capable of removing nitrogen, phosphorus, TSS, and COD from petroleum refining wastewater has the potential for the mitigation of these adverse

environmental impacts.

Chapter III addresses the development of a novel coagulant combination with an expanded shale additive. Effective coagulants remove suspended solids, which has the same positive environmental implications of TSS removal discussed above. Additionally, the development of new effective low-cost coagulants can reduce wastewater treatment costs by two unique mechanisms, 1) replacement of high cost coagulants and 2) reduction of overall plant footprint, which reduces land and construction costs.

The first mechanism for cost reduction is easily understood, if high cost coagulants such as polymeric coagulants can be replaced by lower cost alternatives, operation costs of treatment plants can be reduced. The second mechanism is more abstract but has large economic implications. Land and construction costs for wastewater treatment plants can be very costly. A significant portion of the plant size is consumed by the secondary clarifiers of the plant where suspended solids are removed from the wastewater via coagulation-flocculation. If the rate of settling is increased in the clarifier by adding coagulant that promotes rapid settling, the retention time needed for the wastewater in the clarifier is lowered and therefore a smaller clarifier is needed to treat the same amount of wastewater.

The second method is of interest locally because the City of Logan is replacing the current wastewater treatment lagoons with a new adjacent treatment plant. The soils at this site are not amenable for construction and therefore costly driven pile foundations must be constructed under the entire footprint of the plant. If the City of Logan can find a novel coagulant that effectively treats the wastewater and promotes rapid settling, clarifier size and therefore cost of plant construction can be reduced considerably.

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CHAPTER II

NUTRIENT AND SUSPENDED SOLIDS REMOVAL FROM PETROCHEMICAL  
WASTEWATER VIA MICROALGAL BIOFILM CULTIVATION

This chapter, with slight modifications is published with the following citation:

Hodges, A., Fica, Z., Wanlass, J., VanDarlin, J., & Sims, R. (2017). Nutrient and suspended solids removal from petrochemical wastewater via microalgal biofilm cultivation. *Chemosphere*, 174, 46-48.

Abstract

Wastewater derived from petroleum refining currently accounts for 33.6 million barrels per day globally. Few wastewater treatment strategies exist to produce value-added products from petroleum refining wastewater. In this study, mixed culture microalgal biofilm-based treatment of petroleum refining wastewater using rotating algae biofilm reactors (RABRs) was compared with suspended-growth open pond lagoon reactors for removal of nutrients and suspended solids. Triplicate reactors were operated for 12 weeks and were continuously fed with petroleum refining wastewater. Effluent wastewater was monitored for nitrogen, phosphorus, total suspended solids (TSS), and chemical oxygen demand (COD). RABR treatment demonstrated a statistically significant increase in removal of nutrients and suspended solids and increase in biomass productivity, compared to the open pond lagoon treatment. These trends translate to a greater potential for the production of biomass-based fuels, feed, and fertilizer as value-added products. This study is the first demonstration of the cultivation of mixed culture biofilm microalgae on petroleum refining wastewater for the dual purposes of treatment

and biomass production.

## 1.0 Introduction

Refining of petroleum is a large global industry (Wauquier, 1995), that produces large quantities of wastewater estimated at 33.6 million barrels per day (Diya'uddeen et al. 2011). Petroleum refining wastewater contains nutrients, solids, and organic compounds, providing for a waste that is costly to treat using traditional wastewater treatment methods (Knight, 1999).

Microalgae culture has demonstrated the ability to remove nitrogen, phosphorus, and organic compounds from petroleum refining wastewater (Antic et al., 2006; Chavan and Mukherji, 2010; Madadi et al., 2016); however, the major current biological treatment strategy for petroleum wastewater consists of suspended growth processes. Suspended growth processes often have operational problems associated with proper settleability of sludge and sludge accumulation. These complexities can be avoided through the use of attached growth treatment processes. Research by Chavan and Mukherji (2008) indicated that biofilm growth processes show potential for replacing suspended growth processes, but there has been limited research on treating petroleum wastewater.

Furthermore, many conventional wastewater treatment approaches do not produce value-added products that offset the cost of treatment operations. In contrast, the Rotating Algae Biofilm Reactor (RABR) has demonstrated the ability to effectively remove macronutrients and to produce a variety of bioproducts (Christenson and Sims, 2011; Ellis et al., 2012; Sathish et al., 2014). Successful demonstration of a RABR system for

treatment of petroleum refinery wastewater would provide a novel approach to both wastewater treatment and bioproduct development. This study examined the effectiveness of RABR treatment for the production of microalgae-based biomass and the removal of nutrients, suspended solids, and organic species from petroleum refining wastewater.

## 2.0 Materials and methods

### 2.1 Biofilm growth conditions

One-liter RABRs (0.029 m<sup>2</sup> areal footprint) were constructed and operated as described by Christenson and Sims (2012) (Appendix B, Figure B.1.). Solid braid cotton rope was used as biofilm growth substratum. Open pond lagoons consisted of uncovered basins identically matching the reservoirs containing the rotating drums of the RABR (0.029 m<sup>2</sup> areal footprint) (Appendix B, Appendix Figure B.2.). RABRs and open pond lagoons were artificially illuminated continuously using fluorescent lamps operated at 230±15 µE m<sup>-2</sup> and temperature was maintained at 20±1 °C. RABRs and open pond lagoons were inoculated with polyculture biofilm microalgae represented by a mixture of the wastewater derived cultures described by Fica and Sims (2016) and Wood et al. (2015).

RABRs and open pond lagoons were operated in continuous flow after an initial three-week period of batch operation to acclimate the biofilm microalgae to the wastewater. RABRs were operated in parallel triplicate groups with 24-hr and 48-hr hydraulic retention times (HRT), and duplicate open pond lagoons operated at a 36-hr HRT. RABR biomass productivity was quantified by mechanically scraping the biofilm from the substratum, followed by determination of dry-weight as described by Wood et

al. (2015). Open pond growth lagoon biomass productivity was quantified as the change in TSS concentration in the wastewater with time.

### 2.1.1 Wastewater characteristics

Wastewater was obtained from a refining industry in northern Utah. Wastewater was collected from the influent wastewater stream immediately upstream of the API (American Petroleum Institute) Separator. Wastewater was dilute and homogenous and did not contain distinct oil and water phases. Wastewater characteristics were determined by the contract laboratory Chemtech Ford (Sandy, UT) and are given in Table 1. All studies were performed using a single preserved sample to ensure constant uniformity of the influent wastewater.

**Table 1**

Petroleum refining wastewater composition used as the medium for microalgae cultivation.

Influent Wastewater Constituent	Influent Constituent Concentration
Total Nitrogen (N)	25 mg/L
Total Phosphorus (P)	1.8 mg/L
Total Suspended Solids (TSS)	39 mg/L
Volatile Suspended Solids (VSS)	20 mg/L
Total Dissolved Solids (TDS)	2,470 mg/L
Oil and Grease (HEM)	25 mg/L
Chemical Oxygen Demand (COD)	163 mg/L
Diesel Range Organics	22.2 mg/L
Gasoline Range Organics (GRO)	2.42 mg/L
pH	8.0

## 2.2 Effluent wastewater sampling and analysis

Effluent wastewater from each RABR and lagoon unit was sampled weekly for 12 weeks. Analysis of COD, nitrogen, and phosphorus was performed on filtered samples using HACH reagent sets (Loveland, CO). Procedures used were according to the following methods: HACH 8000 Reactor Digestion for total COD Method, 10072 DR800 HR Test 'N Tube for total nitrogen, and 8190 Digestion Test 'N Tube for total phosphorus. TSS was measured according to Method 2540B (APHA, 2005).

## 2.3 Statistical methods

All effluent wastewater characteristics (N, P, TSS, and COD) were analyzed using 1-way analysis of variance (ANOVA). Subsequent pairwise comparisons of treatment groups were compared using Student's T-tests. P-values <0.005 were considered to be statistically significant.

## 3.0 Theory

RABR wastewater treatment involves a partially submerged rotating drum, with a biofilm supporting growth substratum that allows attached phototrophic microalgal biofilms to be exposed to two growth environments: 1) nutrients in the wastewater, and 2) light and carbon dioxide in the atmosphere. As the biofilm grows, nutrients from the wastewater can be converted to biomass in the biofilm. The biofilm is then removed, through mechanical scraping, allowing for the removal of nutrients from the overall treatment system and for the production of bioproducts from the harvested biomass. General RABR operation is described by Christenson and Sims (2012) and laboratory scale operation is described by Fica and Sims (2016) and Wood et al. (2015).

#### 4.0 Results and discussion

Continuous-flow RABR treatment units demonstrated statistically significant reduction of nitrogen, phosphorus, and TSS compared to the open pond growth lagoon treatment (Figure 1). Effluent concentrations from the 24-hr HRT RABR and 48-hr HRT RABR systems were decreased from the influent concentrations by 18.1 mg/L (72.4%) and 17.7 mg/L (70.8%) for nitrogen, 0.90 mg/L (50%) and 1.00 mg/L (55.6%) for phosphorus, and 20.9 mg/L (53.6%) and 23.9 mg/L (61.3%) for TSS, respectively. Whereas the open pond growth lagoon decreased effluent concentrations of nitrogen and phosphorus by 3.47 mg/L (13.9%) and 0.34 mg/L (18.9%) respectively, and increased TSS by 18.3 mg/L (46.9%). Both RABR groups demonstrated statistically larger reductions of nitrogen, phosphorus, and TSS compared to open pond lagoons, however no statistical difference in removal was found between 24-hr HRT and 48-hr HRT R

To effectively compare biomass productivity of the RABR and the open pond growth lagoon systems, biomass productivity was determined per unit area of areal footprint. Dry biomass was produced at an average rate of  $4.11 \text{ g m}^{-2} \text{ d}^{-1}$  across all RABR groups, compared to  $0.4 \text{ g m}^{-2} \text{ d}^{-1}$  for the open pond lagoon. Visual microscopy indicated that the microalgae community of the RABR system was dominated by filamentous cyanobacteria (Figure B.2.) while open pond lagoon microalgae was largely composed of green microalgae species. Overall, the RABR system demonstrated a significantly larger biomass productivity than the open pond growth lagoon system, and therefore, demonstrated a significantly larger potential to produce biomass-based, value-added products.

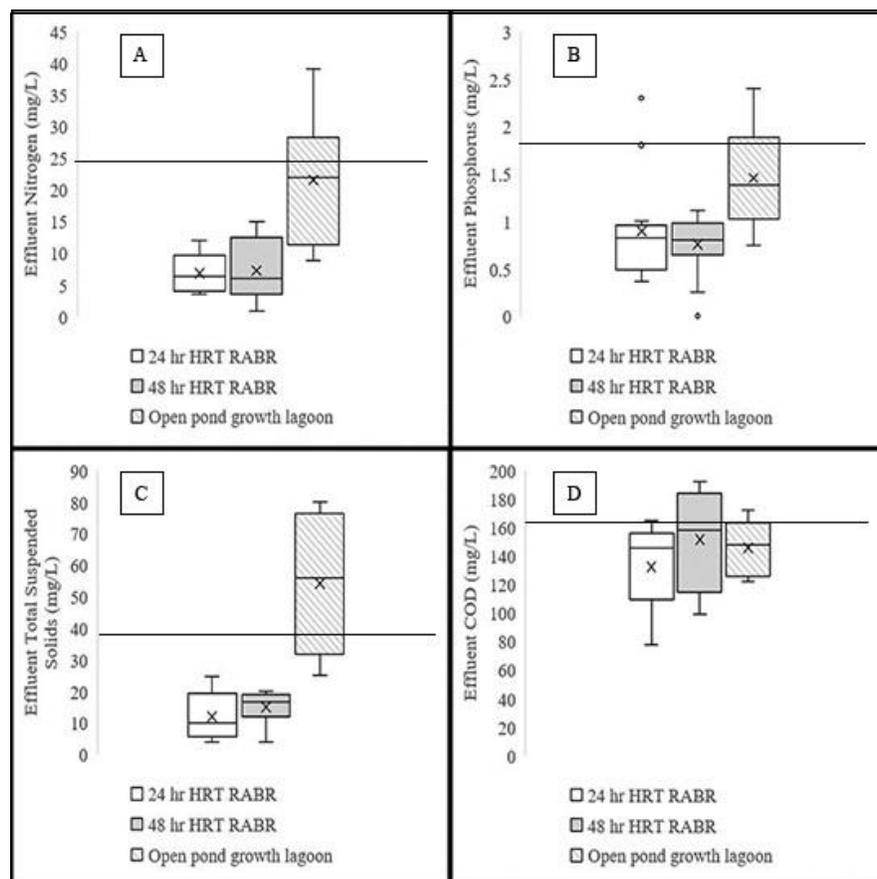
In order to perform a mass balance on nitrogen and phosphorus in the system,

biofilm was assumed to follow the molar C:N:P ratio given by the Redfield Ratio for microalgae of 106:16:1 (Hillebrand, 1999). Biomass productivity of  $4.11 \text{ g m}^{-2} \text{ d}^{-1}$  correlates with a removal of  $17.5 \text{ mg L}^{-1} \text{ d}^{-1}$  nitrogen and  $2.4 \text{ mg L}^{-1} \text{ d}^{-1}$  of phosphorus. Nitrogen removal calculated from the mass balance using the Redfield Ratio closely matches the observed nitrogen removal. This correlation suggests that nitrogen removal was most significantly due to biomass growth and not sorption or solids removal. Calculated phosphorus removal, however, did not closely match observed values. This discrepancy was not unexpected due to the large variation in C:P ratios observed in cyanobacterial cultures (Hessen, 2005).

The RABR technology did not perform differently from the open pond lagoons in the removal of chemical oxygen demand (COD) (Figure 1D). Due to the availability of carbon dioxide as the carbon source for the phototrophic biofilm and the recalcitrant nature of the organic substances in petroleum refining wastewater (Pakula et al., 1998; Sun et al., 2008), a significant reduction in COD due to heterotrophic activity of the biofilm was not expected nor observed.

## 5.0 Design calculations

Using removal rates from the 24-hr HRT RABR and given the  $0.043 \text{ m}^2$  surface area of the laboratory scale RABR, removal rates per unit surface area can be determined (Tables 2,3). Based on a theoretical plant producing 0.1 mgd ( $3.78 \times 10^5$  liters) petroleum refining wastewater as characterized in this study would need to remove 0.3 kg of phosphorus to meet the 1 mg/L State of Utah Secondary effluent guidelines and 5.29 kg of suspended solids to meet the 25 mg/L State of Utah Secondary effluent guidelines.



**Fig. 1.** (A-D) Petroleum effluent wastewater characteristics. Figure 1A Effluent nitrogen concentration. Figure 1B Effluent phosphorus concentration. Figure 1C Effluent total suspended solids concentration. Figure 1D Effluent COD concentration. The sample median is represented by a horizontal line through the box, the mean of the sample is denoted with an x, the lower and upper bounds of the boxes represent the 25th and 75th percentile of sample, whiskers represent maximum and minimum values, and dots represent outliers. Solid lines through each plot represent influent concentration

**Table 2**

Nutrients and suspend solids removal per unit RABR surface area.

Constituent	Observed RABR removal	Removal per m <sup>2</sup> surface area
Nitrogen	17.7 mg d <sup>-1</sup>	411.6 mg m <sup>-2</sup> d <sup>-1</sup>
Phosphorus	0.90 mg d <sup>-1</sup>	20.9 mg m <sup>-2</sup> d <sup>-1</sup>
Suspended solids	20.9 mg d <sup>-1</sup>	486 mg m <sup>-2</sup> d <sup>-1</sup>

**Table 3**

Required RABR surface area to meet State of Utah phosphorus and suspended solids effluent guidelines.

Constituent	Required removal (kg)	Required RABR surface area
Phosphorus	0.3	14300 m <sup>2</sup>
Suspended solids	5.29	10900 m <sup>2</sup>

## 6.0 Conclusions

Microalgae was successfully cultivated on petroleum refining wastewater using both biofilm and suspended open pond systems. Significant reductions in nutrients and suspended solids concentrations were statistically significantly greater using the biofilm (RABR) system compared to the open pond growth lagoon system. Additionally, the RABR system exhibited greater biomass productivity than open pond lagoons, which represents biomass that can be utilized to produce value-added products. This study demonstrated the application of mixed culture microalgae in Rotating Algae Biofilm Reactors (RABRs) as a novel approach in the treatment of petroleum refining wastewater that also provides a biomass feedstock for the production of downstream bioproducts.

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CHAPTER III  
EXPANDED SHALE AS AN ADDITIVE FOR THE COAGULATION-  
FLOCCULATION OF WASTEWATER

Abstract

Municipal and industrial wastewater production in North America is estimated at 85 cubic kilometers per year, and a significant proportion of this wastewater is treated using coagulation-flocculation systems. Therefore, there is an interest to develop effective, low cost coagulants to be utilized in these systems. This study demonstrates the feasibility of a novel, low cost coagulant blend of expanded shale, a ceramic derived from the concrete industry, and an industry standard coagulant, ferric sulfate. Triplicate jar tests were operated using synthetic wastewater flocculated with varying concentrations of either ferric sulfate, expanded shale, or a combination of both components. The wastewater was subsequently analyzed for turbidity removal. The optimum concentration for turbidity removal, 0.1 g/L expanded shale and 0.01 g/ L ferric sulfate, removed 84.7% and 91% of turbidity and suspended solids, respectively. This blend outperformed the ferric sulfate only treatment, demonstrating expanded shale as an effective additive to traditional coagulation-flocculation systems. Additionally, zeta potential was used to determine the mechanism of flocculation for this novel treatment blend.

1.0 Introduction

The use of coagulation-flocculation for the removal of suspended solids and turbidity from wastewater is widely used in many treatment facilities. High levels of total suspended solids (TSS) and associated high water turbidity can reduce dissolved oxygen

to concentrations hazardous to aquatic life through the inhibition of photosynthesis. Coagulation-flocculation removes suspended solids and turbidity from wastewater through the addition of a coagulant. The coagulant counteracts the electrostatic repulsion between the colloidal particle, allowing particles to aggregate and settle out of solution (Weber 1972). A common coagulant combination is aluminum or iron salts used together with high molecular weight polymers (Anastakis et al. 2009). These coagulants, especially polymeric coagulants however are expensive (approximately \$10/kg) and can be a significant contribution to the overall operating costs of a treatment facility (Harrison et al. 2015). Therefore, the development of new, low cost coagulants is needed to reduce operating costs of treatment facilities while providing a high level of treatment for the wastewater.

One potential low cost coagulant is expanded shale. Expanded shale is widely used as concrete aggregate to produce lightweight structural concrete (Weigler 1980). Expanded shale has been demonstrated to reduce turbidity in storm water treatment operations (Hauser, 2005), however knowledge is limited on the use of expanded shale in wastewater systems. This study explores the use of expanded shale both as an independent coagulant and as an additive to a conventional coagulant in order to develop a low cost, effective coagulation-flocculation treatment strategy.

## 2.0 Materials and methods

Expanded shale was obtained from a local concrete aggregate company. Elemental composition was determined by X-ray fluorescence (XRF) by DCM Science Laboratory (Wheat Ridge, CO). Constituents with a mass fraction >1 % listed in Table 4.

**Table 4**  
Expanded shale elemental analysis.

Constituent	Mass fraction (%)
SiO <sub>2</sub>	56.6
AlO <sub>3</sub>	14.2
CaO	6.02
Fe <sub>2</sub> O <sub>3</sub>	4.21
MgO	2.25
K <sub>2</sub> O	1.98

To examine the effects of ferric sulfate and expanded shale in combination as a coagulant system, jar testing was performed. Triplicate Jar tests, as described by ASTM D2035, were operated using synthetic wastewater amended with either ferric sulfate, expanded shale or a combination of both components (Figures B.3.-B.6.) Jar tests (DBT6 batch jar tester EC Engineering, Canada) were operated by flash mixing (300 rpm) for 1 min, followed by 15 minutes of moderate mixing at 30 rpm, and a 30-minute settling time. Concentrations of each coagulant tested were 0, 0.1 g/L, and 1 g/L expanded shale and 0, 0.01 g/L, and 0.1 g/L ferric sulfate, where each concentration of each compound was tested in combination with every other concentration of the other compound.

Synthetic wastewater consisted of 100 mg/L of kaolin powder (Sigma-Aldrich) and 10 mg/L of humic acid (technical grade, Sigma-Aldrich) in distilled water and mixed for 12 hours as described by Anastasakis et al. (2009). Measured turbidity, total suspended solids (TSS), zeta potential, and pH of the synthetic wastewater was 163 Nephelometric Turbidity Units (NTU), 103 mg/L, -65.1 mV and 6.0 respectively.

After the conclusion of each jar test, turbidity (Hach 2100Q, USEPA Method 180.1 compliant) of the supernatant was measured. Supernatant samples were collected from a port is located 133 mm from the bottom of the reactor (1/3 of the reactor height). The optimum treatment combination, as determined by reduction of turbidity, was subsequently tested for removal of suspended solids and solution zeta potential. TSS was measured using standard method 2540B (APHA, 2005). Zeta potential was measured using a Brookhaven Zeta-Plus (Holtville, NY, USA) and is reported as an average of 10 measurements plus or minus the standard error.

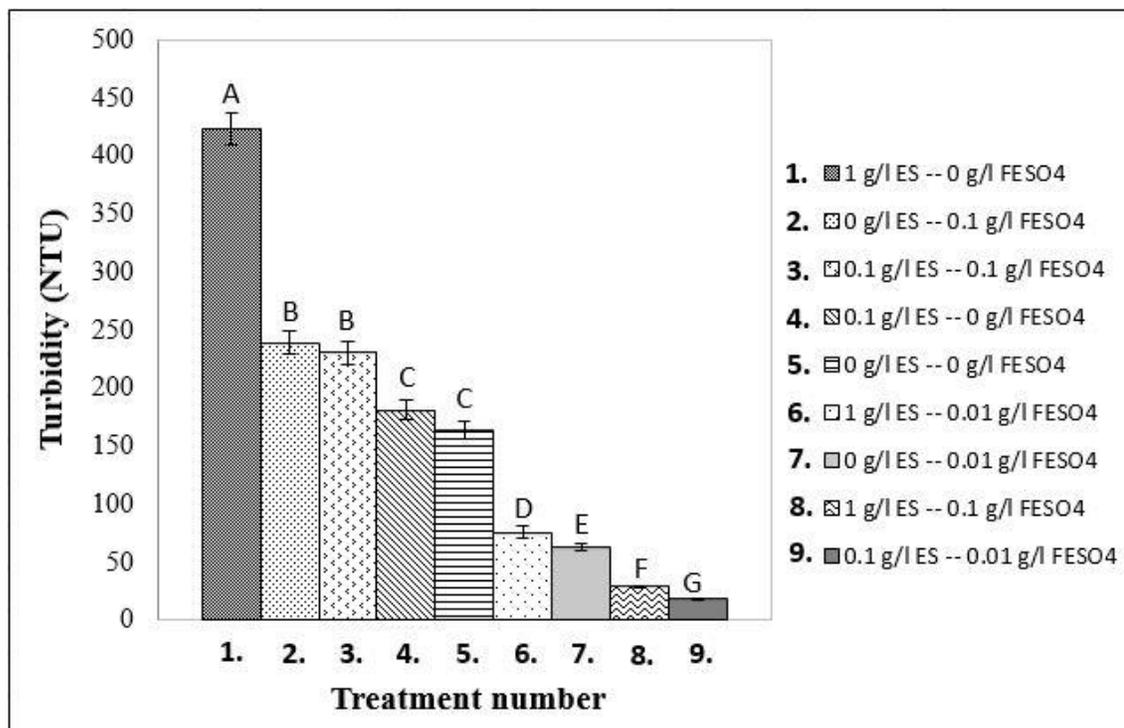
## 2.1 Statistical methods

Jar tests were operated in triplicate. Turbidity from each treatment groups was analyzed using 2-way analysis of variance (ANOVA). Subsequent pairwise comparisons of treatment groups were compared using the Tukey- Kramer method. P-values <0.005 were considered to be statistically significant.

## 3.0 Results and discussion

### 3.1 Flocculation performance

The final turbidity of the wastewater after jar testing varied widely across coagulant combination groups (Figure 1). This variation is statistically significant with a 2-way ANOVA p-value <0.0001. Additionally, pairwise comparisons of the individual treatment combinations indicates that the nine treatment combinations tested can be grouped into seven groups (labeled A-G in Figure 2) that result in statistically different final turbidity values. (p-values < 0.05, Tukey-Kramer Method).



**Fig. 2.** Final turbidity of each jar test. Treatment combinations where different letters indicate statistically significant difference between groups (Tukey adjusted P- value < 0.05). Error bars represent 95% confidence intervals. ES denotes expanded shale, FESO4 denotes ferric sulfate.

The coagulant combination of 0.1 g/L expanded shale and 0.01 g/L ferric sulfate (Treatment 9) resulted in the lowest final turbidity of 20.6 NTU, corresponding to an 87.4% removal of the initial turbidity and a suspended solids removal of 86.5 mg/L (91%). This turbidity reduction is a significant improvement to the flocculation performance of ferric sulfate of the same concentration without expanded shale (Treatment 7) (38.0% vs 87.4%). Moreover, both 10:1 expanded shale to ferric sulfate ratio (m/m) treatments (#8, #9) significantly outperformed the ferric sulfate alone (Treatments 2, 7) and control (Treatment 5, no coagulant amendment) treatments,

indicating that expanded shale could be used in a variety of concentrations as a low cost amendment to current flocculation systems to improve overall performance.

The treatment groups of expanded shale without the addition of ferric sulfate all significantly increased turbidity compared to the control group. Turbidity increased proportionally to the concentration of expanded shale added. This increase in turbidity suggests expanded shale is not an appropriate coagulant when used independently. Additionally, turbidity increase compared to the control was observed in the 0.1 g/L ferric sulfate alone treatment group (Treatment 2). This increase in turbidity is common when coagulant dosage is too large.

### 3.2 Coagulation mechanism

To further characterize the expanded shale-ferric sulfate coagulant combination, the coagulation-flocculation mechanism was explored. Zeta potential, a common indicator of coagulant behavior (Chekli et al. 2015), of the synthetic wastewater ( $-65.1 \pm 6.4$  mV) was significantly neutralized to  $-2.5 \pm 1.6$  mV by Treatment 9 (0.1 g/L expanded shale and 0.01 g/L ferric sulfate). This significant, but not complete, charge neutralization of the wastewater indicates that both charge neutralization and sweep coagulation/adsorption are important mechanisms of coagulation in this system (Gregory and Duan 2001).

Furthermore, ANOVA modelling suggests that both treatment factors, ferric sulfate concentration and expanded shale concentration, and the interaction between the treatment factors all significantly contribute to the final turbidity (p- values  $< 0.001$ ). The statistically significant interaction between ferric sulfate and expanded shale likely

indicates a physical-chemical interaction of the two coagulants in solution.

#### 4.0 Conclusion

This study demonstrated the feasibility of the utilization of expanded shale as a low cost additive to improve performance of traditional coagulation-flocculation systems. The addition of 0.1 g/L of expanded shale improved the turbidity removal capacity of 0.01 g/L ferric sulfate by 49.4%. Furthermore, expanded shale-ferric sulfate coagulant blend was demonstrated to neutralize colloid charge in the process of flocculation.

#### Acknowledgments

The City of Logan, UT, the Huntsman Environmental Research Center (HERC) and The Utah State University Sustainable Waste-to-Bioproducts Engineering Center provided financial support for this study.

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## CHAPTER IV

### SUMMARY, CONCLUSIONS, AND ENGINEERING SIGNIFICANCE

Two wastewater treatment systems were demonstrated to be effective and economically feasible, 1) Rotating Algae Biofilm (RABR) for the treatment of petroleum refining wastewater and 2) Expanded shale as an additive to aid in coagulation-flocculation of wastewater treated with ferric sulfate.

RABR treatment significantly reduced nutrients and suspended solids concentrations in petrochemical wastewater. Reductions were statistically significantly greater using the biofilm (RABR) system compared to the open pond growth lagoon system. Additionally, the RABR system exhibited greater biomass productivity than open pond lagoons, which represents biomass that can be utilized to produce value-added products. This study demonstrated the application of mixed culture microalgae in Rotating Algae Biofilm Reactors (RABRs) as a novel approach in the treatment of petroleum refining wastewater that also provides a biomass feedstock for the production of downstream bioproducts.

Expanded shale was demonstrated as a low cost additive to improve performance of traditional coagulation-flocculation systems. The addition of 0.1 g/L of expanded shale improved the turbidity removal capacity of 0.01 g/L ferric sulfate by 49.4%. Furthermore, expanded shale-ferric sulfate coagulant blend was demonstrated to neutralize colloid charge in the process of flocculation.

## CHAPTER V

### RECCOMENDATIONS FOR FUTURE RESEARCH

#### 1.0 Future research pertaining to Chapter II

The RABR system has been well demonstrated in the laboratory to effectively remove nutrients from wastewater, while producing biomass that can be used as a value added product. The RABR has been much less well demonstrated at the pilot scale. Pilot scale demonstration is imperative to examining the techno-economic viability of the RABR system and must be demonstrated at the pilot scale before industry use.

Although nutrient removal and biomass productivity has been well demonstrated, the removal of specific contaminants of concern such as pharmaceuticals or heavy metals has not been thoroughly explored. Future laboratory studies examining the removal of these contaminants of concern would be a significant contribution to the current body of knowledge.

#### 2.0 Future research pertaining to Chapter III

The coagulant selected by the city of Logan to increase the solids removal speed and efficiency to mediate the problem discussed in Chapter I was the ferrous coagulant BioMag. Future research should compare the effectiveness of the expanded shale with BioMag. A three-factor experiment similar to the experiment described in chapter III combining varying levels of BioMag, expanded shale, and ferric sulfate would determine the relative coagulation effectiveness and also determine if expanded shale or ferric sulfate would be an effective additive to the current BioMag system.

APPENDICES

## APPENDIX A

### REDUCTION OF GREENHOUSE GAS EMISSIONS FROM SEPTIC TANKS VIA COMPOST BIOFILTRATION

#### 1.0 Introduction

Concerns with climate change have led to efforts to reduce the emission of greenhouse gases (GHGs). Methane has been identified as a GHG, with the heat-trapping equivalent to more than twenty times that of carbon dioxide. Using assumptions developed by the Intergovernmental Panel on Climate Change (IPCC), the U.S. Environmental Protection Agency GHG inventory (EPA, 2011) estimated that 76 percent of wastewater sector methane emissions in the United States are from onsite (septic) systems (Figure A1). A project funded by the Water Environment Research Foundation (Leverenze et al., 2011) concluded that study is needed to develop technologies for the control of GHG emissions from on-site wastewater systems.

#### 2.0 Materials and Methods

Bench top packed-bed reactors (PBRs) were used to test the effectiveness of methane removal using municipal compost. A diagram of the reactor can be seen in Figure A2. The PBRs were fed methane (70 % v/v methane in air mixture at 10 mL min<sup>-1</sup>) to mimic concentration and flow of septic tank effluent gas. Rate of methane removal was measured using gas chromatography to analyze influent and effluent methane gas concentration. To estimate the effect of temperature on methane removal temperature controlled reactors at -20, 4, 22, 37 °C. 20 mL were utilized.



### 3.0 Results and discussion

Overall, neither the packed bed reactors nor the temperature controlled batch reactors produced data that was reproducible across multiple trials and therefore any data collected is considered largely invalid.

### 4.0 Future research recommendations

The future of this project lies in laboratory scale batch operated reactors and not continuous flow reactors or field scale work. The initial three objectives of the laboratory studies are 1) demonstrate the ability of septic tank sludge can produce methane gas in vitro, 2) examine feasibility of inoculating compost with a methanotrophic bacteria to enhance methane degradation, 3) examining the microbial composition of methane exposed compost to identify natural methanotrophic bacteria in green waste compost and the change in microbial composition during long exposures to high methane concentrations. A sample protocol to accomplish these three objectives is given below.

To examine the methane potential of septic tank sludge, biomethane potential testing could be performed as described by Angelidaki et al (2009). Triplicate 1 L glass bottles (actual volume 1165 mL) are filled with 50 mL of septic tank sludge and 250 mL of either septic tank wastewater or deionized water. The septic tank sludge and wastewater to be obtained from a residential septic tank. Reactors to be incubated at 37 °C and continuously shaken. Every four days, pressure of the reactors is to be measured using a digital pressure probe and 2 mL gas samples analyzed via gas chromatography (Agilent 7890B). Ideal gas relationships can be used to convert pressure of reactor to gas produced, adjusting for the vapor pressure of water at 37°C.

To examine the feasibility of soil based gaseous methane degradation by compost systems of varying microbial communities, a trial comparing the methane degradation rate of unaltered (natural), sterile, and artificially inoculated compost could be performed. 1 L Replicate reactors containing 300 mL of greenwaste compost could be randomized to receive one of three treatments. 1) No treatment (control), 2) sterilization or 3) inoculation with of *Methylosinus trichosporium* OB3b (ATCC 35070) or similar methanotroph. These reactors are then augmented with methane (CP grade) to produce a 70% v/v concentration of methane in the headspace. Gas samples are to be taken from the headspace every 10 days and analyzed via gas chromatography as described above. After analysis, methane gas is added to each vial to maintain a consistent headspace methane concentration of 70% v/v.

A 70% v/v concentration methane headspace concentration was maintained for six months and every two months the soil is to be sampled and analyzed for metagenomic changes in the microbial community. DNA libraries obtained from compost samples are to be sequenced and the compost metagenome analyzed.

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APPENDIX B  
SELECTED PHOTOGRAPHS

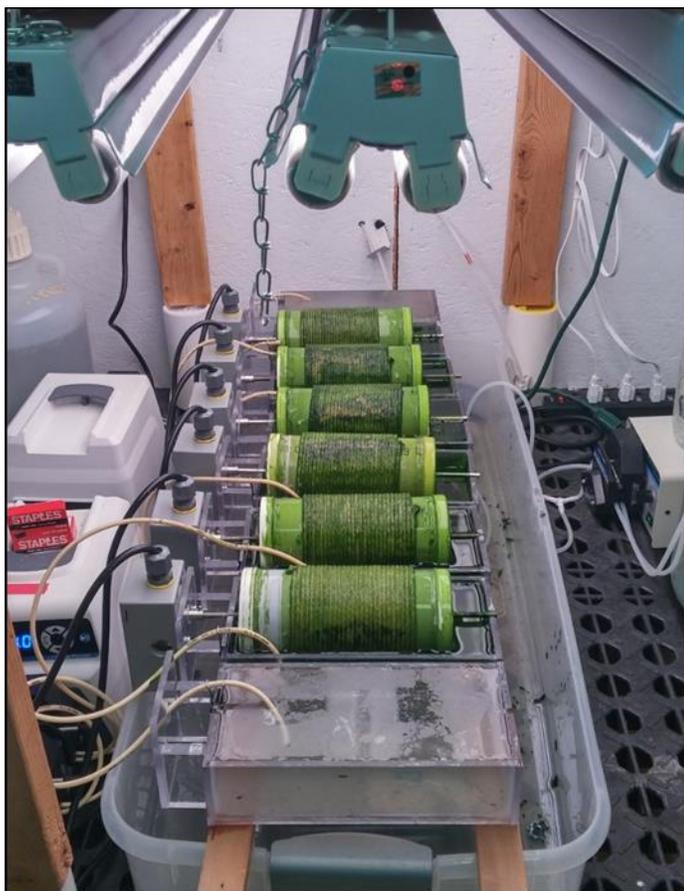


Figure B.1. Microalgal growth on RABR units cultured in petrochemical wastewater.

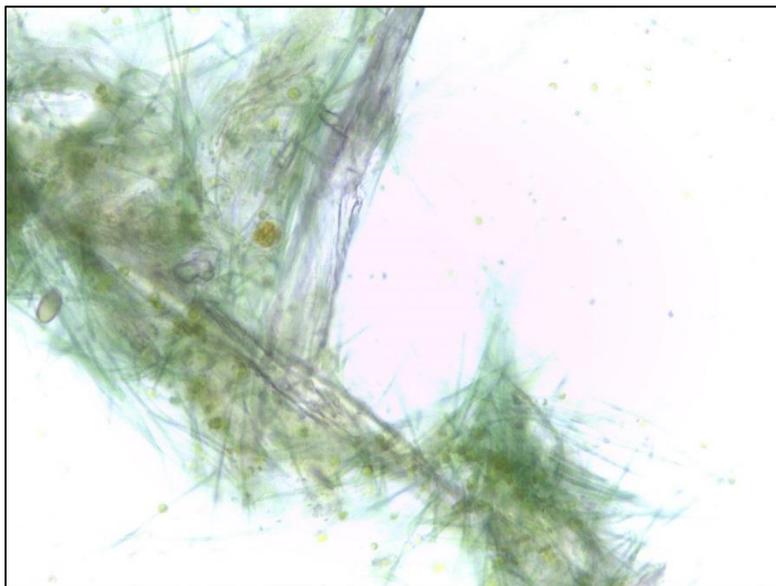


Figure B.2. Micrograph of microalgal growth cultured on RABR units.



Figure B.3. Powdered expanded shale used as a coagulant

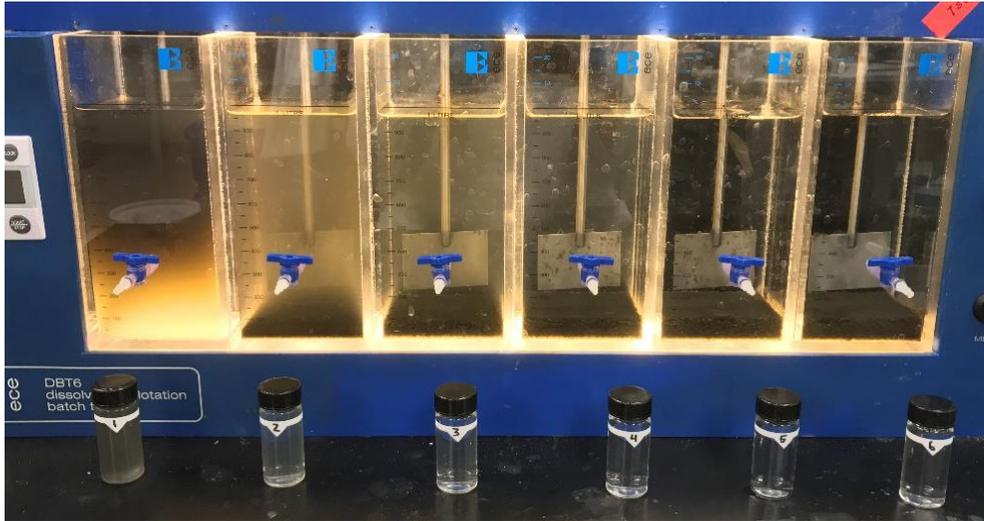


Figure B.4. Jar test demonstrating coagulation-flocculation effectiveness of ferric sulfate in resuspended sludge media. Ferric sulfate concentration left to right 0, 50, 100, 150, 200, 250 mg/L. (Data from this experiment not presented in this document, figure serves as a visual example of successful coagulation-flocculation).



Figure B.5. Jar test demonstrating coagulation-flocculation ineffectiveness of expanded shale in resuspended sludge media. Expanded shale concentration left to right 0, 0.5, 1, 2, 5, 10 g/L. (Data from this not presented in this document, figure serves as a visual example of unsuccessful coagulation-flocculation).



Figure B.5. Jar test demonstrating coagulation-flocculation ineffectiveness of expanded shale in kaolin-humic acid media. Expanded shale concentration left to right 0, 0.5, 1, 2, 5, 10 g/L. (Data from this not presented in this document, figure serves as a visual example of unsuccessful coagulation-flocculation).

## APPENDIX C

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Logan, UT 84322

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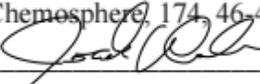
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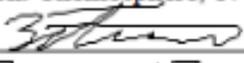
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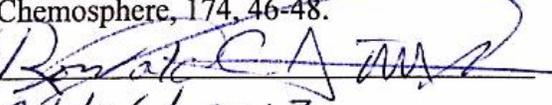
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Signed: 

Date: 04/06/2017

Printed name: RONALD C. SIMS

## CURRICULUM VITAE

**Education**

B.S. Biological Engineering, Minors: Biology, Chemistry summa cum laude Utah State University, Logan, Utah	2016
M.S. Biological Engineering, Utah State University, Logan, Utah	2017

**Leadership Positions**

Voter Registration Chair, Utah State University Government Relations Council	2012-2013
Mexico Team Lead, Engineers Without Borders- USU	2014-2016
VP Public Affairs, Biological Engineering Club	2014-2015
VP of Outreach, College of Engineering Ambassadors	2015-2016

**Other Positions and Employment**

Undergraduate Research Engineer Sustainable Waste-to-Bioproducts Engineering Center Logan, UT	2013-2016
---	-----------

**Honors and Awards**

Utah State University A-pin Recipient Designates 4.0 GPA for a consecutive calendar year	2012-2014
Outstanding Pre-Professional of the Year (Utah State University, Department of Biological Engineering)	2013
Masters Student Researcher of the Year (Utah State University, Department of Biological Engineering)	2017

**Professional Memberships and Activities**

Engineers Without Borders-USA, member	2014-2016
Institute of Biological Engineering, member	2016-2017

Water Environment Federation, member 2017

### **Teaching Activities**

BENG 2330: Engineering Properties of Biological Materials 2015  
Teaching Assistant

BIOL 1030: Medical College Admissions Test (MCAT) Preparation 2016  
Instructor

BENG 3200: Introduction to Unit Operations in Biological Engineering 2016  
Teaching Assistant

### **Grants**

Research and Graduate Studies, Utah State University 2014  
Principal Investigator (PI): Ronald Sims  
Title: Summer Undergraduate Research and Creative Opportunities Grant  
\$2000  
Role: Student Investigator

Research and Graduate Studies, Utah State University 2016  
Principal Investigator (PI): Ronald Sims, Timothy Taylor  
Title: Summer Undergraduate Research and Creative Opportunities Grant  
\$3000  
Role: Student Investigator

### **Publications**

Hodges, Alan, et al. "Nutrient and suspended solids removal from petrochemical wastewater via microalgal biofilm cultivation." *Chemosphere* 174 (2017): 46-48.

### **Presentations**

#### **National**

Reduction of Greenhouse Gas Emissions from Septic Tanks 2016  
via Compost Biofiltration, Institute of Biological Engineering,  
7 April, Greenville, SC

Cyanobacterial Dominated Biofilm Cultivation in Wastewater 2016  
derived from Petroleum Refining, Algae Biomass Summit,  
23 October, Phoenix, AZ

Novel use for Cement Production Byproducts as Chemical  
Coagulant and Flocculant, Institute of Biological Engineering,  
2 April, Salt Lake City, UT 2017

### **Regional**

Biomass and Therapeutics from Cyanobacterial Biofilms  
Cultured Produced Water (Oilfield Wastewater) at Laboratory  
and Outdoor Scale, Synthetic Biomanufacturing Institute Review,  
10 February, Logan, UT 2014

Transitioning the Benefits of Algal Growth to the Byproducts  
of Oil and Natural Gas Production, Research on Capitol Hill,  
27 January, Salt Lake City, UT 2015

Production of High Value Products from Oil and Natural Gas  
Produced Water Using Algae, Synthetic Biomanufacturing Institute Review,  
12 February, Logan, UT 2015

### **Local**

Algal Growth on Non Potable Water Sources,  
Student Research Symposium,  
17 April, Logan, UT 2014

Effects of Total Dissolved Solids on Algal Culture,  
SURCO research symposium,  
14 September, Logan, UT 2014

The Effects of Sterilization on Mechanical Properties of Polysulfone,  
Student Research Symposium,  
18 April, Logan, UT 2015

### **Community Service**

Access Hospice, Patient Companion 2014-2016

Logan Regional Hospital, Volunteer 2015-2016