AN EXPERIMENTAL INVESTIGATION OF MICROALGAL DEWATERING EFFICIENCY OF BELT FILTER SYSTEM

By

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Submitted to the graduate degree program in Mechanical Engineering and the Graduate Faculty of the University of Kansas in partial fulfillment of the requirements for the degree of Doctor of Philosophy.

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ABSTRACT

Profitable large-scale production of biofuel from microalgae has not yet been demonstrated. A major bottleneck is high operational cost of microalgal harvesting. This is due to small cell size and dilute microalgal suspension. A belt filter system is preferred over other dewatering technologies as it has lower energy consumption. However, a microalgal feed concentration of 10 – 40 g dry wt. /L is required prior to dewatering on a belt filter system. The objective of this study was to investigate the microalgal dewatering efficiency of a belt filter system. A prototype belt filtration system designed for feed concentration of 50 g dry wt. /L was used for this investigation. A mixed laboratory culture of freshwater species dominated by three eukaryotic green microalgae (*Chlorella vulgaris, Scenedesmus sp.,* and *Kirchneriella sp.)* was cultivated in wastewater effluent. Bench-scale gravity filtration tests were conducted to determine the filtration belt mesh needed for the prototype system. Based on the test results a 70 micron mesh size resulted in the highest microalgal recovery rate and was subsequently used for all dewatering tests conducted in this study. Belt dewatering tests conducted on untreated microalgal suspensions – pond water at the KU Field Station and stationary growth phase samples from the microalgal lab culture – resulted in negligible recovery. The highest concentration of microalgal suspension available for testing on the prototype belt filtration system was 6 g dry wt. /L obtained from biomass settling tanks at the Lawrence, Kansas domestic wastewater treatment plant that resulted in 84% biomass recovery. To further investigate this, 54 Liters of 4 g dry wt. /L were produced from bench-scale flocculation using an alum dosage of 200 mg/L at pre-test pH value of 6.5. Results of belt dewatering tests indicated that the percent of microalgae recovered for 4 g dry wt. /L suspension, 46%, was significantly lower than 6 g dry wt. /L suspension. Sealed filter section would likely improve the microalgal

recovery (subsequently reducing the number of filtration passes required for maximum microalgal recovery).

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INTRODUCTION

Background and Motivation

Climate change policy and concerns regarding future energy security have stimulated an unprecedented increase in the production of bioenergy sources that have the potential to reduce future greenhouse gas emissions (Smith et al., 2012). Microalgae are of particular interest because many of the resources required for their mass cultivation can be provided by waste streams (e.g., municipal wastewater: (Sturm & Lamer, 2011); carbon dioxide from industrial flue gas: (Brentner et al., 2011)), and because microalgal cells synthesize many different harvestable bioproducts having a wide variety of compositions and uses (Menetrez, 2012). In particular, microalgae possess many favorable characteristics as a biofuel feedstock, including rapid growth rates and high lipid contents (Chen et al., 2011), high areal energy (Chisti, 2007; Hu et al., 2008), and the ability to avoid undesirable 'food versus fuel' conflicts via the cultivation of microalgal biomass on marginal lands (Singh & Gu, 2010). Nonetheless, profitable large-scale production has not yet been demonstrated (NRC, 2012).

The high operational costs associated with microalgal harvesting are a major challenge (Uduman et al., 2010) due to the very dilute nature of the microalgal suspension and their small cell size (Grima et al., 2003). An optimal harvesting method for microalgae should be independent of the species being cultivated, and should also have a low chemical and energy demand (Amaro et al., 2011).

Specific Aims

The long term goal of this project is to advance the study on harvesting microalgae for biofuel production. Belt filter system is a potential dewatering technology due to its low energy consumption (Grima et al., 2003) and operational costs (Spellman, 1997). Investigators have found that microalgal concentration of 10 - 40 g dry wt. / L is needed prior to dewatering on a belt filter (Grima et al., 2003; Sturm & Lamer, 2011). Goal of this work was to investigate the microalgal dewatering efficiency of belt filter system. The objective of the first study was to design and develop a prototype belt filter system and perform preliminary dewatering tests. The second study investigates dewatering efficiency of belt filter system for microalgal suspensions with concentrations below 10 g dry wt. /L.

Dissertation content

This document contains five chapters. Chapter 1 consists of an introduction to the field of study. Chapter 2 consists of a background of published literature in the field of study. Chapter 3 consists of a manuscript reporting the background, methods and results of the study to design and develop a prototype belt filter system and perform preliminary dewatering tests. Chapter 4 consists of a manuscript reporting the background, methods and results of study to investigate dewatering efficiency of belt filter system for microalgal suspensions with concentrations below 10 g dry wt. /L. Chapter 5 consists of the summary of the body of work.

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CHAPTER TWO: BACKGROUND

2.1 Algal biology

Algae are recognized as one of the oldest life-forms (Falkowski & Raven, 1997). They are thallophytes, i.e. lacking roots, stems and leaves, have *Chlorophyll a* as the primary photosynthetic pigment and lack a sterile covering around reproductive cells (Lee, 2008).

Algae are considered to be a potential biofuel due to its high lipid content – energy storage and structural molecules – than other biofuel feedstock sources (Table 1).

Table 2.1 Comparison of oil yield of biodiesel sources (Chisti, 2007).

Crop	Oil yield (Liter/ hectare)
Corn	172
Soybean	446
Canola	1190
Jatropha	1892
Coconut	2689
Oil palm	5950
Microalgae (70% oil by wt. in biomass)	136,900
Microalgae (30% oil by wt. in biomass)	58,700

There are two basic types of cells in the algae, prokaryotic and eukaryotic. Prokaryotic cells (cyanobacteria) lack membrane-bounded organelles (plastids, golgi bodies and flagella) and

eukaryotic cells have organelles that allow them to survive and reproduce. The three main classes of eukaryotic algae are – green algae (*Chlorophyta*), red algae (*Rhodophyta*) and diatoms (*Bacillariophyta*). Eukaryotic algae are preferred over prokaryotic algae as they have higher lipid content (Williams & Laurens, 2010). Algae can also be classified based on their size (Hossain et al., 2008) – micro or macro – and the medium in which they grow – freshwater or marine. Microalgae are commonly used in the production of biodiesel as they have higher lipid content and are easier and faster to grow compared to macroalgae (Shay, 1993).

Microalgae production mechanisms are autotrophic and/or heterotrophic. For autotrophic microalgae, photosynthesis is a key component of their survival (Falkowski & Raven, 1997; Zilinskas Braun & Zilinskas Braun, 1974). Heterotrophic microalgae require an external source of organic compounds as well as nutrients (Lee, 2008). Mixotrophic microalgae production mechanism integrates autotrophic and heterotrophic processes. Large-scale autotrophic production of microalgae using systems such as ponds and photobioreactors is commonly used. Microalgae cultivated in open ponds or closed photobioreactors optimally yield concentrations on the order of 0.1 and 4 g dry wt. /L solids content. Cultivation is followed by one or two-step harvesting process resulting in sludge that typically has a concentration of 150 – 250 g dry wt. /L. For higher solids content prior to the final step - lipid extraction - dewatering would be followed by drying. Wet solvent extraction is the subject of current research (Levine et al., 2010). Production to processing of microalgae is shown in Figure 2.1.

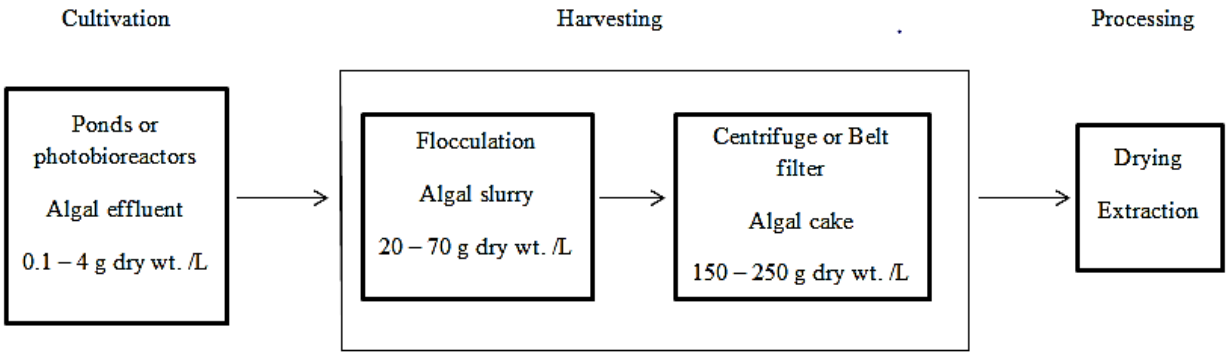

Figure 2.1 Schematic of microalgal production and processing (Shelef et al., 1984).

Profitable large scale production of biofuel from microalgae has not yet been demonstrated (NRC, 2012). A major bottleneck is high operational cost of microalgal harvesting (Uduman et al., 2010). This is due to the small cell size and dilute nature of microalgal suspension. An optimal harvesting method for microalgae should be independent of the algal species being cultivated, and should also have a low chemical and energy demand (Amaro et al., 2011). The goal of this research is to advance the study on harvesting microalgae for biofuel production

2.2 Microalgal harvest methods

2.21 Centrifugation

In this method, centrifugal forces are applied to the solution to aid the separation of solids and liquids. Microalgal solution is added to a bowl spinning at high speed. The spinning action creates a centrifugal force on the solid particles which tend to settle against the wall (Spellman, 1997).

Percent of microalgae recovered depends on the settling characteristics of the cells, slurry residence time in the centrifuge and settling depth (Grima et al., 2003). It is the preferred microalgal recovery method for producing extended shelf-life concentrates for aquaculture hatcheries and nurseries (Grima et al., 2003). Heasman et al. (2000) investigated the extent of cell recovery for three different acceleration factors – 13,000 × g, 6000 × g and 1300 × g for 10 microalgal species. Percent of microalgae recovered were > 95% at 13,000 × g. Decrease in acceleration resulted in lower microalgal recovery. Although centrifugation is a highly effective method for harvesting microalgae, it has a high energy demand and is expensive. Also, the exposure of microalgal cells to high gravitational and shear forces can damage cell structure (Knuckey et al., 2006).

2.2.2 Flocculation

Microalgal cells form stable suspensions due to – (1) Small cell size; (2) Low specific gravity; and; (3) Negative surface charge causing intracellular repulsion forces. In order to neutralize the charge, cationic flocculants are added to microalgal suspension to facilitate aggregation. An ideal flocculant would be non-toxic, inexpensive and effective in low concentration (Grima et al., 2003). Flocculation can also be used as a pre-treatment step prior to harvesting using other methods. Flocculation consists of rapid mixing of coagulant followed by flocculation or slow mixing where the particles agglomerate to form flocs (Davis & Cornwell, 1998). The final step is decantation (Figure 2.2).

Figure 2.2 Step by step representation of microalgal flocculation (Granados et al., 2012).

2.2.2.1 Inorganic flocculants

Inorganic flocculants (alum, ferric chloride, etc.) have been widely used in wastewater treatment. The mechanism of flocculating particles is charge neutralization where negative charge on the microalgal surface is cancelled due to adsorption of positively charged flocculant (Davis & Cornwell, 1998). Clarification efficiency of inorganic flocculant increases with increase in ionic charge and is highly sensitive to pH. Demerits of the process are the high dosage requirements and possible cell lysis as in the case of aluminum salts (Papazi et al., 2009). Also, residual metal salts may negatively affect the medium reuse and the quality of desired product (Estevez et al., 2001; Mojaat et al., 2008).

2.2.2.2 Organic flocculants

In contrast to inorganic flocculants, coagulation using organic biodegradable flocculants such as chitosan and grafted starch is less sensitive to pH and have lower dosage requirements (Tenney et al., 1969). In addition to reducing or neutralizing the surface charge on cells, organic flocculants bring particles together by physically linking one or more particles through a process

called bridging (Tenney et al., 1969). Factors affecting flocculation performance are polymer molecular weight, charge density of molecules, dosage, microalgal concentration, mixing intensity, ionic strength and pH of the suspension (Grima et al., 2003). The raw material cost of the organic flocculants needs to be further reduced to become a commercially viable option for harvesting microalgae.

2.2.2.3 Autoflocculation

Autoflocculation is the phenomenon of chemical flocculation of microalgal cells in the presence of calcium and magnesium ions at high pH (Vandamme et al., 2012). Study conducted by Vandamme et al. (2012) investigated different methods to induce autoflocculation of the microalga *Chlorella vulgaris*. Study results indicated a 50-fold increase could be achieved using calcium hydroxide with both a low cost and a low environmental risk. The effects of both the base used for flocculation and the acid used for pH neutralization on the economic feasibility and the environmental impact of the process should be considered (Wu & Ye, 2007).

2.2.2.4 Bioflocculation

Spontaneous flocculation assumed to be caused by extracellular polymer substances in the medium is called bioflocculation (Larkum et al., 2012). The main advantage of this method is that it is chemical free. Additionally, the use of bioflocculant enhanced the growth rate of microalgae in a recycled medium, whereas the growth activity was inhibited when a cationic salt was applied alone (Zheng et al., 2012). Whole microbes such as microalgae (Salim et al., 2011),

fungi or bacteria could also be used for bioflocculation. This is commonly seen in wastewater where microalgae and bacteria grow together in the presence of carbon source forming flocs that can be harvested. In the study conducted by Zhou et al. (2013), a filamentous pellets-forming fungal strain (*A. oryzae*) was isolated from municipal wastewater sludge successfully. With continuous agitation provided, microalgae and fungal strain *A. Oryzae* grown in the same culture formed fungus-algae pellets. However, using bacteria or fungi as bioflocculants may result in microalgal contamination.

2.2.3 Filtration

Filtration and screening processes both separate solids from liquids by passing a suspension through permeable medium that retains the solids (Shelef et al., 1984). In this method, the suspension is passed through a screen with a specific aperture size. Particles either collect on the surface or flow through according to their size. The two main types of screening devices are microstrainers and vibrating screen filters.

Microstrainers consist of rotary drum with fine mesh filters and a continual backwash. They have several advantages, such as simplicity in function and construction, easy operation, low investment, negligible abrasion as a result of absence of quickly moving parts and high filtration ratios (Chen et al., 2011). The efficiency of the microstrainers depends on the microalgal concentration. A higher microalgae concentration can result in blocking of the screen, while a low microalgal concentration can result in insufficient capture (Wilde et al., 1991). The two

major limitations of microstrainers are incomplete solids removal and difficulty in handling solids fluctuations.

Filtration requires a pressure drop to be applied across the system in order to force the fluid through the filter. The extent of the pressure required for the medium determines which type of driving force is used: gravity, vacuum or pressure (Shelef et al., 1984). Some of the commonly used pressure and vacuum filters are vacuum drum filter, suction filter, chamber filter and belt filter (Table 2.1). Among these filters, belt filter was found to have the lowest energy consumption and highest concentration factor with a continuous mode of operation. Filtration methods were seen to be effective for large celled microalgal species (> 70 microns) but were inefficient for algal species with a cell size less than 30 microns (Mohn, 1980). The major drawback of this method is membrane fouling.

Table 2.2 Microalgal harvesting performance of pressure and vacuum filters (Grima et al., 2003; Mohn, 1980)

Type	Machine and Make	Operational mode	Concentration procedure	Suspended Solids (%) in concentrate	Energy (kWh)	Relative harvesting cost	Reliability
Pressure Filter	Netzch Chamber Filter; Netzch	Discontinuous	One step	22 - 27	0.88	0.4	Very high
	Belt filter; Bellmer	Continuous	Needs preconcentration to ~ 4% total suspended solids	18	0.5	1.1	
	Suction filter	Discontinuous	Original and pre-concentrated suspension	16			Good
	Cylindrical sieve rotators; Englesmann	Continuous	One step and for pre-concentration	7.5	0.3	1.9	Sufficient
	Filter Basket; Seitz Dinglinger	Discontinuous	For pre-concentration	5	0.2	0.48	Good
Vacuum Filter	Non precoat vacuum drum filter; Dorr Oliver	Continuous	One step	18	5.9	3.9	Low
	Potato starch precoat vacuum drum filter; Nivob, Walther	Continuous after precoating	Needs 2- to 15-fold pre-concentration	37			Good
	Suction filter	Discontinuous	One step	8	0.1	4.5	Satisfactory
	Belt filter; Dinglinger	Continuous	For pre-concentration	9.5	0.45	0.88	Good
	Filter thickener; Schenk	Discontinuous	For pre-concentration	5-7	1.6	3.2	Satisfactory

Does not include labor. Relative harvesting costs are calculated on the basis of operational cost of a self-cleaning plate separator being 1.0.

In cross-flow filtration, the retentate is recirculated across the membrane, keeping the cells in suspension and minimizing fouling (Uduman et al., 2010) (Figure 2.3). The two types of membranes that are used for this process are microfiltration membrane and ultrafiltration membrane. Zhang et al. (2010) found that ultrafiltration concentrated an algal culture by 150-fold under conditions of pulsated air scouring combined with backwashing. An integrated system composed of a ceramic tubular membrane and a hollow fiber membrane accomplished 99% media recovery and concentrated the biomass by 100-fold using a low energy input (Bhave et al., 2012). Membrane replacement and pumping limit large scale harvesting by cross-flow filtration (Chen et al., 2011).

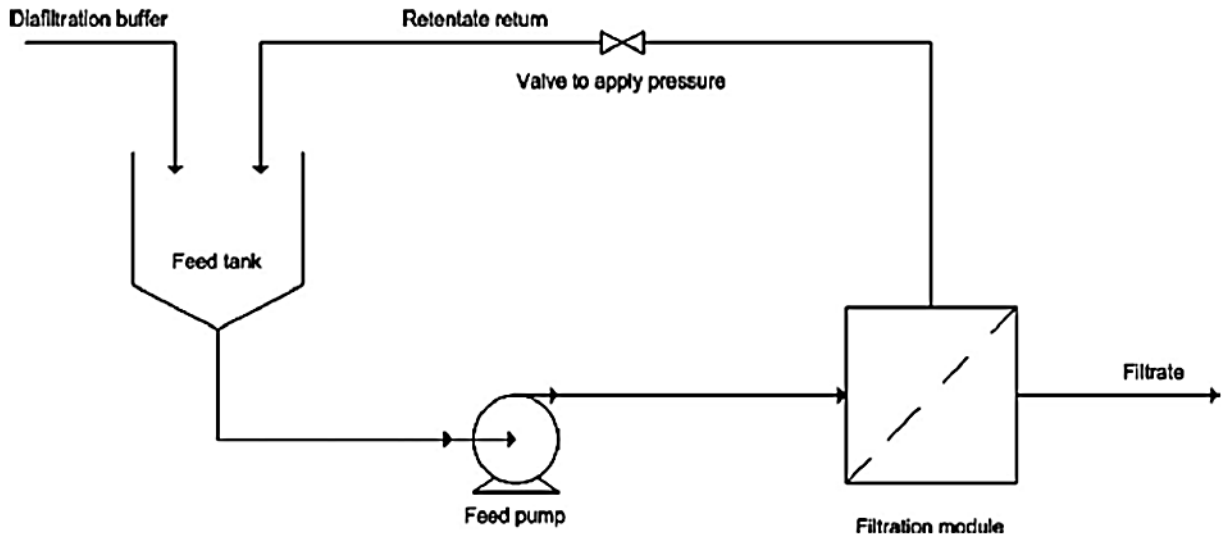

Figure 2.3 Schematic of cross-flow filtration unit (Uduman et al., 2010).

Dynamic cross-flow filtration is an improvement over cross-flow filtration because this method uses the turbulence over the membrane filter to generate higher shear stress on the membrane surface (Brou et al., 2002; Torras et al., 2009). It also reduces the expense associated with the

equipment and membrane replacement (Rios et al., 2010). The major limitation of this method is its high energy demand.

2.2.4 Gravity sedimentation

Gravity sedimentation is a widely used separation technique in wastewater treatment processes. Factors affecting particle settling velocity of untreated microalgae (given by Stoke's law) are gravity force, particle diameter, density of medium, density of particle and medium viscosity. Lamella separators and sedimentation tanks are used for gravity sedimentation. Gravity sedimentation results in high microalgal harvesting efficiency only when preceded by flocculation. A recent study conducted by Wang et al. (2013) evaluated a downward flow inclined gravity settler for its effectiveness in dewatering *Scenedesmus dimorphus* and *Chlorella vulgaris*. Results showed 72% efficiency in biomass recovery and low operating costs. Application of this technology on an industrial scale is yet to be proven.

2.2.5 Flotation

Flotation is a gravity separation process in which gas bubbled through a microalgal suspension gets attached to the particles and carry them to the surface and accumulate as float which can then be skimmed off (Shelef et al., 1984). Separation efficiency of process is inversely related to bubble size and instability. Hanotu et al. (2012) emphasized that small bubbles take longer time to rise making them more susceptible to aggregate with the microalgae particles compared to large bubbles. Particle diameters from 10 µm to 500 µm can be used for flotation. Chen et al.

(1998) noted that flotation was more beneficial in microalgal removal than sedimentation. The three flotation techniques are dissolved air flotation, dispersed air flotation and electrolytic flotation.

2.2.5.1 Dissolved air flotation

Of the three flotation techniques, dissolved air flotation is the most commonly used (Matis, 1995). This method involves dissolving air in water under pressure which is then released at atmospheric pressure in the flotation tank. The released air bubbles aggregate with the microalgae particles. These floating aggregates are later skimmed off (Figure 2.4). Factors determining effectiveness of DAF harvesting of microalgae include the pressure of the tank, recycle rate, hydraulic retention time, and floating rate of the particle (Chen et al., 2011). High microalgal recovery rates were obtained only when combined with flocculation (Henderson et al., 2010). Autoflotation of microalgae by photosynthetically produced dissolved oxygen after flocculation with alum was studied by Bare et al. (1975). Results showed 80-90% microalgal recovery with the microalgal float concentrations reaching 6%. However this method requires a pre-concentration of at least 16 mg/L. Recent studies (Cheng et al., 2010; Henderson et al., 2010) found ozone flotation to improve the process efficiency as they release polymers suggested to be biopolymers through cell lysis during flotation that make the bubble surface more hydrophobic.

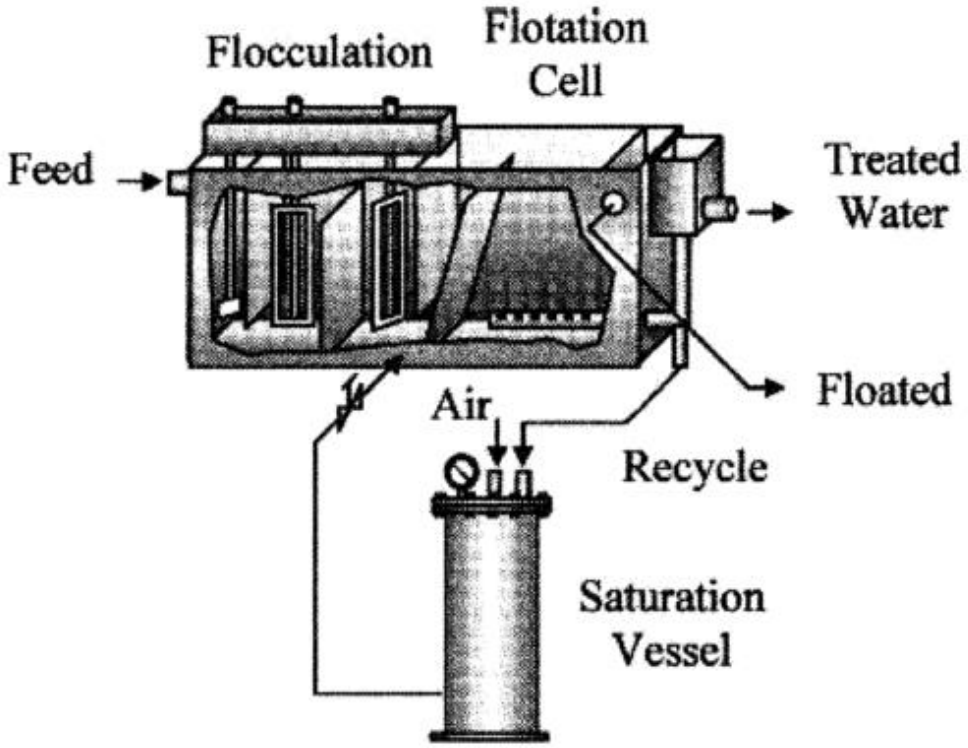

Figure 2.4 Schematic of dissolved air flotation unit (Rubio et al., 2002). Water stream pre-saturated with dissolved air is introduced into the system containing flocculated microalgal culture. The air bubbles aggregate with the microalgal cells and float to the surface.

2.2.5.2 Dispersed air flotation

This method involves the formation of bubbles by a high speed mechanical agitator and an air injection system. Gas introduced at the top is mixed with liquid and allowed to pass through a disperser, which creates bubbles ranging from 700 to 1500 μm in diameter (Rubio et al., 2002). Chen et al. (1998) studied dispersed air flotation for removal of *Scendesmus quadricauda* from water using three types of surfactants - nonionic X-100, cationic N-Cetyl-N-N-N-trimethylammonium bromide, and anionic sodium dodecylsulphate. Surfactants prepare the

surface of the microalgal particles for flotation by changing its hydrophobicity, which improves microalgal-bubble attachment (Phoochinda et al., 2004). Dispersed air flotation was successful for the microalgal removal using cationic CTAB.

2.2.6 Electrophoreses techniques

Electrolytic harvesting methods involve removal of microalgal particles from water-based medium solutions by movement in an electric field (Aragón et al., 1992). There are several benefits to using electrochemical methods, including environmental compatibility, versatility, energy efficiency, safety, selectivity, and cost effectiveness (Mollah et al., 2004). Compared to other harvesting methods, electrolytic methods were seen to be more effective for harvesting marine microalgal species. The high ionic strengths induced high conductivity that improved the overall efficiency of the process. However, high concentrations of residual chlorine ions lower medium reusability and cell viability (Kim et al., 2012)

2.2.6.1 Electrolytic coagulation

In this method, cations such as Al^{3+} and Fe^{3+} produced from the anode react with water to form positively charged metal hydroxides which then aggregate with the negatively charged microalgal surface. Study conducted by Azarian et al. (2007) showed that microalgal removal efficiency increased with an increase in electrical power but may result in deterioration of process stability.

2.2.6.2 Electrolytic flocculation

In this method, negatively charged microalgae move towards the anode and lose their charge upon reaching it. This leads to the formation of aggregates (Uduman et al., 2010). Poelman et al. (1997) reported 80 to 95% microalgal recovery using electrolytic flocculation in 35 minutes. Decreasing the voltage reduced energy consumption but also led to slower microalgal removal rate.

2.2.6.3 Electrolytic flotation

In this method, the cathode is made from an inactive metal that generates hydrogen bubbles from water electrolysis. The air bubbles aggregate with the microalgae particles (Alfafara et al., 2002; Azarian et al., 2007). Results of study conducted by Alfafara et al. (2002) indicated an increase in microalgal recovery with increase in electrical power. The two main disadvantages of this process include scaling of the cathode and high cost of power rectifiers.

2.2.7 Ultrasound

Bosma et al. (2003) investigated acoustically induced aggregation followed by enhanced sedimentation as a potential microalgal harvesting method. This is a non-fouling harvesting method that can be operated continuously without inducing shear stress preserving the structure and properties of microalgal cells. Factors determining process efficiency were feed flow rate, microalgal concentration and ratio between flow throughput and feed flow rate. Scale-up of this technology is limited by the high energy costs.

2.2.8 Magnetic Separation

In this method the microalgal cells are separated from the liquid suspension by the functional magnetic particles driven by an external magnetic field (Haukanes & Kvam, 1993; Li et al., 2009; Qu et al., 2009; Wang et al., 2007; Wang et al., 2008; Yang et al., 2008). Study carried out by Xu et al. (2011) found that 98% of the microalgal cells were adsorbed and then separated by an external magnetic field using naked Fe_3O_4 particles. A recent study conducted by Hu et al. (2013) where naked Fe_3O_4 particle were used for harvesting marine microalga recovered 95%. Factors affecting process efficiency were pH, nanoparticle dosage and microalgal growth phase of culture medium. Advantages of magnetic separation are the relatively short time periods for harvesting microalgae and the reusability of the culture medium (Hu et al., 2013; Xu et al., 2011).

Summary of performance of the harvesting techniques are listed in Table 2.3.

Table 2.3 Advantages and limitations of microalgal harvest methods

Harvesting method	Advantages	Limitations
Centrifugation	Highly effective on dilute microalgal suspension	1. High energy demand 2. The exposure of microalgal cells to high gravitational and shear forces can damage cell structure
Inorganic flocculation	Highly effective on dilute microalgal suspensions	1. High dosage required 2. Negatively affect the medium recycling and fuel composition
Organic flocculation	1. Biodegradable 2. Lower dosages required compared to inorganic flocculants	High raw material cost
Autoflocculation	Low cost and low environmental risk	Not yet proven on an industrial scale
Bioflocculation	Chemical free method	Not yet proven on an industrial scale
Filtration: Pressure and vacuum devices	Belt filter – low energy consumption	Membrane fouling
Cross-flow filtration	Low fouling rates	High energy demand
Gravity sedimentation	Low energy consumption	High microalgal recovery rates only when combined with flocculation
Flotation	More effective than sedimentation	High microalgal recovery rates only when combined with flocculation
Electrophoreses techniques	Chemical free method	Cathodes prone to fouling
Ultrasound	1. Non-fouling harvesting technique 2. Preserves the structure and properties of microalgal cells	Scale-up limited by high energy costs
Magnetic separation	1. Short time period for microalgal harvesting 2. Reusability of the culture medium	Not yet proven on an industrial scale

Each harvesting method has its advantages and limitations. Combining two or more harvesting methods can improve the overall efficiency of the process (Table 3).

Table 2.4 Performance of two-step microalgal harvesting methods

Primary harvesting step	Secondary dewatering step	Performance	Reference
Bioflocculation	Centrifugation	Centrifugal energy consumption reduced by 90%	Salim et al. (2011)
Organic flocculation; chitosan	Filtration	Reduced cost of filter material, required processing time and energy input	Xu et al. (2013)
Magnetically induced submerged membrane filtration system	Centrifugation	Lowered centrifugal energy consumption and reduced the processing volume by > 90%	Bilad et al. (2012)
Electroflocculation	Dispersed air flotation	98.9% recovery efficiency	Xu et al. (2010)

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CHAPTER THREE: DESIGN AND DEVELOPMENT OF A PROTOTYPE BELT FILTER SYSTEM

3.1 Introduction

The objective of the study was to design and develop a prototype belt filter system and perform preliminary dewatering tests. Belt filter system is a potential dewatering technology due to its low energy consumption (Grima et al., 2003) and operational costs (Spellman, 1997). Belt filter system is a widely used sludge dewatering technology. A standard belt filter consists of a belt passing over a number of rollers. Dewatering mechanism consists of gravity drainage followed by compression shear where the feed is sandwiched between the primary and secondary belt. Cake collected on the filter is approximately 30% (w/v) (Sturm & Lamer, 2011).

Biodiesel production of wet microalgal biomass at bench-scale was recently reported (Levine et al., 2010). The desirable % solids content would depend on the lipid extraction method. For this study, the solids content required for oil extraction was assumed to be 90% total suspended solids (Lardon et al., 2009) and hence, the need for drying. The four main drying methods are flash drying, freeze drying, drum drying and air/sun drying. Flash drying is a method of spraying dewatered product into hot air stream. This method removes moisture rapidly but can also lead to the deterioration of microalgal pigment (Desmorieux & Decaen, 2005). Freeze drying removes moisture present in the material by lowering the pressure to directly convert the water to gas. However, the major drawback of this method is that it is too expensive for use in large-scale commercial recovery of microalgal products (Grima et al., 2003). Drum drying involves applying the material to be dried on a heated drum, and later scraped off. This method

produces low quality products (Pushparaj et al., 1993). Sun drying is used to dry the material by exposing it to direct solar radiation. Quality of the end product is highly dependent on the weather conditions (Shelef et al., 1984). Due to its low energy and cost requirements, air/ sun drying was the chosen method for this study.

3.2 Material and methods

For this study, a prototype belt filter system was designed and developed. A mixed culture of microalgal species dominated by three eukaryotic green algae (*Chlorella vulgaris, Scenedesmus sp.,* and *Kirchneriella sp*.) was cultivated in domestic wastewater effluent from the Lawrence, Kansas wastewater treatment plant. To determine the needed filtration belt mesh for the prototype system, bench-scale gravity filtration tests were conducted on microalgal samples at their stationary growth phase, 1.5 ± 0.3 g dry wt. /L. These tests used a range of polyester mesh sizes from 10 to 200 microns. Belt dewatering tests were conducted on microalgal suspensions – pond water at the KU Field Station, stationary growth phase samples from the microalgal lab culture and microalgal suspension collected from the settling tanks at Lawrence, Kansas wastewater treatment plant.

3.2.1 Design and development of belt filter system

A prototype belt filter system was designed and developed (Figure 3.1). The design was based on filtration tests conducted on feed concentration of 50 g dry wt. /L microalgal suspension (Appendix A). One million gallons per day of raw 3 g dry wt. /L pond water filtration is the equivalent of 12.5 tons of dried algae output. This translates to 60,000 gallons of 50 g dry wt. /L microalgae solution. The prototype is a 1% scale of a system proposed to process 60,000 gallons of 50 g dry wt. /L microalgal suspension. The filtered product was air dried on the belt over a period of time. A doctor blade was installed at the end of the drying section to scrape off the microalgal cake. The prototype was used as a test bed for further development of the process. Ultimately providing the information needed for a full scale system.

3.2.2 Microalgae cultivation

For all tests conducted in the study, the mixed-species microalgae were cultured in a 272 Liter glass photobioreactor with an operating volume of 208 Liters. This photobioreactor was initially filled with pre-chlorination wastewater effluent collected from the secondary treatment stage of the Lawrence, KS, wastewater treatment plant. Then an inoculum was added that was comprised of a natural mixed species assemblage of three eukaryotic green algae - *Chlorella vulgaris, Scenedesmus sp.,* and *Kirchneriella sp*. (Figure 3.2). 650 grams of inorganic nitrogen (supplied as KNO_3) and 160 grams of inorganic phosphorus (supplied as KH_2PO_4) were added to the photobioreactor and replenished on a weekly basis to provide nutrients for the growing

microalgal community. Light was provided by LED light panels (~ 265 µmol/ [m^2s]) with a 12 hour on, 12 hour off light: dark cycle.

Because wastewater effluent typically contains insufficient inorganic carbon for optimal microalgal growth (Benemann et al., 2003), commercial-grade CO_2 was bubbled into the photobioreactor. The water column pH in the photobioreactor was controlled using a pH controller (Milwaukee Instruments, MC122) to regulate the flow of CO_2. For this experiment the pH of the photobioreactor was set at 6.5 and the room temperature was maintained at 23 $\pm$ 1℃. To provide turbulent mixing, room air was bubbled into the tank at a rate of 4.6 Liters/minute using four aerators placed at each of the four corners of the tank. This turbulent mixing helped to maintain the microalgal cells in suspension during cultivation. Microalgal biomass measurements were made at different stages of post-inoculation growth using a calibrated UV/Vis Spectrophotometer (Thermo Fisher Scientific Model G10S) followed by a standard total suspended solids test (Becker, 1994). Typically the microalgal cells reached their stationary growth phase 8 days after inoculation.

3.2.3 Gravity filtration tests

The purpose of this test was to determine the filter mesh size that resulted in the highest microalgal biomass recovery rate (g m^{-2} s^{-1}) was calculated using equation (1):

$$\text{Biomass recovery rate} = \frac{W_{cake}}{FA \times FT} \qquad (1)$$

where W_{cake} is the mass of the wet microalgal cake collected on the filter in grams, FA is the filter area (1.7×10^{-3} m^2), and FT is the filtration time in seconds.

Samples of microalgal suspension with biomass concentration of 1.5 $\pm$ 0.3 g dry wt. /L were used for the gravity filtration tests, which were conducted for a range of mesh sizes (all Polyester) from 10 to 200 microns. The test setup comprised of a Sigma-Aldrich vacuum filter assembly for 47 mm Whatman GF/C glass filter with glass support (Product # Z290432). 25 milliliters of microalgal suspension was filtered, and the required filtration time was recorded. The filtration assembly was then disassembled and filter was removed using forceps. The microalgal cake was carefully scraped off of the filter and collected in a pre-weighed aluminum weigh boat; the wet weight of the microalgal cake was recorded. Each filtration test was conducted five times.

3.2.4 Belt dewatering test procedure

Belt Dewatering tests were conducted on microalgal suspensions – pond water at the KU Field Station, stationary growth phase samples from the microalgal lab culture and flocculated suspension from the settling tanks at Lawrence, Kansas wastewater treatment plant.

The filter area consisted of a mesh screen (MD Building Products 1 ft. x 2 ft. Aluminum Albras Lincane Sheet – Model # 56012). Microalgal suspension was pumped into the filter section of the system. The belt filter mesh used in this testing was the 70 micron polyester mesh identified in the earlier filtration testing (Figure 3.3A). The belt speed, on the dewatering system, was set at 0.7 millimeters per second – slowest belt speed on the system. This was done to improve the recovery for dilute microalgal suspensions. The depth of the microalgal solution in the filter section was controlled by a level sensor driving a pumping system (Figure 3.3B). After leaving

the filtering section on the belt dewatering system the microalgae was allowed to air dry on the belt. Then the microalgal cake was scraped off manually and the weight was recorded. The percent of dried microalgae recovered was calculated from the following equations:

The percent recovery (PR) of microalgae recovered was calculated using equations 3 and 4 below:

$$PR\ (\%) = \left(\frac{M_D}{M_I}\right) \cdot 100 \qquad (2)$$

$$M_I = \frac{C_I \cdot V_I}{10^3} \qquad (3)$$

where M_D is the recovered mass of the dried microalgae (in grams); M_I is the initial total suspended solids mass in the microalgal suspension (in grams); C_I is the initial concentration of the microalgal solution (in milligrams dry weight/Liter); and V_I is the filtered volume of microalgal solution (Liters).

3.3 Results and Discussion

3.3.1 Microalgal cultivation results

Microalgal culture in the 272 Liter glass photobioreactor achieved a concentration of 1.5 $\pm$ 0.3 g dry wt. /L at the stationary growth phase in 8 days (Table 3.1). Studies on closed photobioreactors such as tubular or flat plate (systems) have reported biomass concentrations on the order of 2 g/L with a maximum of 5 g dry wt. /L (Pulz, 2001). The lower biomass productivity in this study was probably due to reduced light intake caused by the photobioreactor structure.

3.3.2 Determination of filter mesh size

The effectiveness of the belt filter system is measured by microalgal biomass recovery rate. The biomass recovery rate depends on various factors such as filter feed rate, belt speed (Spellman, 1997) and mesh size. For this study, mesh size was the only parameter that was considered due to lack of sufficient volume of concentrated microalgal suspension. The mesh size that resulted in the highest recovery rate was chosen. Based on the test results, microalgal recovery rate for 70 micron mesh size was significantly higher than the recovery rates for all other mesh sizes (see Figure 3.4, $n = 5$, one-way ANOVA, $p < 0.05$). Microalgal biomass recovery rate was assumed to be independent of the concentration of the microalgal suspension and the feed volume.

3.3.3 Preliminary belt dewatering test results

Percent of microalgae recovered using 70 micron polyester filter belt is shown in Table 3.2. Low microalgal recovery from belt filter dewatering tests conducted on untreated microalgal suspensions indicated a need for pre-concentration prior to dewatering. This is consistent with the findings of previous studies on belt filter dewatering that indicated a need for feed suspension concentration of 10 – 40 g dry wt. /L (Grima et al., 2003; Sturm & Lamer, 2011). The highest concentration of microalgal suspension available for testing on the prototype belt filtration system was 6 g dry wt. /L obtained from biomass settling tanks at the Lawrence, Kansas domestic wastewater treatment plant. For a biomass concentration of 6 g dry wt. /L, 84 % of the microalgae were recovered after six successive dewatering tests. 76% of the microalgae were recovered in two successive filtrations. Multiple filtration passes were required due to leakages in the filter section of the system.

3.4. Conclusions

The objective of this study was to design and develop a prototype belt filter system and perform preliminary dewatering tests. A prototype belt filter system was designed and developed. Preliminary dewatering tests conducted on untreated microalgal suspension resulted in negligible recovery. The 6 g dry wt. /Liter microalgal suspension yielded a maximum of 84% recovered microalgae. The results of this study indicate that microalgal suspension with concentrations as low as 6 g dry wt. /L can be effectively recovered with a belt filter system. The next step in this line of research is to further investigate dewatering efficiency of belt filter system for microalgal suspensions with concentrations below 10 g dry wt. /L.

3.5 References

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Table 3.1 Optical density and biomass concentration measurements of microalgal culture over a cultivation period of 8 days.

Culture time (days)	**OD_{600nm}**	**Biomass concentration (g dry wt. /L)**
2	5.4 $\pm$ 0.45	0.7 $\pm$ 0.09
4	8.2 $\pm$ 1.6	1.1 $\pm$ 0.3
6	11.3 $\pm$ 0.5	1.45 $\pm$ 0.1
8	12.5 $\pm$ 1.5	1.5 $\pm$ 0.3

Table 3.2 Belt dewatering test performance – untreated versus treated microalgal supension. Belt dewatering tests were conducted using 70 micron mesh size filter on sample suspensions to determine the percent of microalgae recovered (mean ± standard deviation, n = 3).

	Microalgal suspension concentration	Feed Volume	Cumulative microalgae recovered
	(grams/Liter)	(Liters)	(%)
KU Field Station – untreated pond water	0.045	150	negligible
Microalgal lab culture	1.5 ± 0.3	170	6
WWTP settling tanks[a]	6 ± 0.1	6	84.7 ± 0.06

[a] 6 consecutive belt dewatering tests were conducted

Figure 3.1 Design and development of belt filter system - A 3D CAD drawing in Autodesk Inventor 2011.

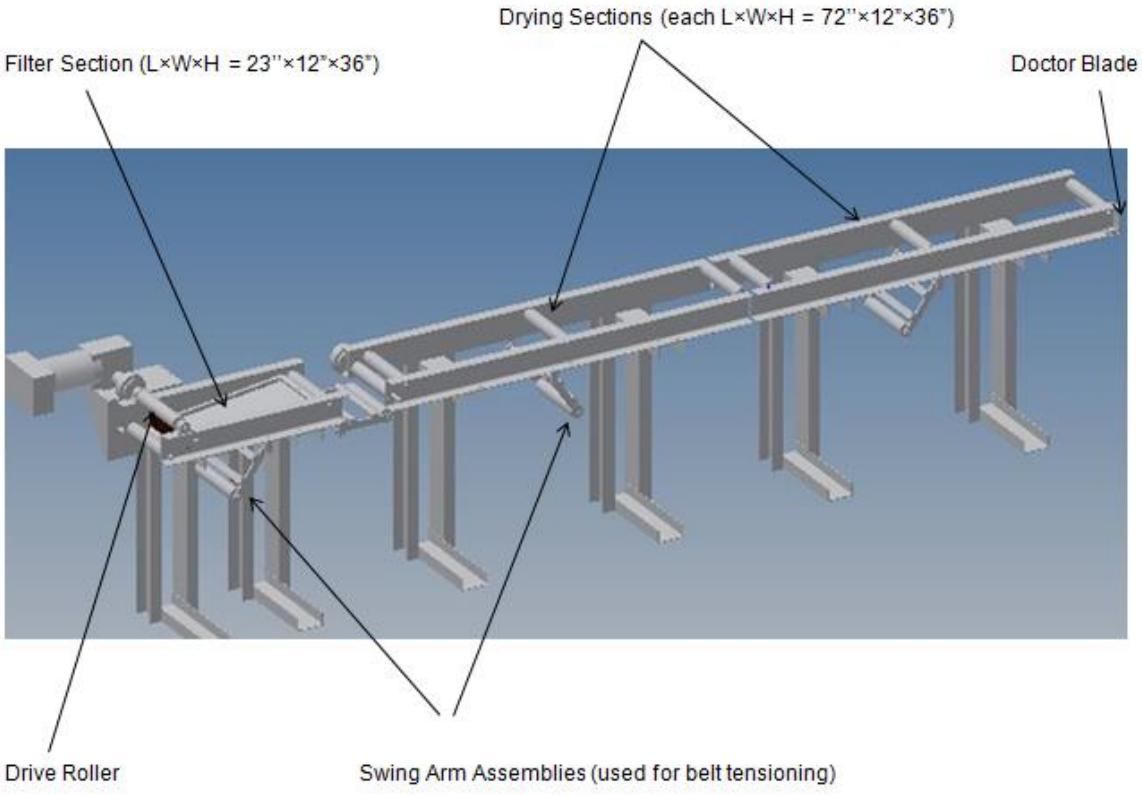


Figure 3.2 Electron micrograph images of microalgal species cultivated in the photobioreactor. Magnification factor = 40*x*.

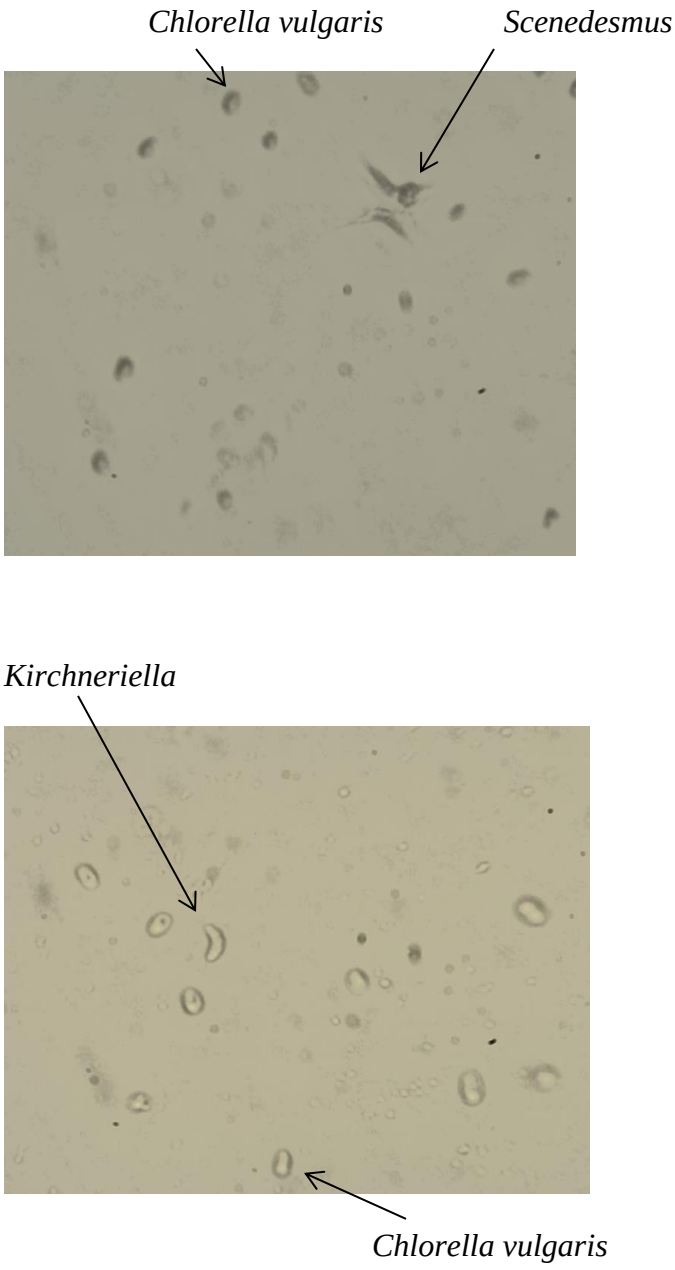


Figure 3.3 Belt filter dewatering test set-up

A) Prototype belt filter dewatering system

B) Filter section with level sensor driving the pumping system.

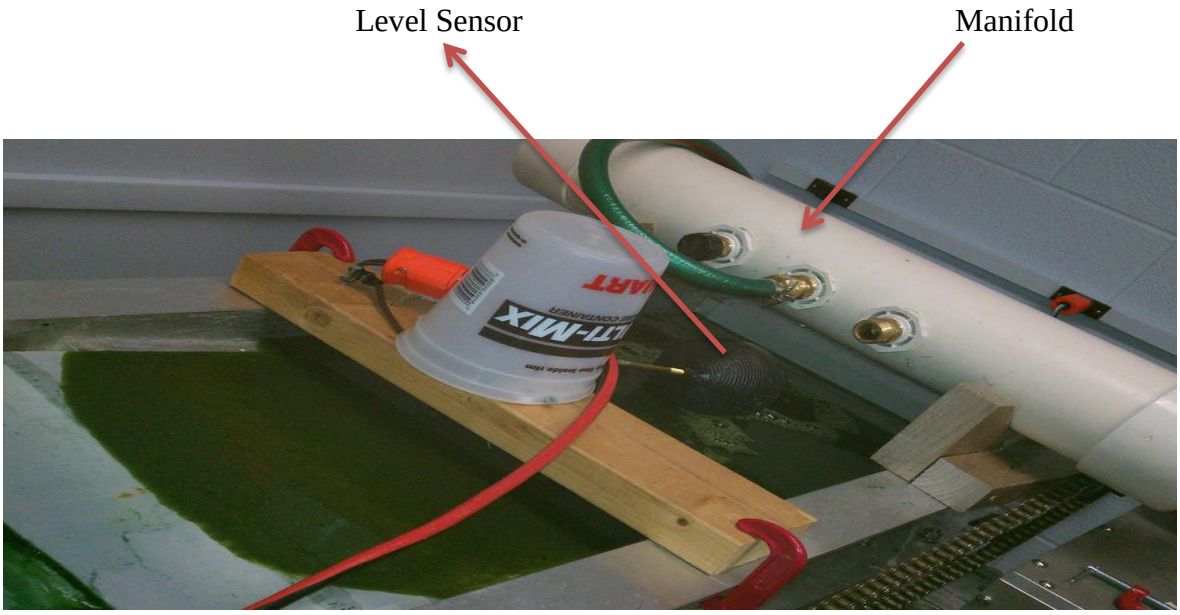
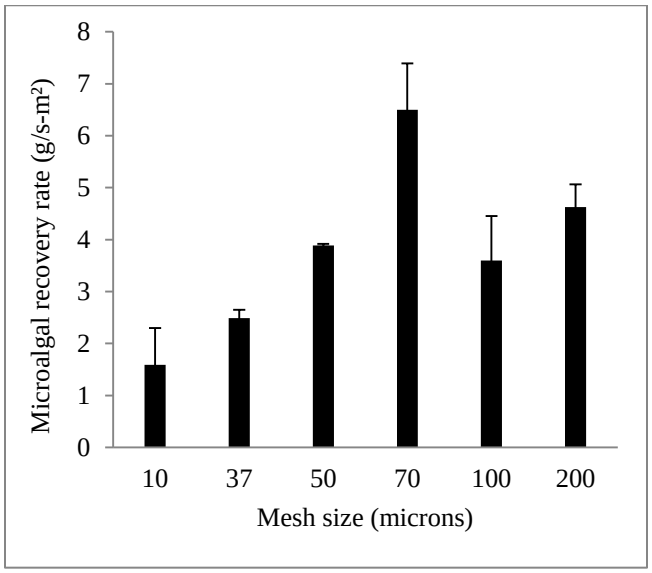


Figure 3.4 Determination of mesh size with the highest microalgal biomass recovery rate.



Error bars represent the standard deviation (n = 5) of measured values of microalgal recovery rate. Microalgal recovery rate for the filter with a mesh size of 70 microns was significantly higher than the recovery rates for all other mesh sizes (One-way ANOVA, $p < 0.05$).

CHAPTER 4: BELT FILTER DEWATERING OF TREATED MICROALGAL SUSPENSIONS WITH CONCENTRATION BELOW 10 G DRY WT. /L

4.1 Introduction

Investigators have suggested that microalgal concentration of 10 - 40 g dry wt. / L is needed prior to dewatering on a belt filter (Grima et al., 2003; Sturm & Lamer, 2011). To further investigate this, a prototype belt filter dewatering system was designed and developed by the authors (Chapter 3 - Figure 1). The design was based on filtration tests conducted on 50 g dry wt. /L microalgal suspension. The prototype is a 1% scale of a system proposed to process 60,000 gallons of 50 g dry wt. /L microalgal solution per day. Gravity filtration tests were conducted on microalgal samples at their stationary growth phase to determine the filtration belt mesh needed for the prototype system. These tests used a range of polyester mesh sizes from 10 to 200 microns. Based on the test results a 70 micron mesh size resulted in the highest microalgal recovery rate (Chapter 3 - Figure 2).

The highest concentration of microalgal suspension available for testing on the prototype belt filter system was 6 g dry wt. /L obtained from biomass settling tanks at the Lawrence, Kansas domestic wastewater treatment plant. Dewatering tests of this suspension, using 70 micron polyester filter belt, resulted in 84% biomass recovery. The results of this initial testing suggested that concentrations of microalgal suspensions less than 10 g dry wt. /L could be recovered. This led to the current experimental investigation of belt dewatering on microalgal suspensions with concentrations less than 10 g dry wt. /L.

4.2 Materials and methods

For this study, flocculant type, dosage and pH that were the most efficient and cost-effective for the microalgal suspension (Chapter 3 – Section 2.2) were determined using jar tests. The results of the jar tests were then used to prepare sufficient volume of concentrated microalgal suspension for the belt filtration testing. A total of 54 liters of 4 g dry wt. /L microalgal suspension were produced and dewatered on the belt filtration system with a 70 micron polyester filter mesh.

4.2.1 Flocculation tests

Three different flocculants were chosen for testing – chitosan powder, aluminum potassium sulphate dodecahydrate and zetag 7650. Both chitosan powder and aluminum potassium sulphate dodecahydrate (an inorganic cationic flocculant), were obtained from Sigma-Aldrich Company Ltd. (Missouri, USA). Zetag 7650, a high molecular weight synthetic cationic polymer used for sludge dewatering (Danquah et al., 2009), was obtained from Southwest Engineers (Louisiana, USA).

Jar tests were conducted to determine the flocculation conditions (flocculant type, dosage and pH) that were the most efficient and cost-effective. Three flocculant mixtures were evaluated: (1) Aluminum sulfate (Alum) alone; (2) Alum combined with zetag 7650 (10:1 by mass); and (3) Alum combined with chitosan (10:1 by mass). Stock solutions for each of the three flocculants - chitosan (Divakaran & Pillai Sivasankara, 2002), alum, and zetag 7650 (Tillman, 1996) - were prepared at concentrations 10 g/L, 1 g/L and 1 g/L, respectively. Jar tests were then performed

on a multi-jar magnetic stirrer using 500 mL samples with a biomass concentration of 1.5 $\pm$ 0.3 g dry wt. /L taken from the photobioreactor. Flocculation was conducted for a range of pH and dosage values for each of the three flocculant mixtures (Figures 4.1 and 4.2). Pre-test pH values of the microalgal samples were adjusted using 0.1M NaOH or 0.1 M HCl. The desired flocculant mixture was added to the microalgae samples and mixed rapidly at 100 rpm for 60 seconds, followed by slow mixing at 60 rpm for 15 minutes. After flocculation, the suspension was allowed to settle for a period of 30 minutes. Flocculant performance was then evaluated as clarification efficiency (Equation 1).

$$\text{Clarification Efficiency (\%)} = \left(1 - \frac{OD_s}{OD_f}\right) \times 100 \quad (1)$$

where OD_s is the optical density of the supernatant after flocculation of the microalgal suspension, and OD_f is the optical density of the feed sample. Optical density was measured at 600 nm for all samples, using a 1 cm path length cuvette.

The combined dosage and pH level that resulted in the highest clarification efficiency (Equation 1) was determined for the following two mixtures - (1) Alum and chitosan (10:1 by mass), and (2) Alum and zetag 7650 (10:1 by mass). The highest clarification efficiencies of the two above-mentioned flocculant mixtures were then compared to that of alum alone (Dosage = 10 mg/L; pH = 6.5). Because the clarification efficiencies of the three flocculant mixtures were essentially the same, the lowest cost flocculant was chosen to prepare the concentrated microalgal suspension for the belt dewatering tests.

4.2.2 Bench-scale flocculation

52 Liters of microalgal cultures harvested at their stationary growth phase concentration (1.5 $\pm$ 0.2 g dry wt. /L) were pumped into a 56 L graduated cylinder equipped with a spigot to allow decantation of the flocculation product. A 1.2 HP variable speed mixer with an axial-flow impeller was used to mix the microalgal suspension at 700 rpm for 60 seconds, followed by slow mixing at 60 rpm for 15 minutes. The flocculated microalgal suspension was then allowed to settle for 2 hours, and at the end of the settling period, approximately 5 Liters of ~ 4 g dry wt. /L concentrated microalgal suspension were collected. This procedure was repeated multiple times until a total of 54 Liters of ~ 4 g dry wt. /L concentrated microalgal suspension were collected. Three belt dewatering tests were conducted, using 18 Liters of the 54 Liter concentrated microalgal suspension for each test.

Belt dewatering test procedure is the same as section 3.2.4

4.3 Results and Discussion

4.3.1 Determination of optimum flocculant type, dosage and pH for stationary growth phase culture

At a fixed alum dosage (10 mg/L), flocculation performance of chitosan improved as the dosage was increased up to a maximum of 20 mg/L. With the alum dosage fixed at 10 mg/L, the flocculation efficiency of zetag 7650, starting at 5 mg/L, decreased with increasing zetag dosage. Danquah et al. (2009), who had similar results, suggested that over dosage of high molecular weight polymers led to a formation of elastic colloids reducing the effectiveness of the polymer

as a flocculant. Clarification efficiencies for pH and dosage values for the two flocculant mixtures are listed in Figure 1.

A series of tests were conducted, using alum as the flocculant, for a range of pH and dosage values. The results of this testing showed an almost linear increase in clarification efficiency up to an alum dosage of 200 mg/L. At an alum dosage of 200 mg/L the pre-test pH was varied from 4.5 to 9. For the alum dosage of 200 mg/L there were no significant differences in the clarification efficiency for a pre-test pH range from 5 to 6.5 (Figure 4.2A, one-way ANOVA, $p > 0.05$). Clarification efficiencies were significantly lower for all other tested pH values at 200 mg/L dosage (Figure 4.2A and 4.2B). The additions of alum, for all dosages tested, increased the pH of the microalgal suspension by 0.5 ± 0.1 pH units. For further testing a pre-test pH value of 6.5 was chosen to reduce the cost involved in lowering the pH of the microalgal solution from 7 to 6.5.

Comparing the highest clarification efficiencies of the two flocculant mixtures, alum + chitosan and alum + zetag 7650, with that of alum alone (Dosage = 10 mg/L; pH = 6.5) showed no significant improvement in clarification efficiency (Figure 4.3, one-way ANOVA, $p > 0.05$). Since alum was the most cost effective further testing was focused on this flocculant.

4.3.2 Belt filter dewatering test performance

A 70 micron mesh polyester filter belt was used for all belt filter testing. The percent of microalgae recovered during belt filter testing is shown in Table 4.1. The 4 g dry wt. /Liter microalgae suspension yielded a maximum of 46% recovered microalgae compared to 84% from the 6 g dry wt. /Liter microalgae suspension. Biomass losses of microalgae embedded in the filter belt, and not recoverable, ranged from 3 to 7%. The need for multiple filtration passes of the microalgal suspension was primarily due to significant leakage in the filter test section of the belt filter system.

4.4 Conclusions

The objective of this study was to investigate the dewatering efficiency of belt filter system for microalgal suspensions with concentration below 10 g dry wt. /L. The percent of microalgae recovered for 4 g dry wt. /L suspension was significantly lower than 6 g dry wt. /L suspension. Sealed filter section would likely improve the microalgal recovery (subsequently reducing the number of filtration passes required for maximum microalgal recovery).

4.5 References

Danquah, M.K., Ang, L., Uduman, N., Moheimani, N., Forde, G.M. 2009. Dewatering of microalgal culture for biodiesel production: exploring polymer flocculation and tangential flow filtration. J. Chem. Technol. Biot., 84, 1078-1083.

Divakaran, R., Pillai Sivasankara, V.N. 2002. Flocculation of algae using chitosan. J. Appl. Phycol., 14, 419-422.

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Sturm, B.S.M., Lamer, S.L. 2011. An energy evaluation of coupling nutrient removal from wastewater with algal biomass production. Appl. Energy, 88, 3499-3506.

Tillman, G.M. 1996. Basic Water Treatment: Troubleshooting and Problem Solving. CRC Press.

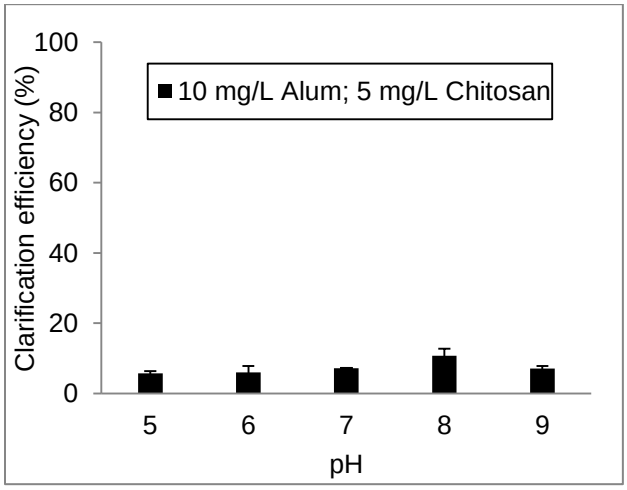
Table 4.1 Belt dewatering test performance – 4 g dry wt. /L versus 6 g dry wt. /L microalgal suspension.

	4 g dry wt. /L suspension	6 g dry wt. /L suspension
Number of successive filtrations (#)	Cumulative microalgae recovered (%)	Cumulative microalgae recovered (%)
1	23.26 $\pm$ 7.2	65 $\pm$ 6.5
2	31.7 $\pm$ 5.9	76 $\pm$ 5.7
3	36.6 $\pm$ 6.6	82 $\pm$ 5.8
4	40.5 $\pm$ 4.3	83.3 $\pm$ 3.6
5	43.7 $\pm$ 0.7	84.2 $\pm$ 1.7
6	46.1 $\pm$ 0.1	84.7 $\pm$ 0.06

Belt dewatering tests were conducted using 70 micron mesh size filter on sample suspensions, 18 Liters of 4 g dry wt. /L and 6 Liters of 6 g dry wt. /L, to determine the percent of microalgae recovered (mean $\pm$ standard deviation, n = 3).

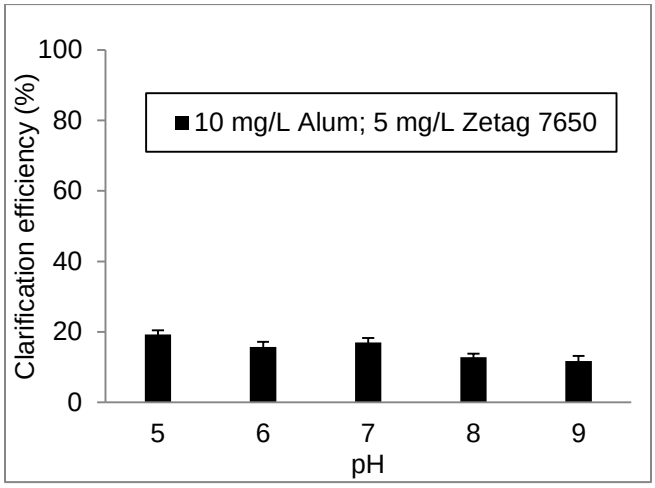
Figure 4.1 Determination of pH and dosage values that result in the highest clarification efficiency for the two flocculant mixtures – alum + chitosan and alum + zetag 7650. Jar tests were conducted on microalgal suspensions with an initial biomass concentration of 1.5 $\pm$ 0.3 g dry wt. /L to measure clarification efficiency (mean $\pm$ standard deviation; n = 3).

A) A coagulation dose of 10 mg/L for alum and 5 mg/L for chitosan was used for the tested pH range



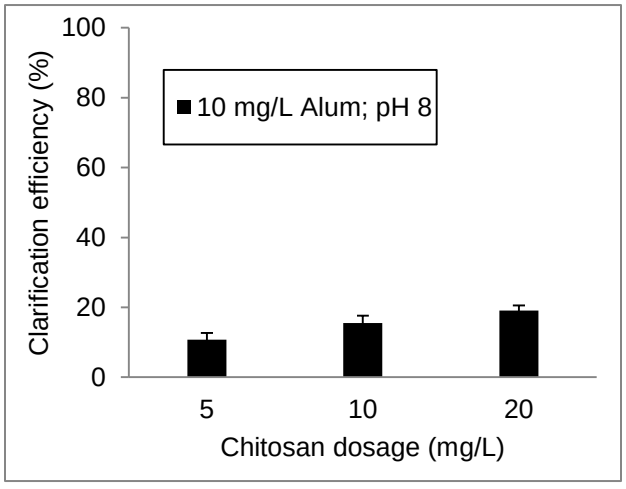
Clarification efficiency at pH value of 8 was significantly higher than all other clarification efficiencies. Four one-way ANOVA tests were conducted comparing clarification efficiency at pH value 8 with each of the other pH values and $p < 0.05$ for every test.

B) A coagulation dose of 10 mg/L for alum and 5 mg/L for zetag 7650 was used for the tested pH range



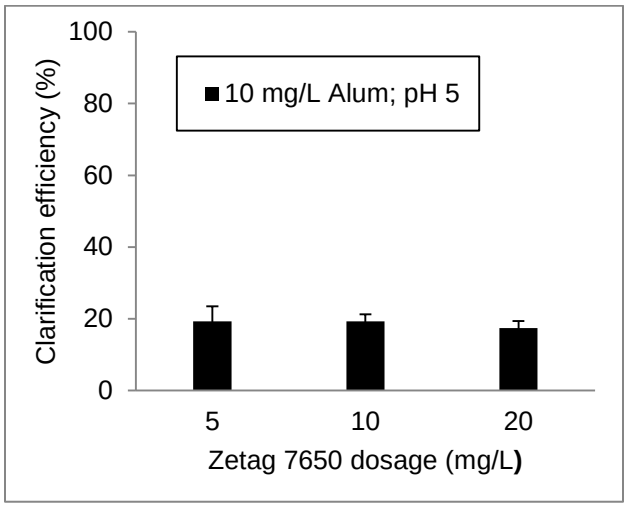
Clarification efficiency at pH value of 5 was significantly higher than all other clarification efficiencies. Four one-way ANOVA tests were conducted comparing clarification efficiency at pH value 5 with each of the other pH values and $p < 0.05$ for every test.

C) At a fixed alum dosage of 10 mg/L, a pH of 8 was used for a range of chitosan dosages



Clarification efficiency at chitosan dosage of 20 mg/L was significantly higher than all other clarification efficiencies. Two one-way ANOVA tests were conducted comparing clarification efficiency at 20 mg/L dosage with each of the other dosages and $p < 0.05$ for every test.

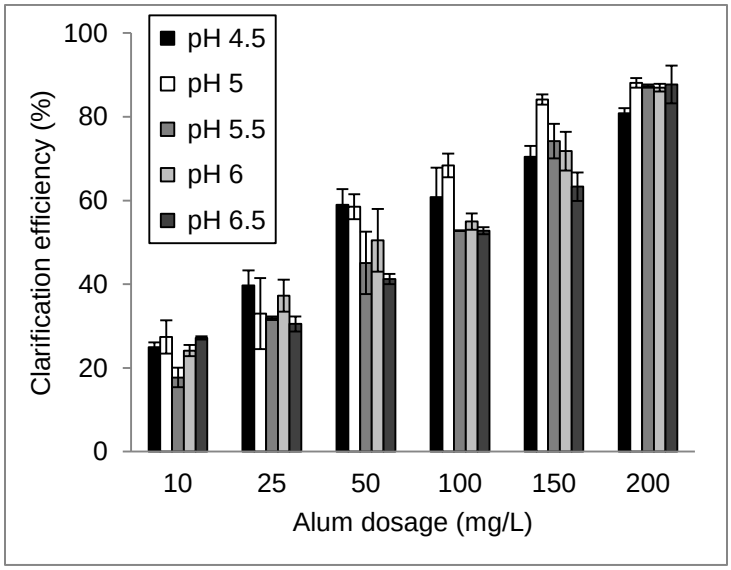
D) At a fixed alum dosage of 10 mg/L, a pH of 5 was used for a range of zetag 7650 dosages



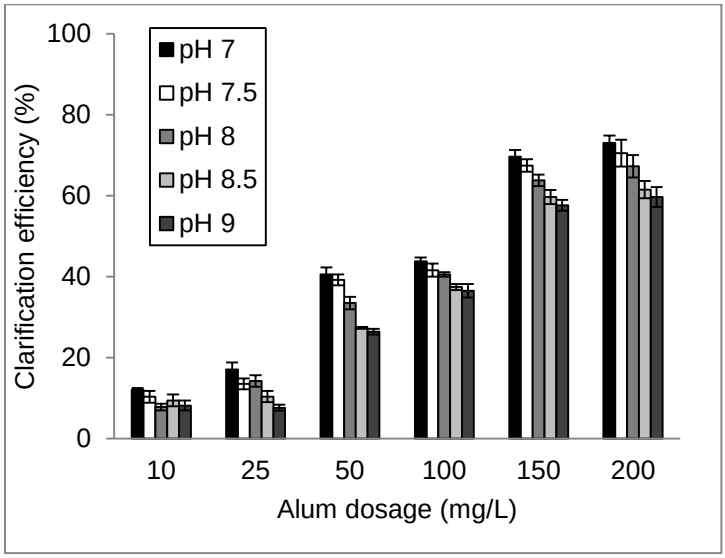
There were no significant differences in clarification efficiencies at zetag 7650 dosages of 5 and 10 mg/L (One-way ANOVA, $p > 0.05$). Clarification efficiency at 5 mg/L dosage was significantly higher than the 20 mg/L dosage (One-way ANOVA, $p < 0.05$).

Figure 4.2 Determination of pH and dosage values that result in the highest clarification efficiency for alum. Jar tests were conducted on samples of the microalgal suspension at their stationary phase of growth with an initial biomass concentration of 1.5 ± 0.3 g dry wt. /L to measure clarification efficiency (mean ± standard deviation; n = 3) of alum.

A) A pH range of 4.5 to 6.5 in 0.5 pH increments for the tested dosage range

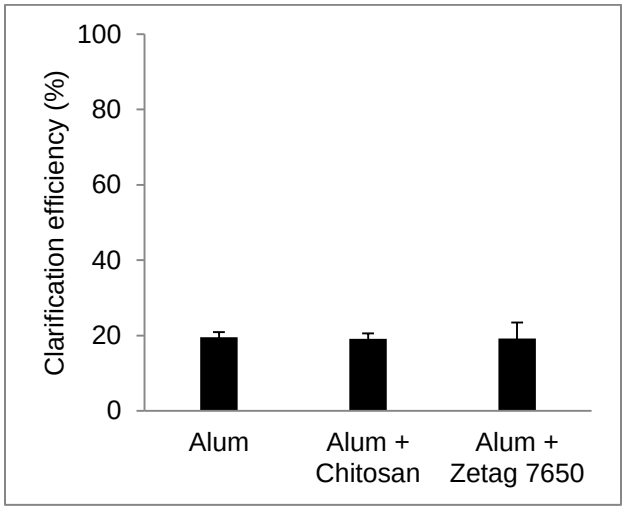


B) A pH range of 7 to 10 in 0.5 pH increments for the tested dosage range



For the alum dosage of 200 mg/L there were no significant differences in the clarification efficiency for a pre-test pH range from 5 to 6.5 (One-way ANOVA, $p > 0.05$). Clarification efficiencies were significantly lower for all other tested pH values at 200 mg/L dosage.

Figure 4.3 Determination of flocculant mixture – alum, alum + chitosan and alum + zetag 7650 – that results in the highest clarification efficiency.



Comparing the highest clarification efficiencies of the two flocculant mixtures, alum + chitosan and alum + zetag 7650, with that of alum alone (Dosage = 10 mg/L; pH = 6.5) showed no significant improvement in clarification efficiency (One-way ANOVA, $p > 0.05$).

CHAPTER FIVE: SUMMARY

The primary goal of this work was to investigate the microalgal dewatering efficiency of a belt filter system. A prototype belt filter dewatering system was designed and developed. A mixed culture of microalgal species dominated by three eukaryotic green algae (*Chlorella vulgaris, Scenedesmus sp.*, and *Kirchneriella sp*.) was cultivated in domestic wastewater effluent from the Lawrence, Kansas wastewater treatment plant. Bench-scale gravity filtration tests were conducted on microalgal samples at their stationary growth phase to determine the filtration belt mesh needed for the prototype system. These tests used a range of polyester mesh sizes from 10 to 200 microns. Based on the test results a mesh size of 70 microns resulted in the highest microalgal recovery rate and was subsequently used for all belt dewatering tests conducted in this study.

Preliminary belt filter dewatering tests conducted on untreated microalgal suspension resulted in low to negligible recovery. The highest concentration of microalgal suspension available for testing on the prototype belt filtration system was 6 g dry wt. /L obtained from biomass settling tanks at the Lawrence, Kansas domestic wastewater treatment plant. Dewatering tests of this suspension resulted in 84% biomass microalgal recovery. The results of this initial testing suggested that concentrations of microalgal suspensions less than 10 g dry wt. /L could be effectively recovered.

To further investigate this, 54 Liters of 4 g dry wt. /L were produced from bench-scale flocculation using an alum dosage of 200 mg/L at pre-test pH value of 6.5. Flocculant type,

dosage and pH level that resulted in the highest clarification efficiency were determined from jar tests conducted on stationary growth phase microalgal samples. Results of belt dewatering tests with 70 micron polyester filter mesh recovered 46% percent microalgae for 4 g dry wt. /L suspension.

Conclusions and Recommendations

Low microalgal recovery from preliminary belt filter dewatering tests conducted on untreated microalgal suspensions with concentrations ranging from 0.045 to 1.5 g dry wt. /L indicated a need for pre-concentration of feed suspension prior to dewatering. This is consistent with the findings of previous studies on belt dewatering where 10 – 40 g dry wt. /L microalgal feed concentration was needed. This led to experimental investigation on dewatering efficiency of belt filter system for treated microalgal suspensions with concentrations below 10 g dry wt. /L. Belt dewatering tests were conducted on microalgal suspensions with concentration of 4 g dry wt. /L and 6 g dry wt. /L. The study results indicate that microalgal concentrations as low as 6 g dry wt. /L can be effectively recovered from belt filter system. For microalgal suspension with concentration of 4 g dry wt. /L, the percent of algae recovered dropped significantly. This could be partly attributed to the leakages in the filter section of the system. Sealed filter section would likely improve the microalgal recovery (subsequently reducing the number of filtration passes required for maximum microalgal recovery).

Limitations and Future work

For this study, mesh size was the only machine parameter that was considered due to lack of sufficient volume of concentrated microalgal suspension. For the same reason, the only performance characteristic investigated was percent of microalgae recovered.

The next step in this line of research would involve sealing the filter section of the system. To improve the percent of microalgae recovered, effect of machine parameters such as belt speed and filter feed rate must be further explored. Belt filter performance characteristics such as flow throughput and biomass recovery rate need to be investigated as it takes the time taken to recover the biomass into consideration. Future studies must address several questions. Firstly, can bioflocculation replace chemical flocculants on an industrial scale? Secondly, can large scale microalgal cultivation systems effectively yield concentrations on the order of 6 g dry wt. /L removing the need for flocculation prior to belt dewatering? Finally the question that needs to be addressed is whether the belt filter system would satisfy the requirements of an optimal microalgal harvesting technique – low energy and chemical demand, low operational costs and be scalable to larger sizes. Belt filter has a continuous mode of operation and can be up-scaled. The results of the study indicate that the system could effectively recover concentrations as low as 6 g dry wt. /L thereby reducing the chemical demand. Experimental optimization of machine parameters such as belt speed needs to be conducted to improve the belt filter recovery for concentrations below 6 g dry wt. /L to further reduce the dependence on flocculation. The final step would be to perform energy and cost analysis of the system.

APPENDIX A. BELT FILTER SYSTEM – CALCULATION OF DESIGN PARAMETERS

Reference: ME 644 Project – Algae dewatering; Team members – Sean McConville, Steve Thomas, Alex Hanish and Fabian Schmidt. December 09, 2009.

Design parameters were calculated based on preliminary bench-scale gravity filtration tests followed by air drying. Tests were conducted using 0.133 oz of 5% (w/v) microalgal suspension using polyester cloth with a hole area of 0.1104 in^2 (Table A.1) for filter cross-sectional area of 1.5 in^2. The filter belt used a 200 thread count woven in a tight dutch weave. Individual thread diameters are 0.0054 inches with 80 weaves per linear inch.

Table A.1 Results of preliminary bench-scale tests conducted on 5% (w/v) microalgal suspension

Test parameters	**Results**
Time filtered before clogging	2 minutes
Air drying time using absorbent belt	15 minutes

The prototype would be a 1% scale of a system proposed to process 60,000 gallons of 5% (w/v) total suspended solids microalgal solution per day.

Solution:

Volume filtered per minute = 0.0665 oz/minute = 0.748 gal/day

If a filter with CSA 1.5 in^2 had a flow throughput of 0.75 gallon/day, the area required to filter 60,000 gallons of 5% (w/v) solution per day $= \frac{1.5 \times 0.748}{60{,}000}$ = 835.4 ft^2/minute

A 10% prototype of the full scale system would have a CSA of 84 ft^2/minute. For a 10 ft wide belt a belt velocity of 8.4 ft/minute would be needed.

Length of filter section = Belt velocity × Filtering time

= 17 ft

Length of drying section = Belt velocity × Drying time

= 126 ft

For a 1% prototype of the machine, the length of filter and drying sections would be 1.7 ft and 12.6 ft, respectively.

APPENDIX B. DETAILED DESIGN DRAWINGS OF PROTOTYPE BELT FILTER SYSTEM

All units in CGS system. Material used Aluminum 6061-T6.

All drawings created in Autodesk Inventor Professional 2011.

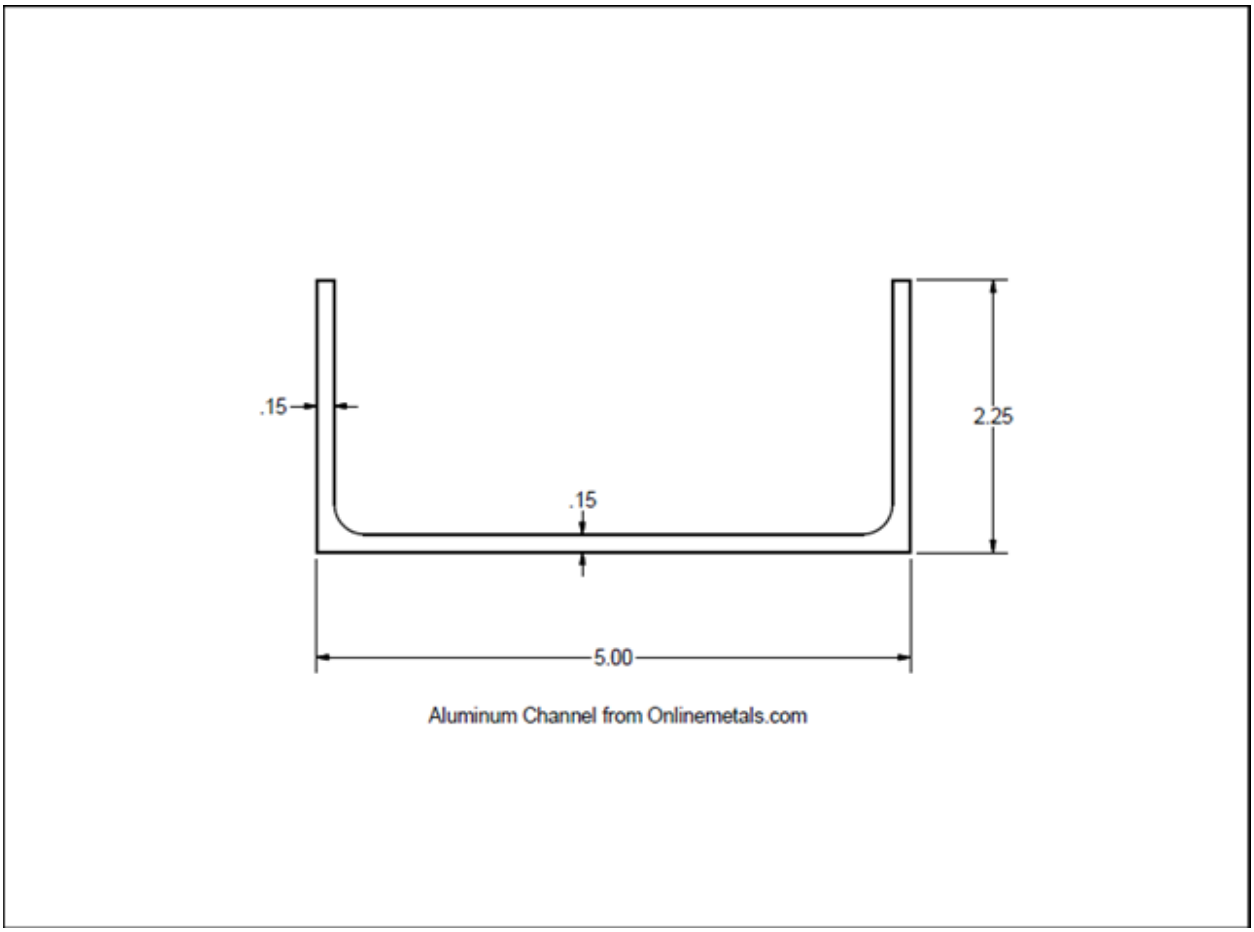


Figure B.1 Aluminum channel

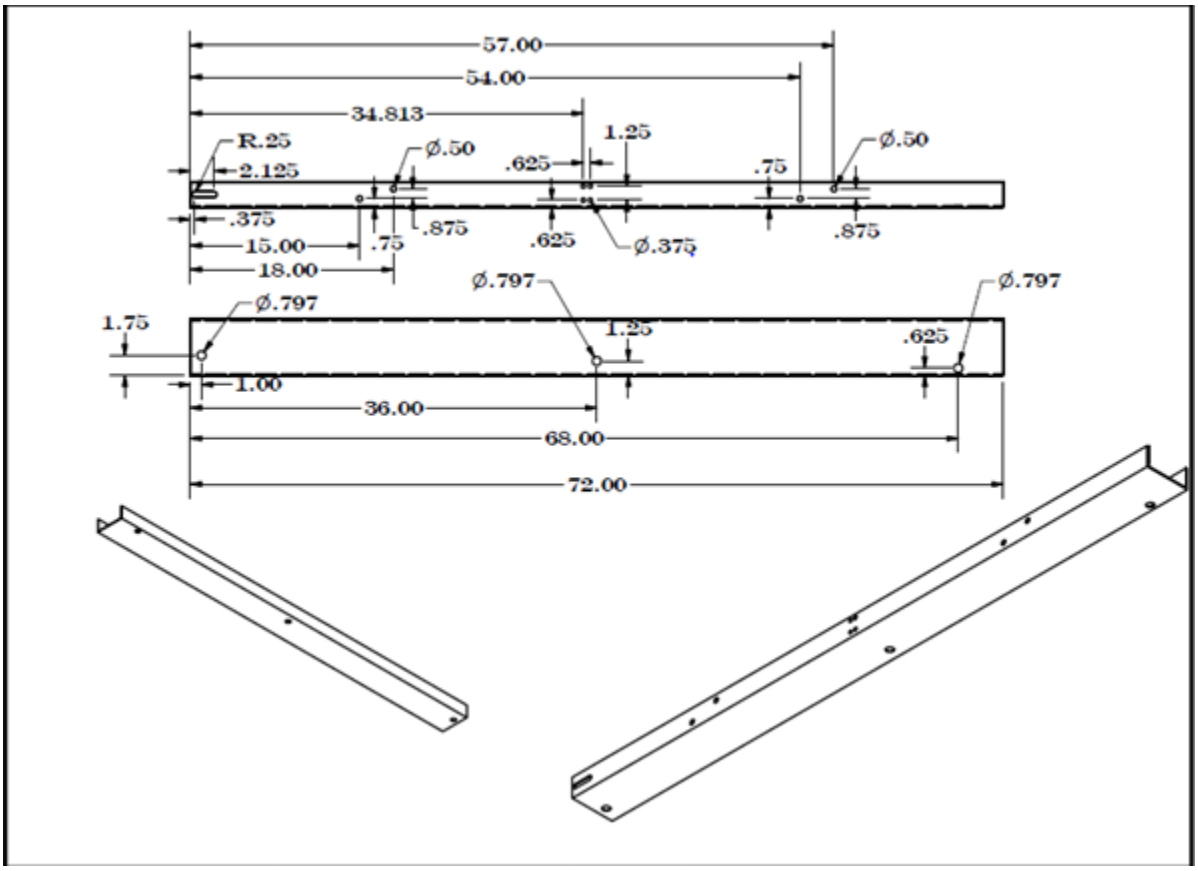


Figure B.2 Top rail section 3 back

Quantity: 1

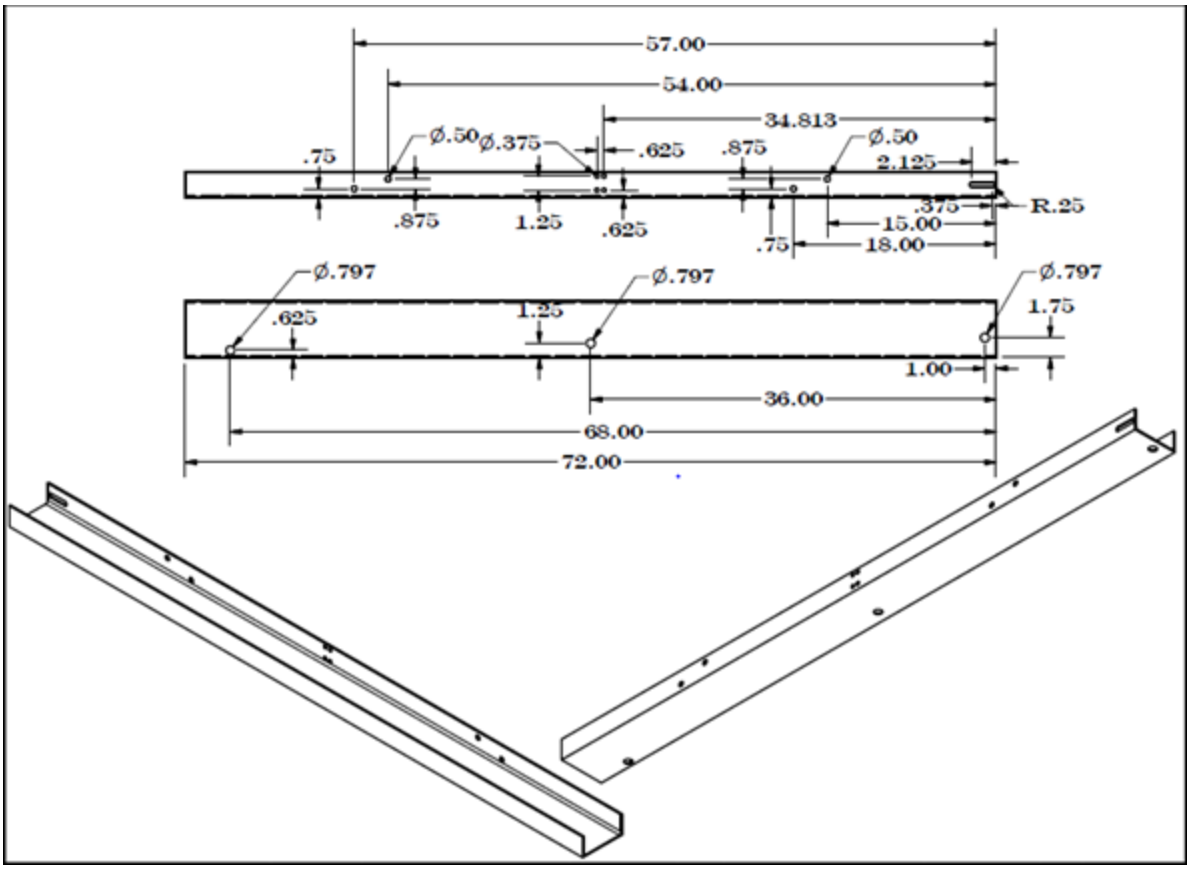


Figure B.3 Top rail section 3 front

Quantity: 1

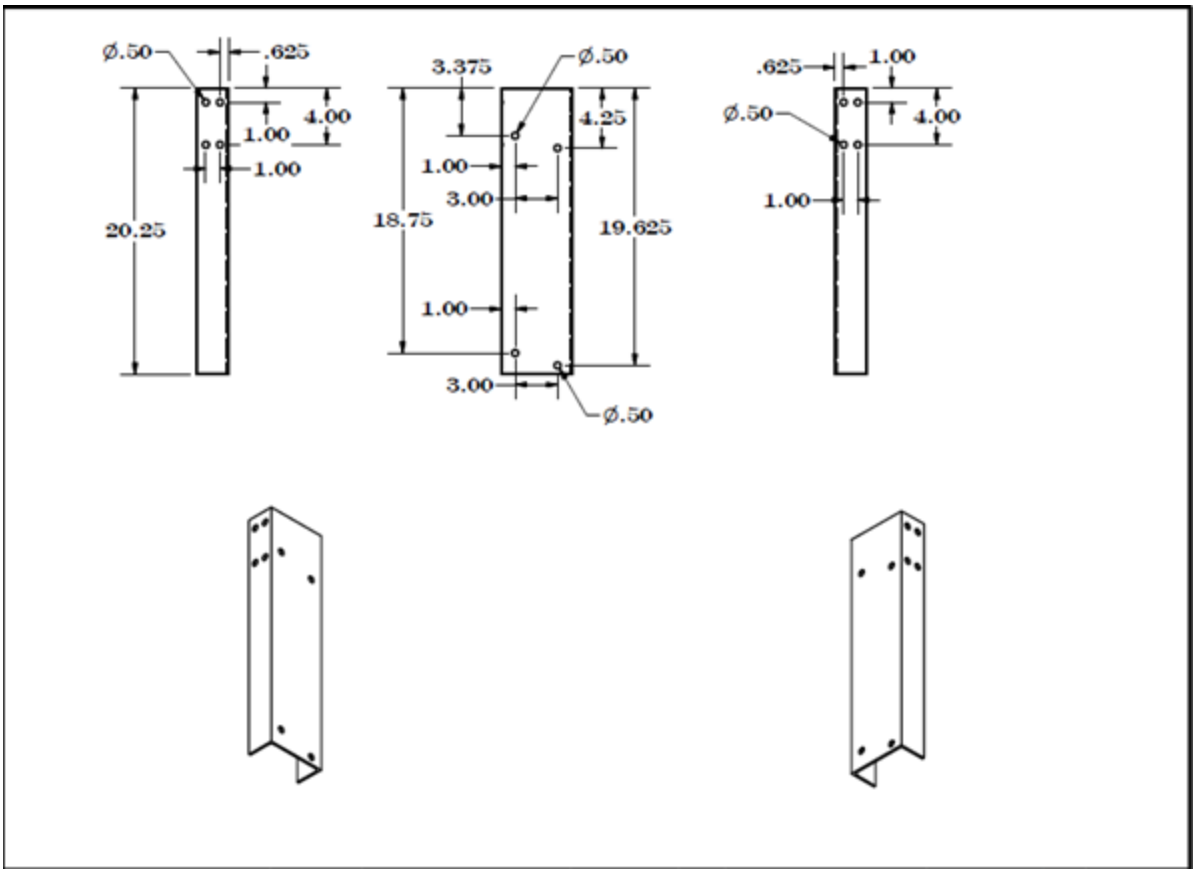


Figure B.4 Cross support top

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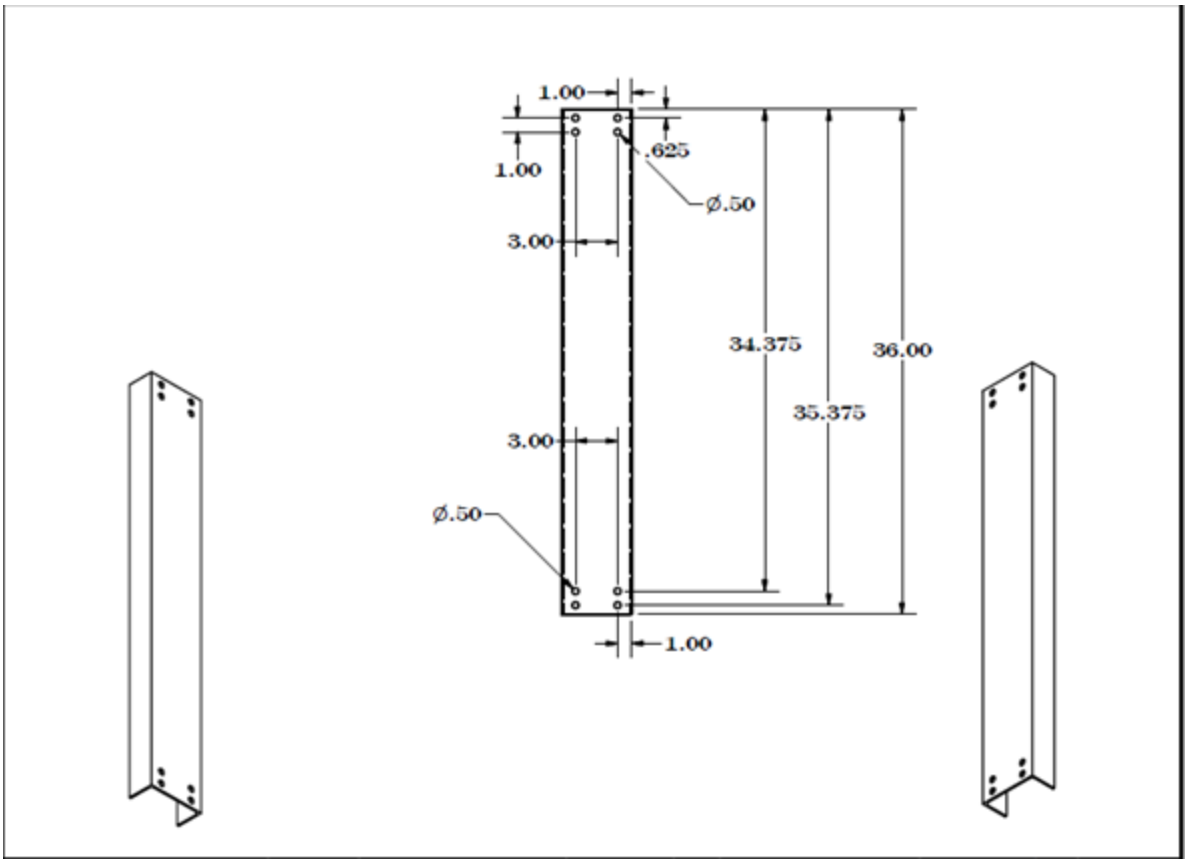

Figure B.5 Side support leg

Quantity: 10

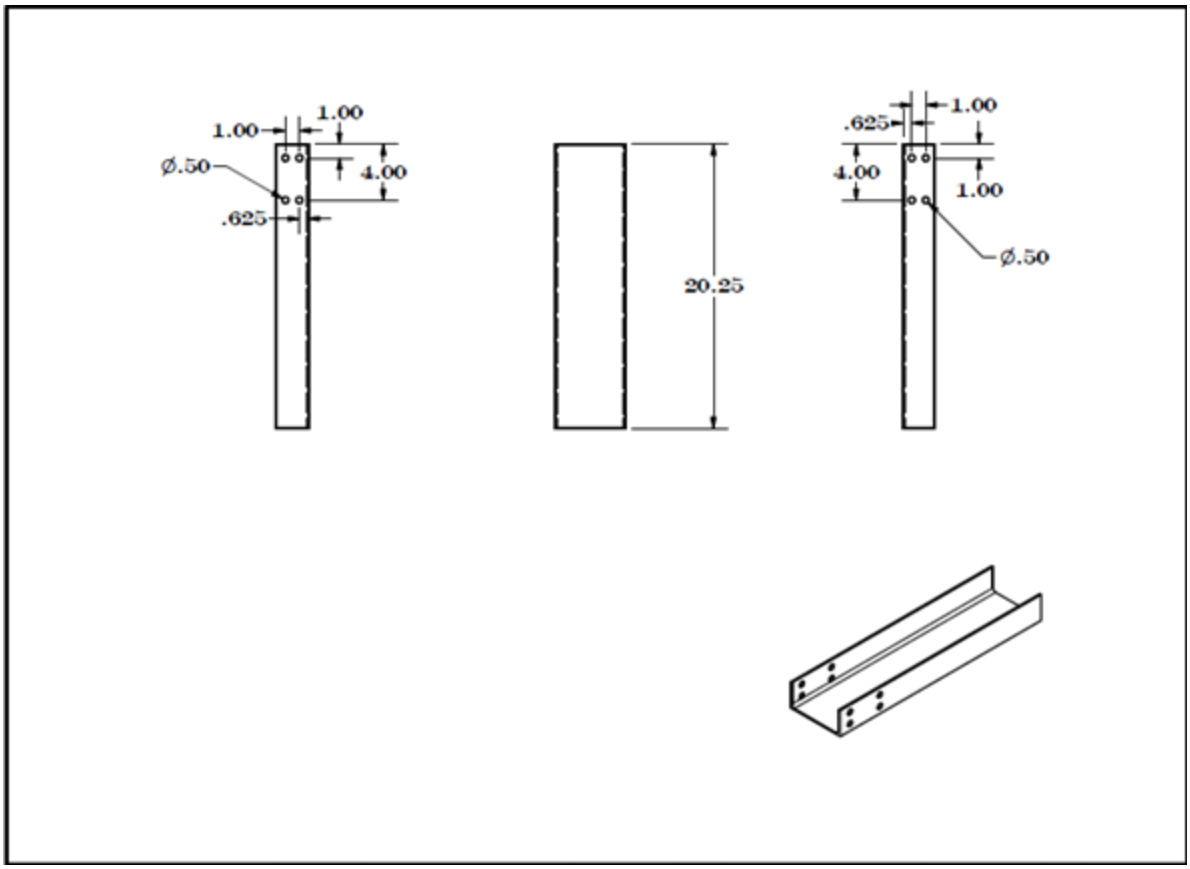

Figure B.6 Foot support

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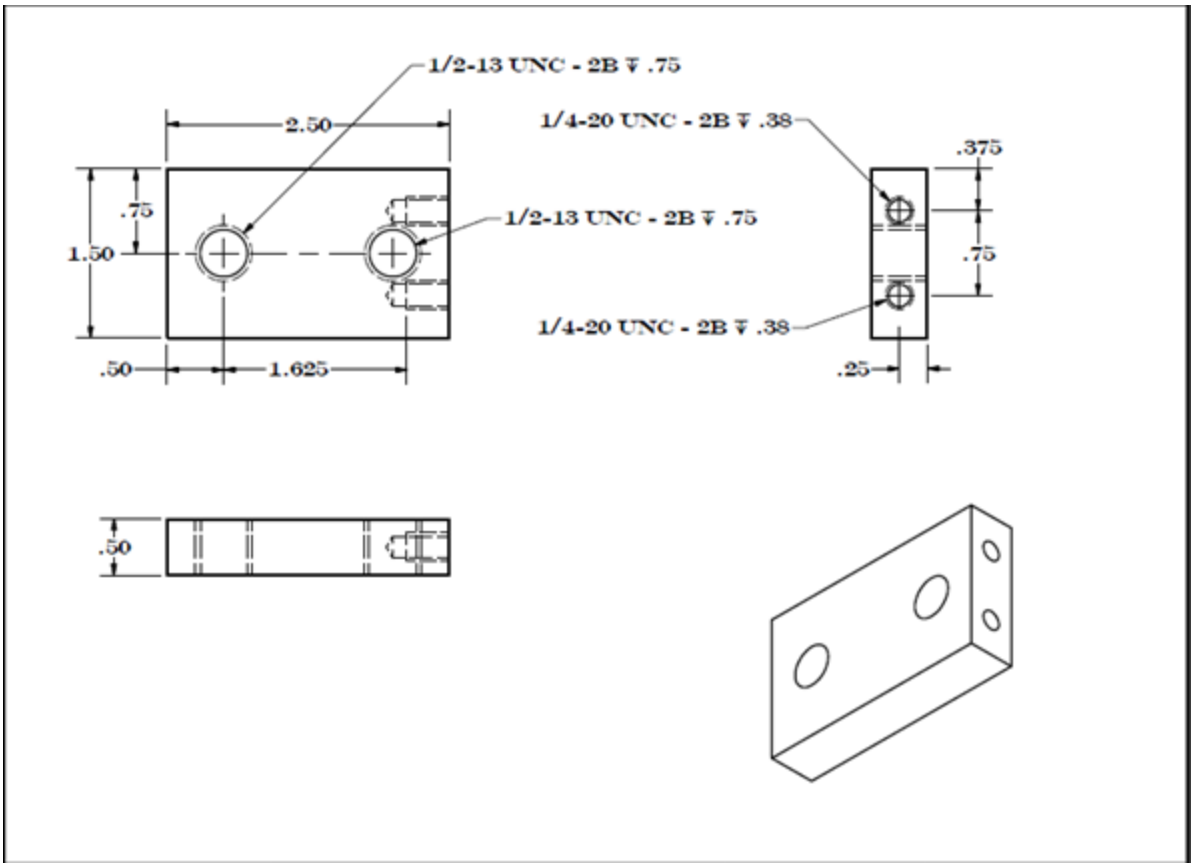

Figure B.7 Scraper block

Quantity: 2

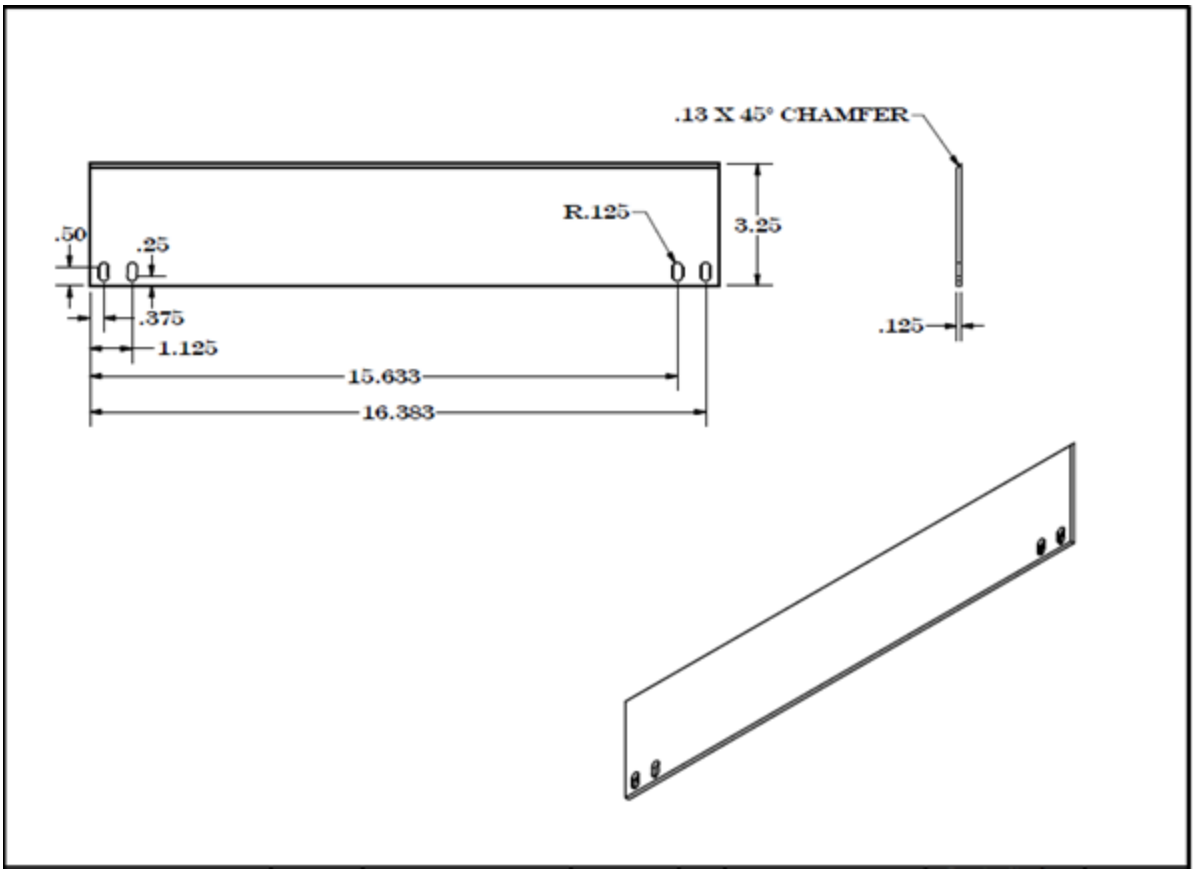

Figure B.8 Scraper blade

Quantity: 1

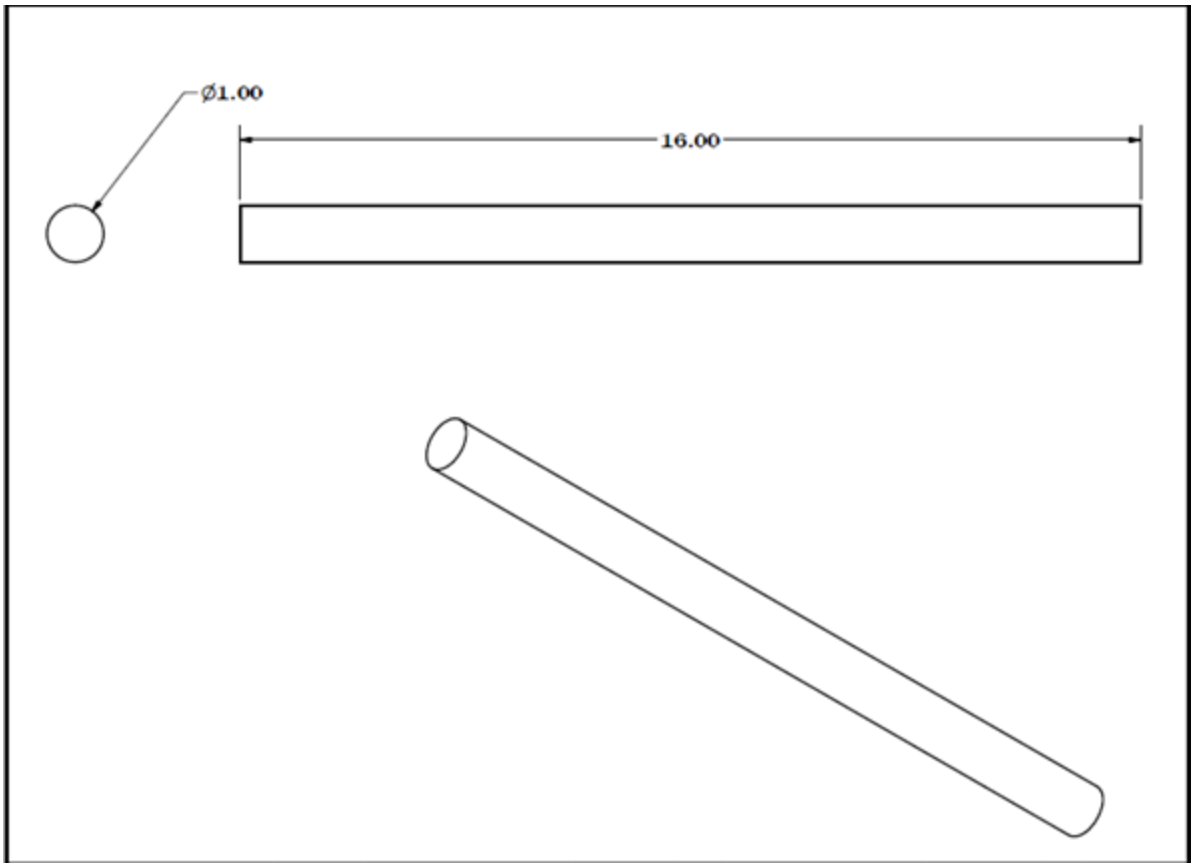

Figure B.9 Swing arm pivot rod

Quantity: 4

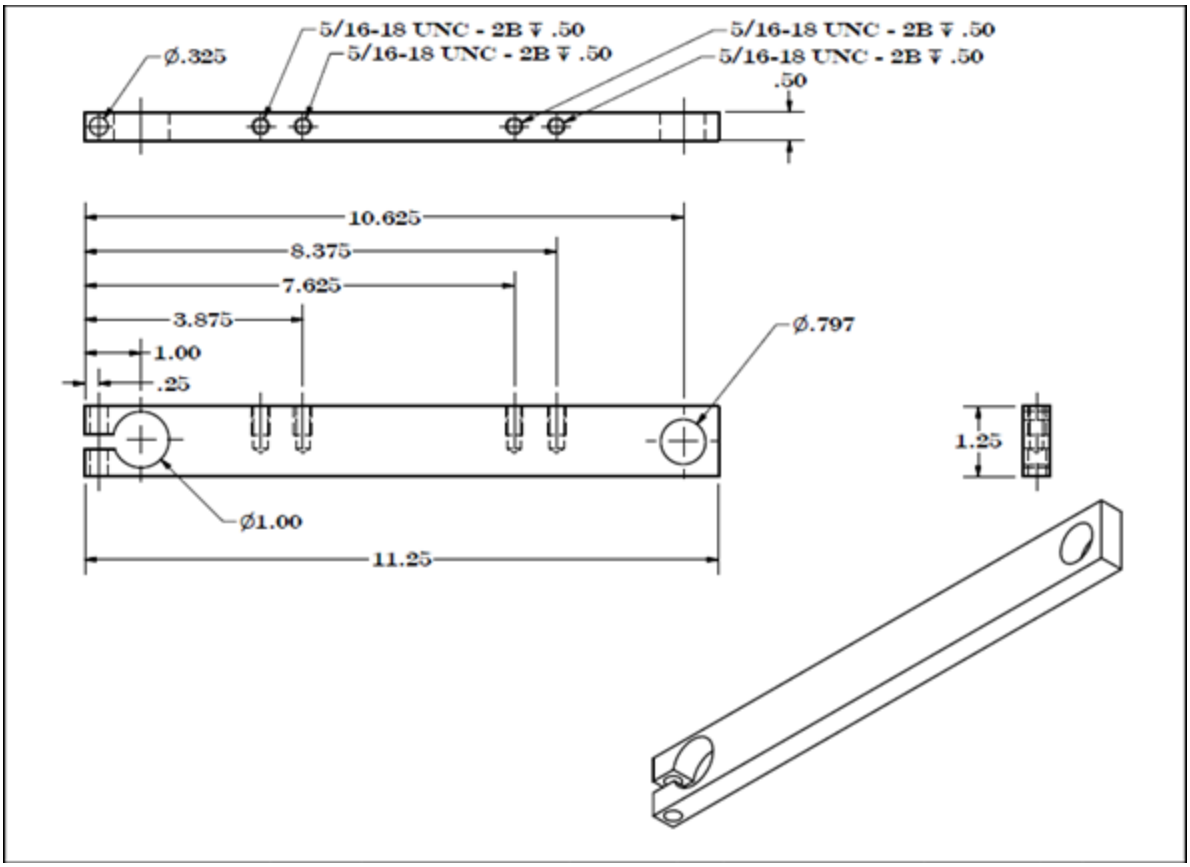

Figure B.10 Swing arm

Quantity: 8

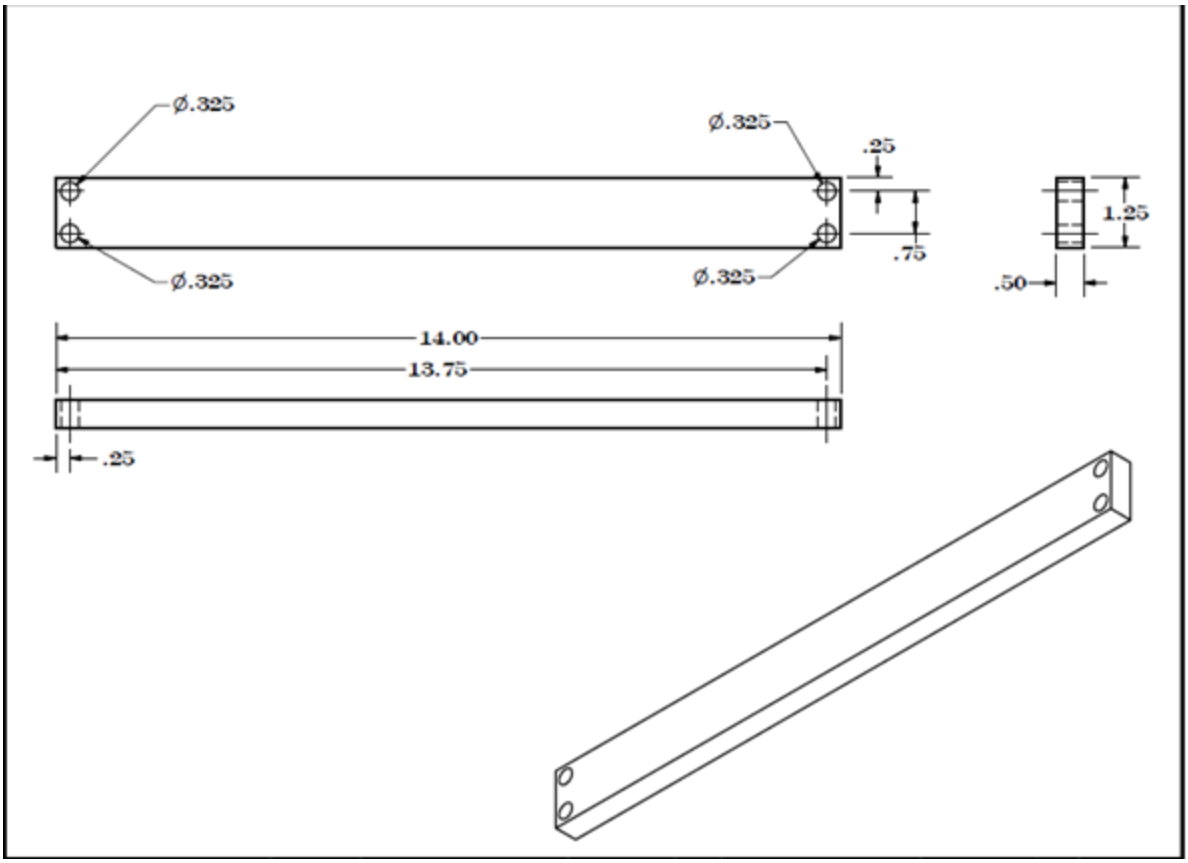

Figure B.11 Swing arm cross brace

Quantity: 8

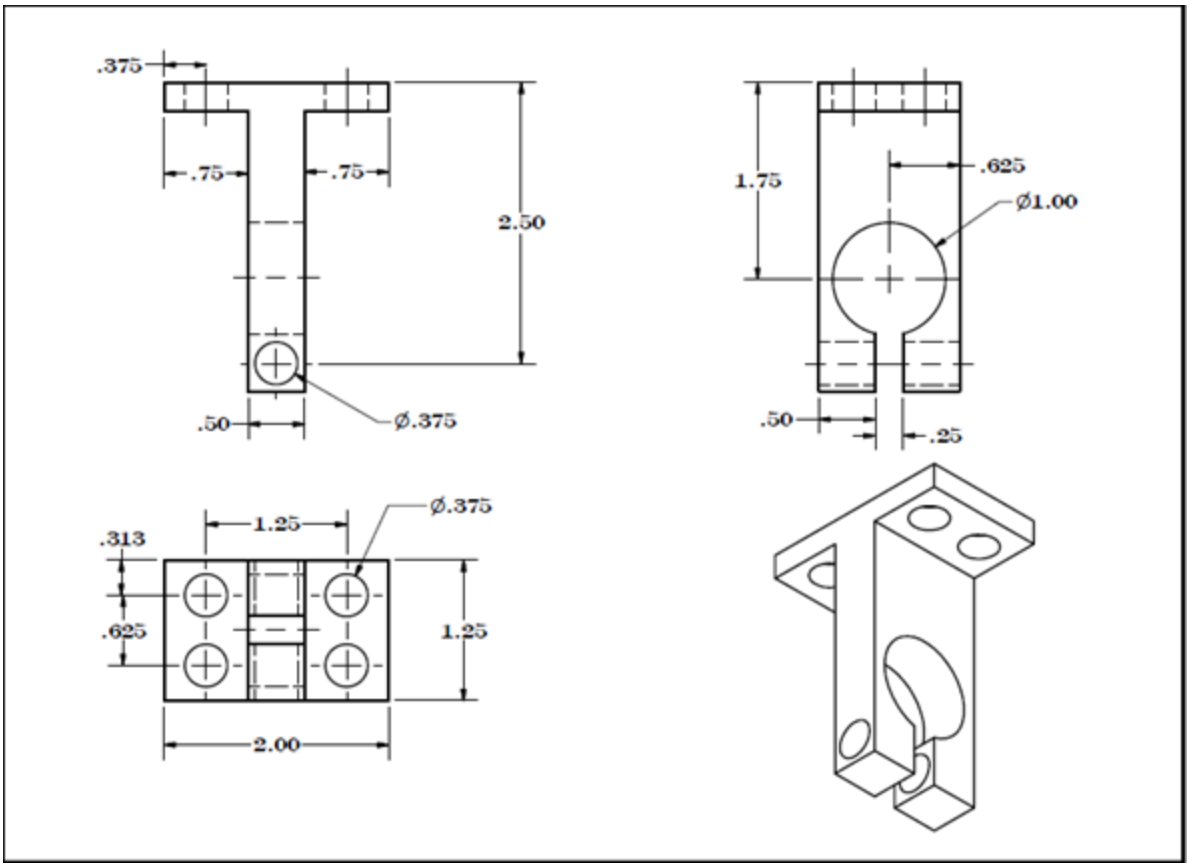

Figure B.12 Swing arm bracket

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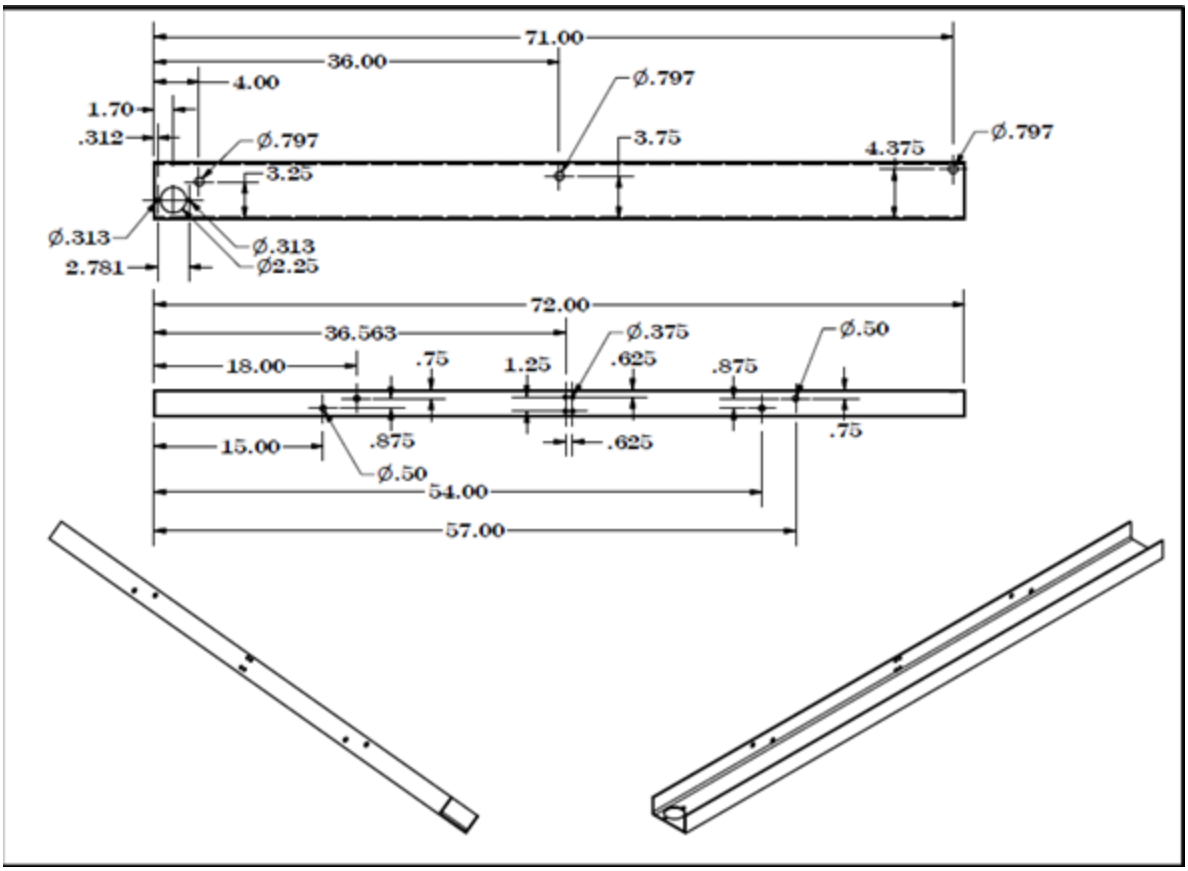

Figure B.13 Top rail section 2 back

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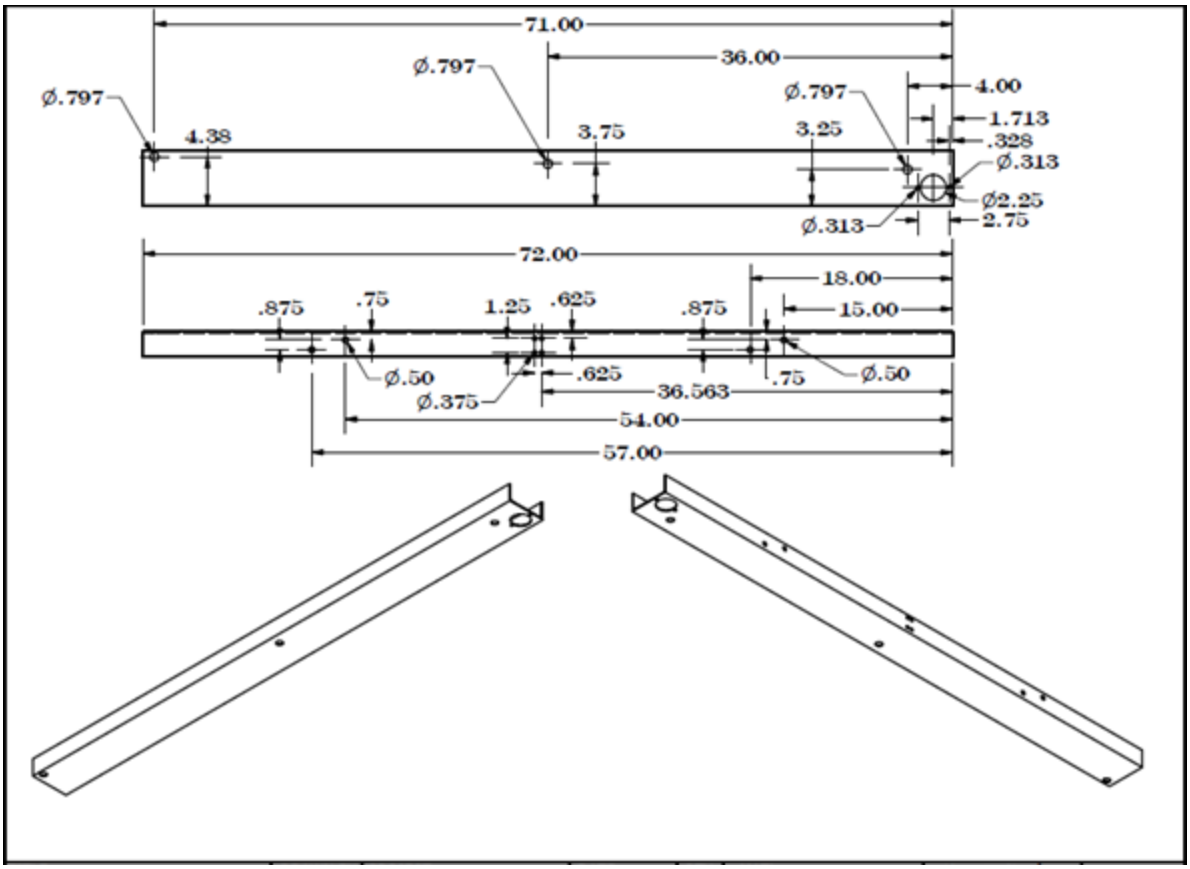

Figure B.14 Top rail section 2 front

Quantity: 1

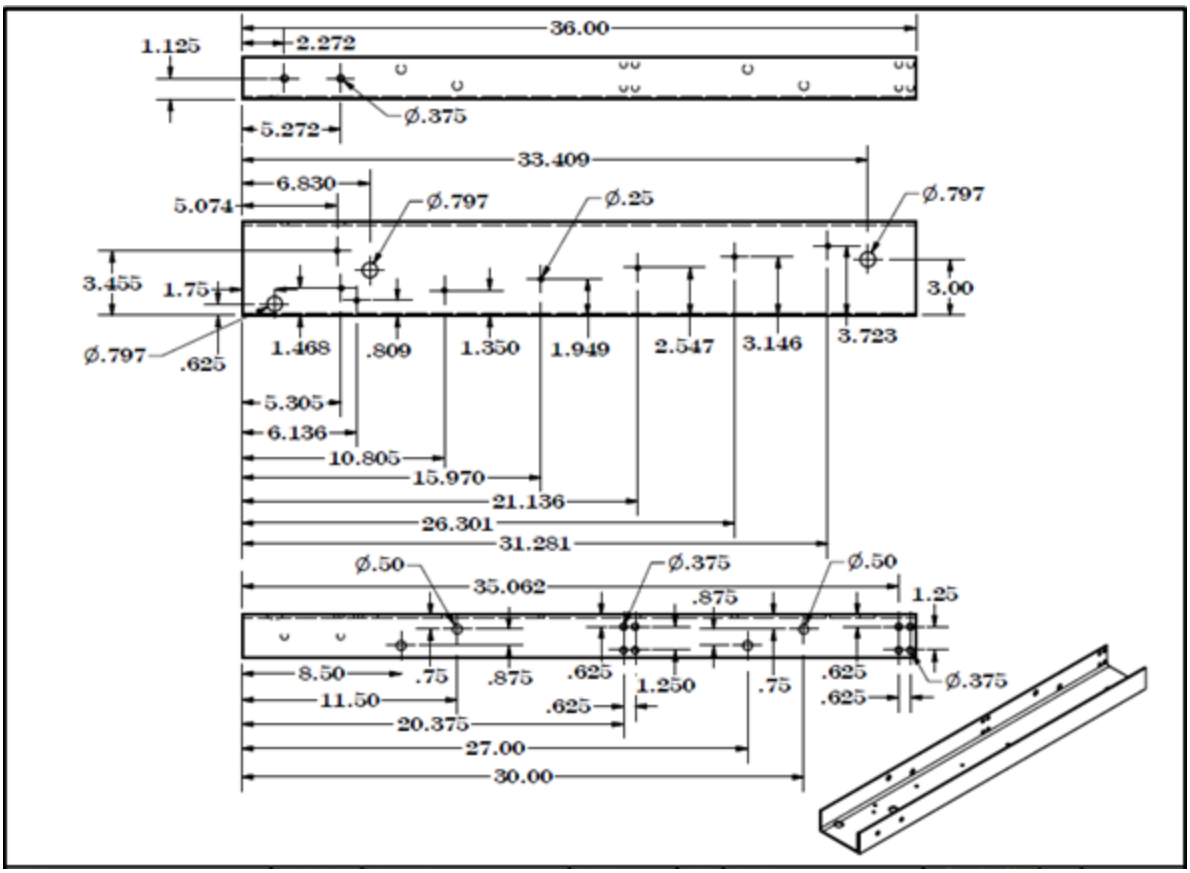

Figure B.15 Section 1 top back rail

Quantity: 1

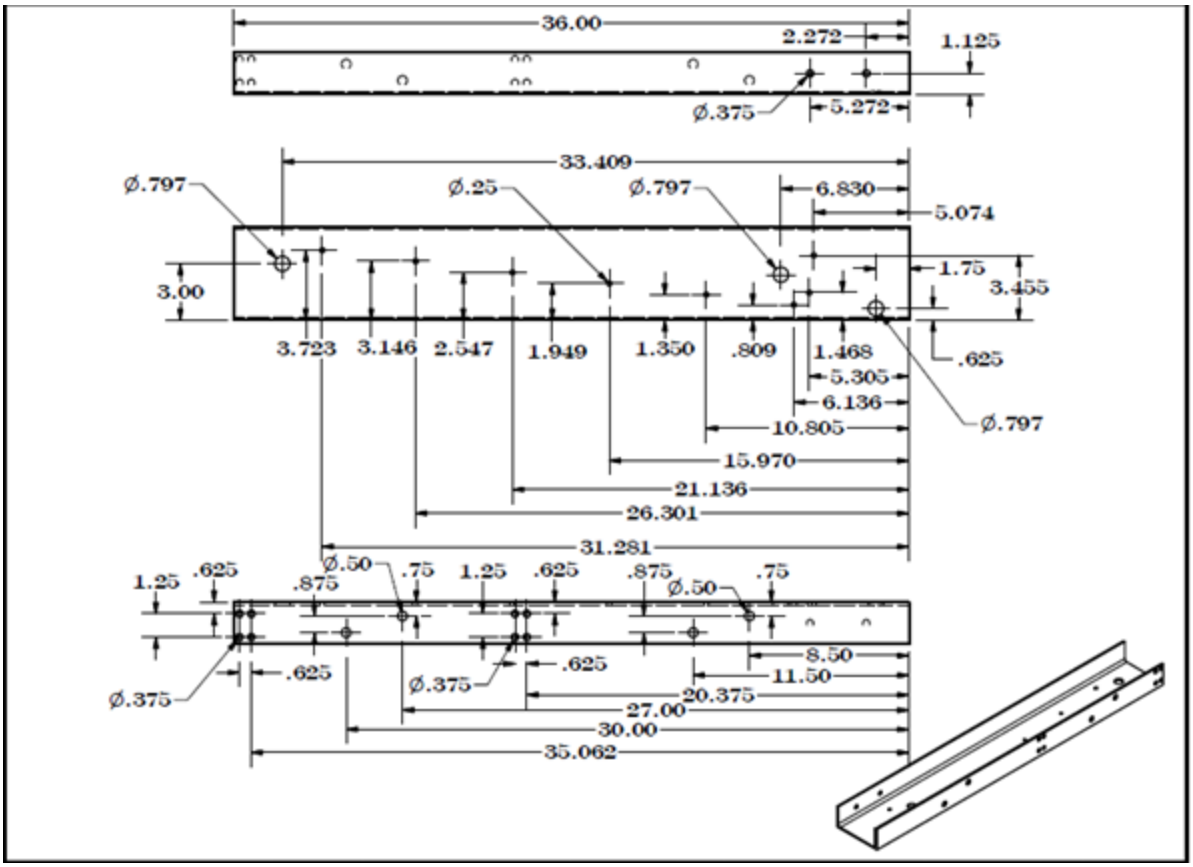

Figure B.16 Section 1 top front rail

Quantity: 1

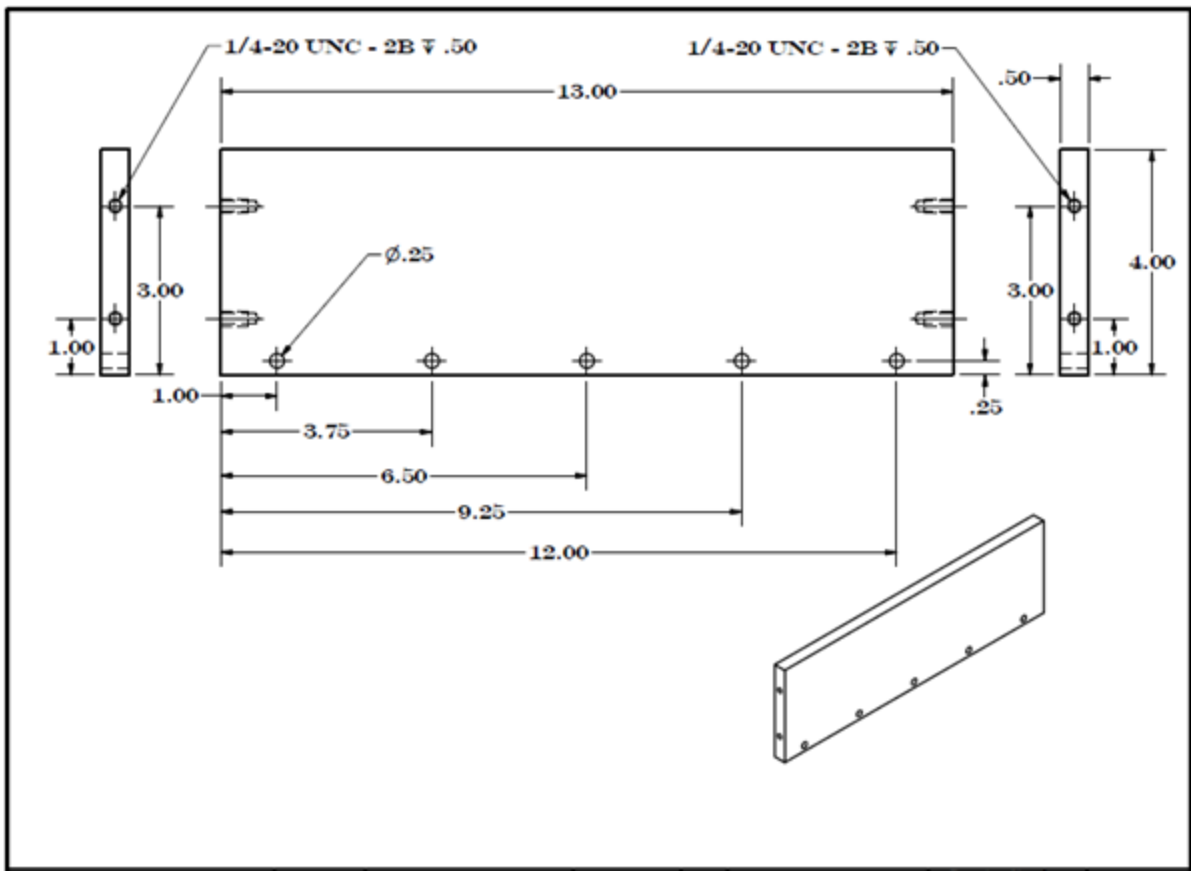

Figure B.17 Filter pan back

Quantity: 1

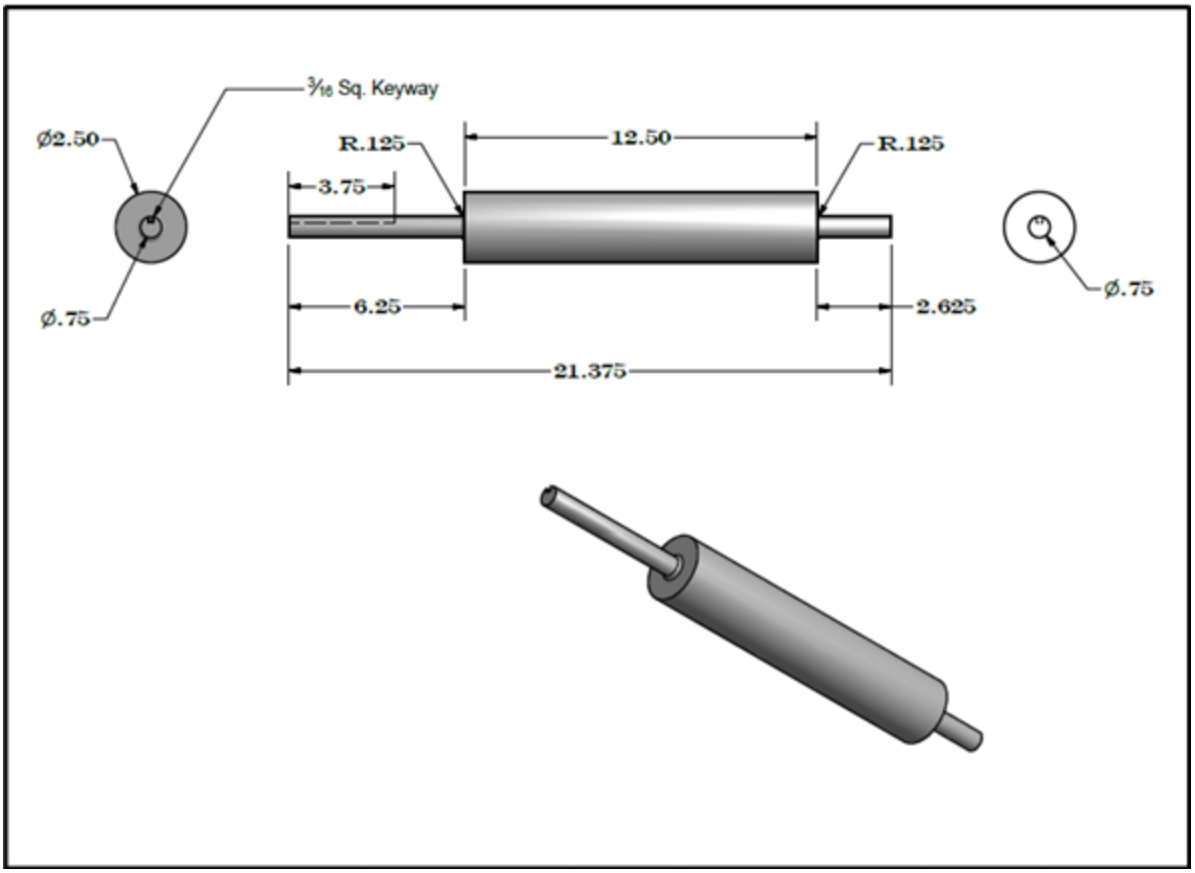

Figure B.18 Power shaft

Material: Stainless Steel

Quantity: 2

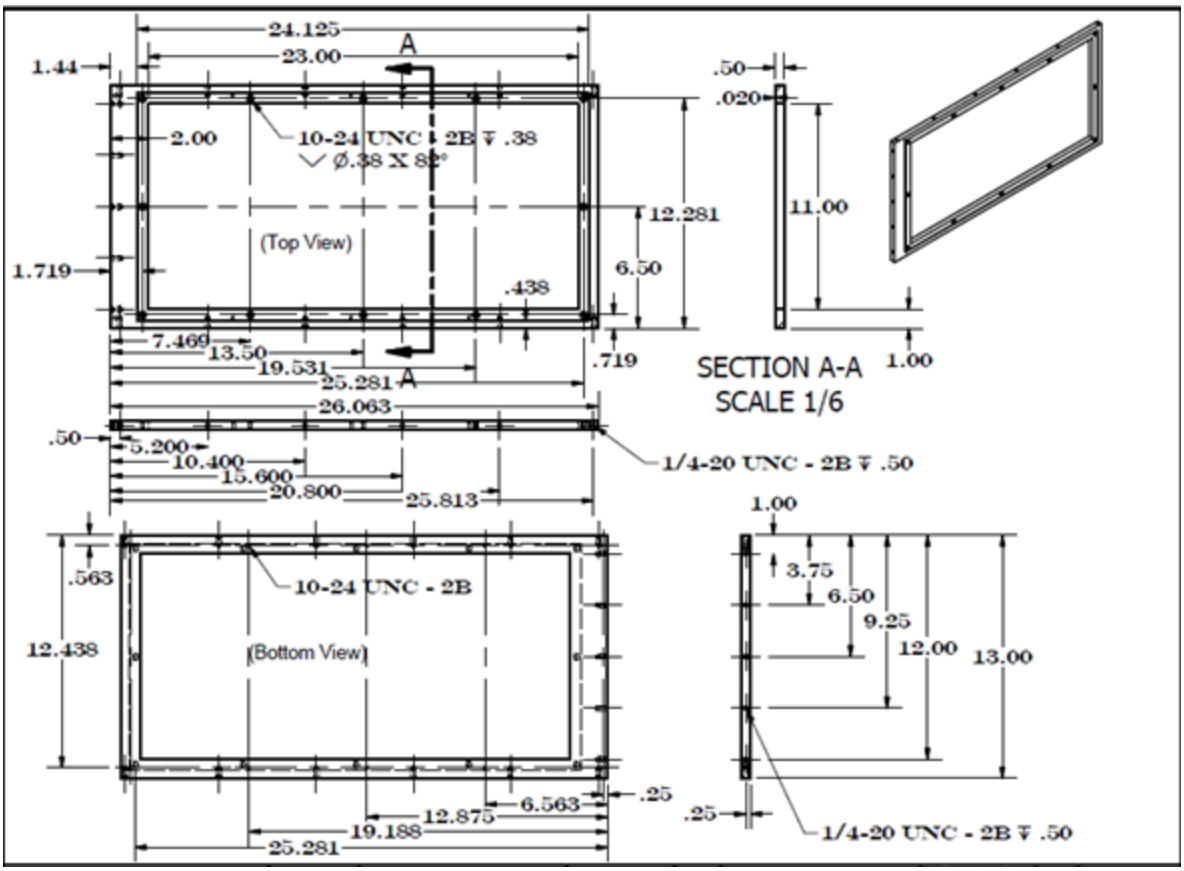

Figure B.19 Filter pan base

Quantity: 1

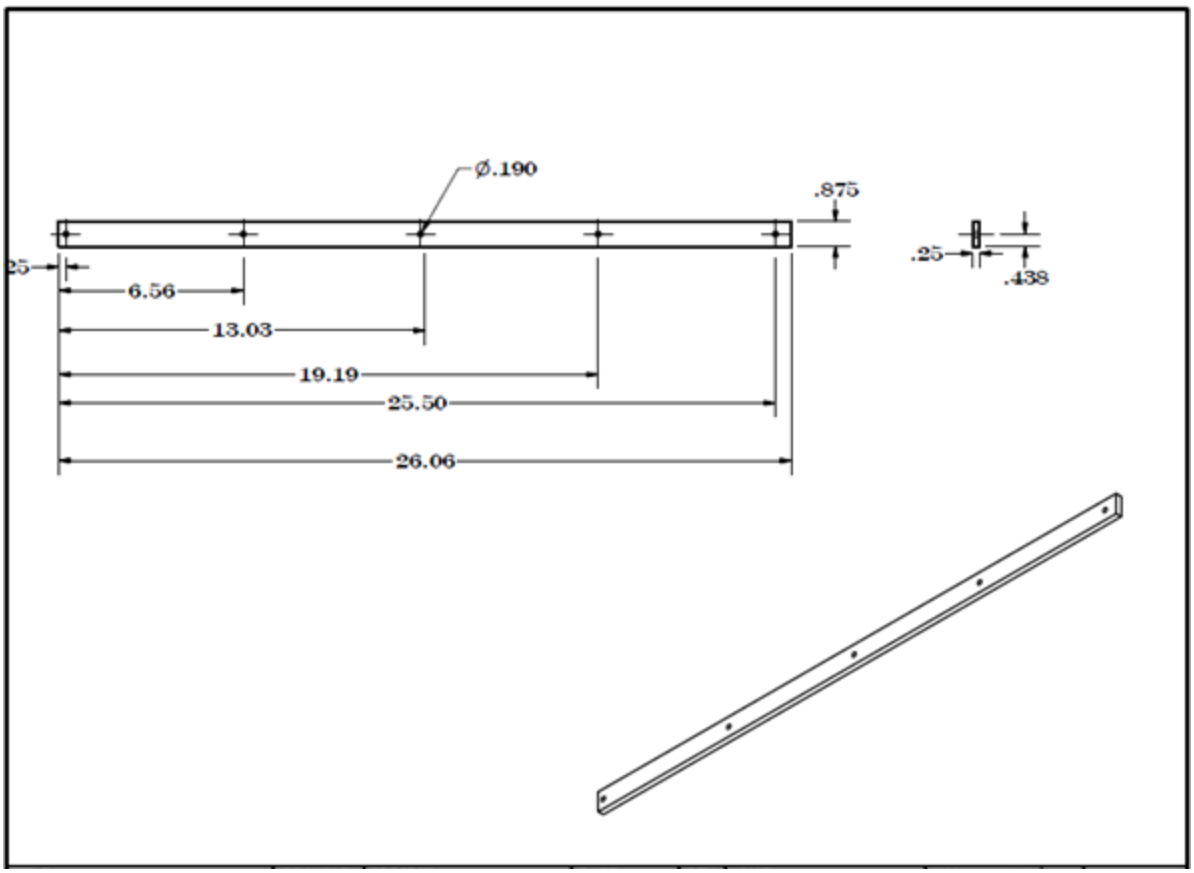

Figure B.20 Drip pan spacer

Quantity: 2

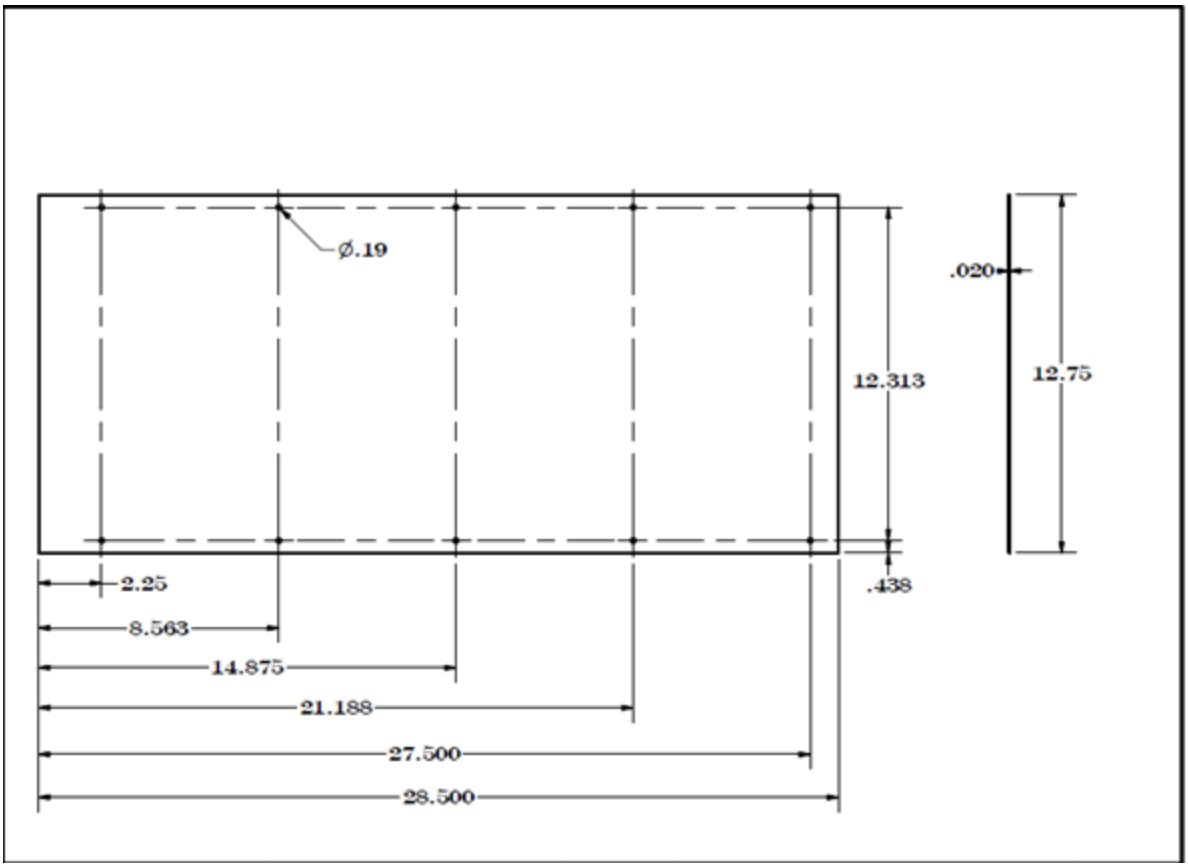

Figure B.21 Drip pan

Quantity: 1

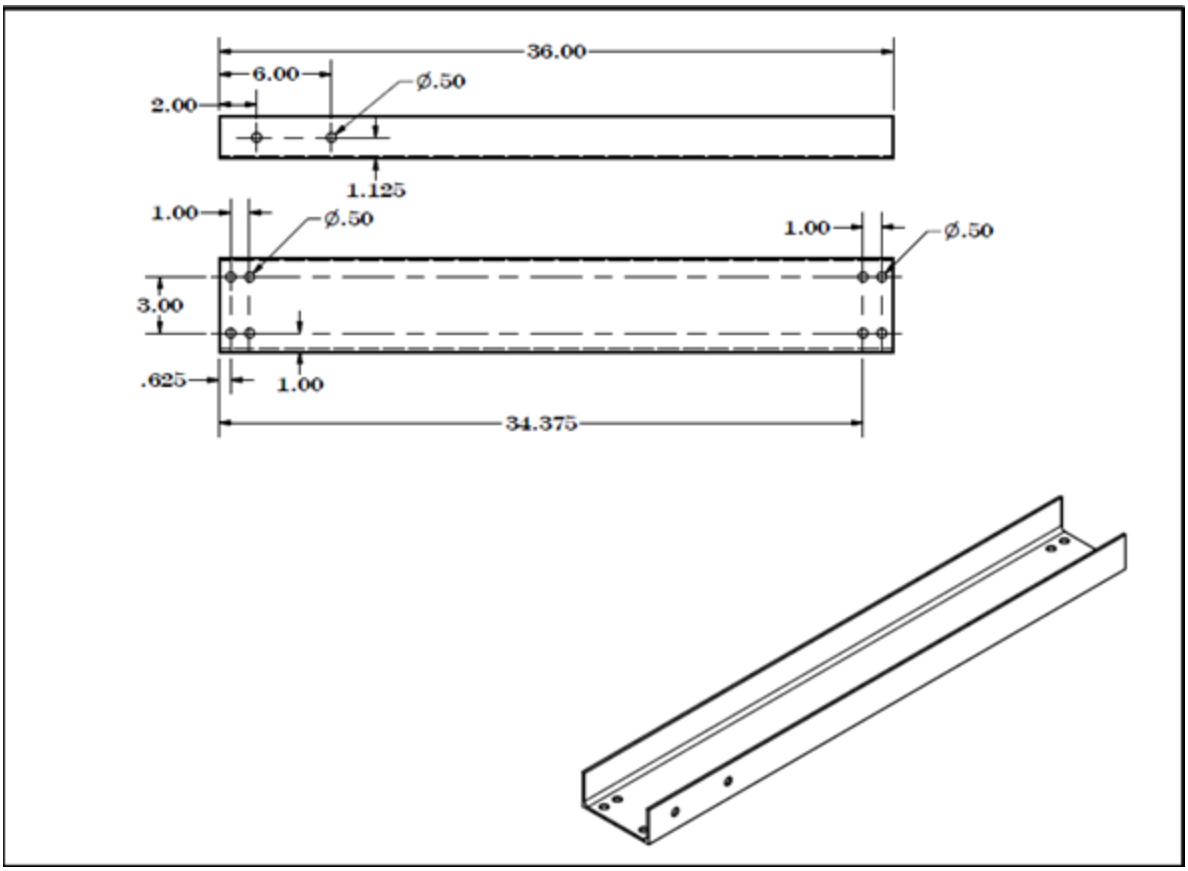

Figure B.22 Side support leg 1A

Quantity: 1

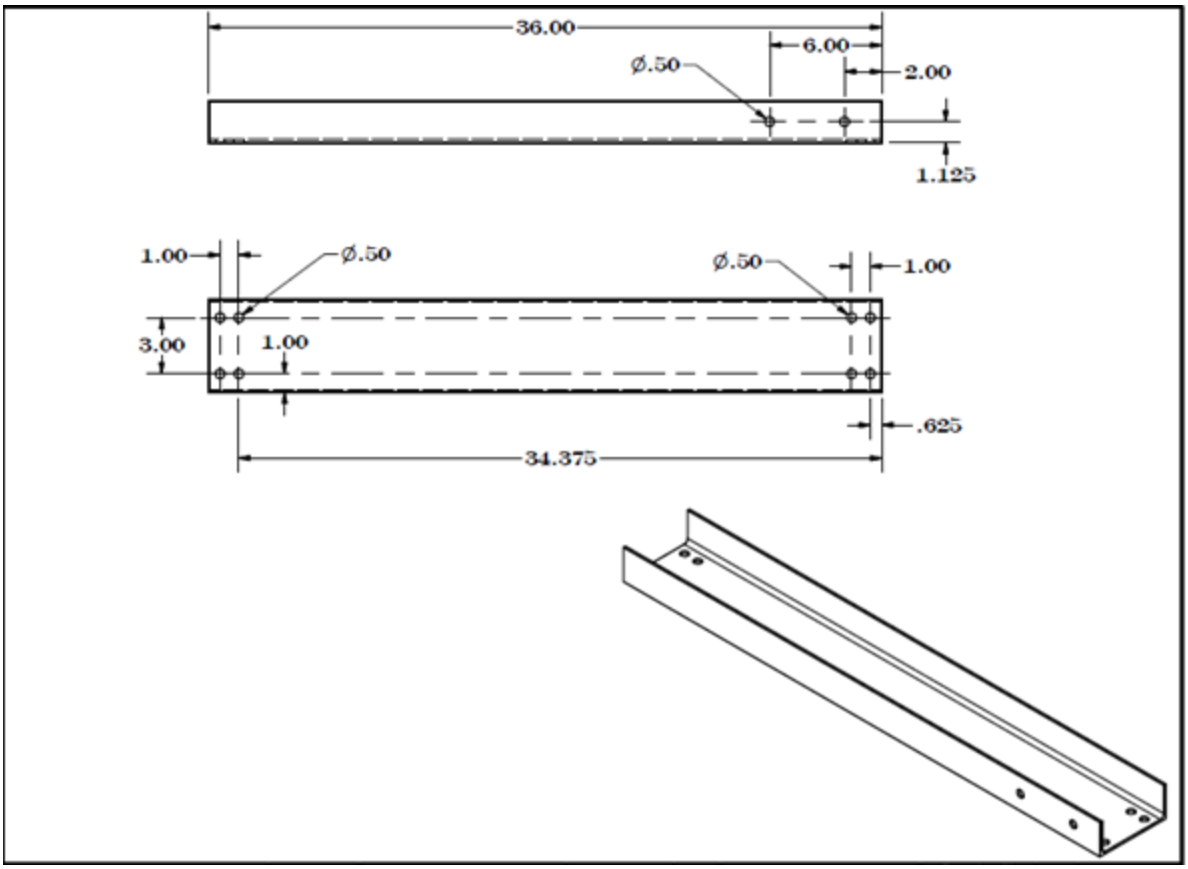

Figure B.23 Side support leg 1B

Quantity: 1

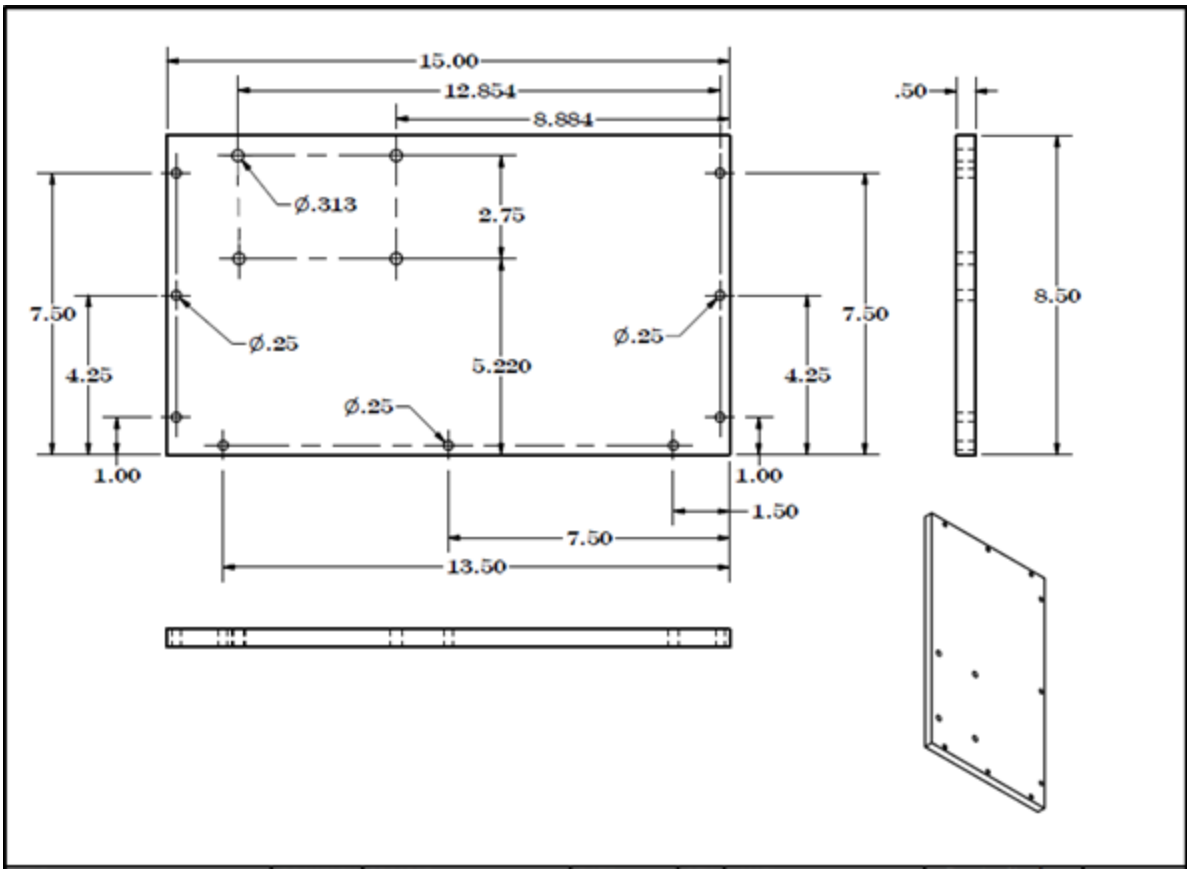

Figure B.24 Motor mount top plate

Quantity: 1

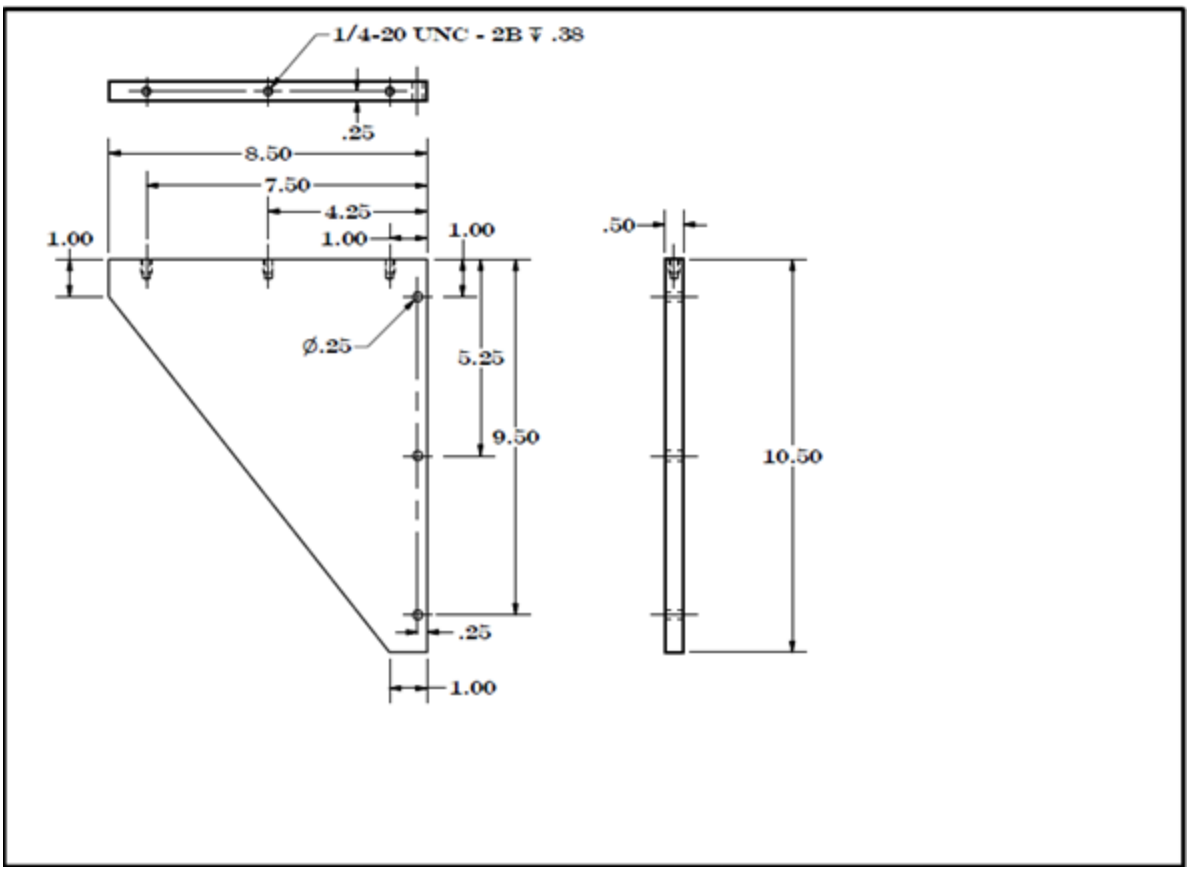

Figure B.25 Motor mount side plate

Quantity: 2

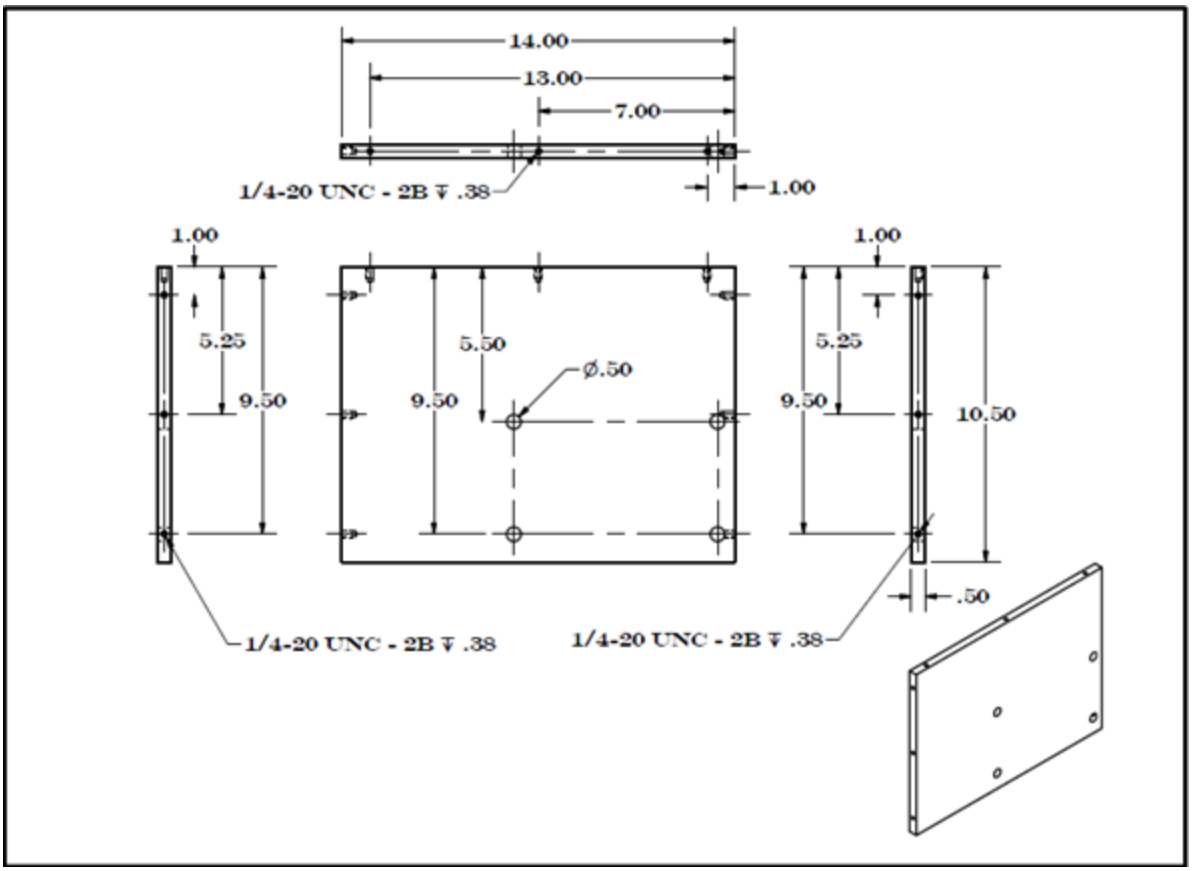

Figure B.26 Motor mount back plate

Quantity: 1

APPENDIX C. BENCH-SCALE MICROALGAL RECOVERY RATE TESTS

Purpose of this test was to determine the percent microalgal recovery for suspensions at a series of concentrations ranging from 2 to 6 g dry wt. /L.

Preparation of microalgal concentrates

Samples of microalgal suspension with a concentration 6 $\pm$ 1 g dry wt. /L obtained from the settling tanks at the Lawrence wastewater treatment plant, Kansas, was re-suspended in deionized water to form sample suspensions at a series of three concentrations ranging from 2 g/L to 6 g/L dry weight. Gravity filtration tests were conducted for each concentrate. The test procedure is listed in the section below.

Microalgal recovery rate tests

The purpose of this test was to determine the percent of microalgae recovered after filtration for microalgal suspensions with concentrations ranging from 2 g/L to 6 g/L dry weight. The test setup was the same as that for mesh size determination. All concentration measurements were made using standard total suspended solids test. Gravity filtration tests were conducted using 70 micron mesh size filter. 5 milliliter samples of concentrated microalgal suspension were used for this test. Each test was conducted in triplicate. The microalgal cake collected on the filter was air dried for 12 hours at ambient conditions. After the 12 hour drying period, dried algae on the filter was scraped off and collected in pre-weighed aluminum weigh boats. The percent of microalgae recovered was calculated using equations 1 and 2.

$$PR\ (\%) = \left(\frac{M_D}{M_I}\right) \cdot 100 \qquad (1)$$

$$M_I = \frac{C_I \cdot V_I}{10^6} \qquad (2)$$

where M_D is the mass of the dried microalgae in grams, M_I is the mass of incoming total suspended solids in grams, C_I is the initial concentration of the microalgal solution in milligrams/Liter and V_I is the initial volume of the microalgal solution in milliliters.

Results of filtration tests are shown in Figure C.1.

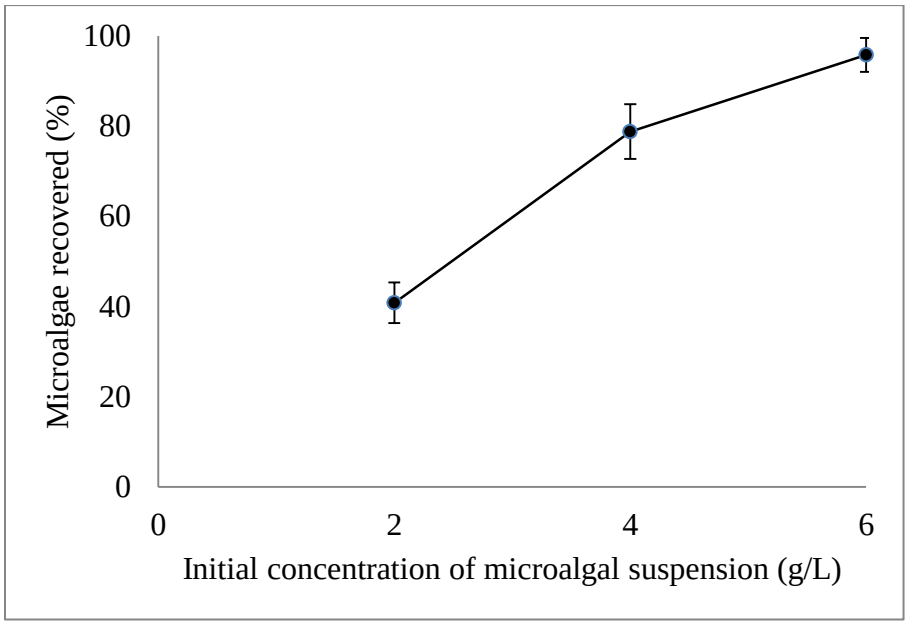

Figure C.1 Bench scale microalgal recovery rate test results for concentrations below 10 g dry wt. /L. Error bars represent standard deviation of measured values of microalgae recovered (n = 3).

This results correlates well with belt filter dewatering test results where a concentrations as low as 6 g dry wt. /L can be effectively recovered.

APPENDIX D. FLOCCULANT MATERIAL SAFETY DATA SHEETS

Alum Material Safety Data Sheet

SIGMA-ALDRICH *sigma-aldrich.com*

Version 5.0

Revision Date 09/03/2012

Print Date 12/23/2013

1. PRODUCT AND COMPANY IDENTIFICATION

Product name: Potassium alum dodecahydrate

Product Number: P7971

Brand: Sigma-Aldrich

Supplier: Sigma-Aldrich

3050 Spruce Street

SAINT LOUIS MO 63103

USA

Telephone: +1 800-325-5832

Fax: +1 800-325-5052

Emergency Phone # (For both supplier and manufacturer): (314) 776-6555

Preparation Information: Sigma-Aldrich Corporation

Product Safety - Americas Region

1-800-521-8956

2. HAZARDS IDENTIFICATION

Emergency Overview

OSHA Hazards

No known OSHA hazards

Not a dangerous substance according to GHS.

HMIS Classification

Health hazard: 0

Flammability: 0

Physical hazards: 0

NFPA Rating

Health hazard: 0

Fire: 0

Reactivity Hazard: 0

Potential Health Effects

Inhalation May be harmful if inhaled. May cause respiratory tract irritation.

Skin May be harmful if absorbed through skin. May cause skin irritation.

Eyes May cause eye irritation.

Ingestion May be harmful if swallowed.

3. COMPOSITION/INFORMATION ON INGREDIENTS

Synonyms: Aluminum potassium sulfate dodecahydrate

Potassium aluminum sulfate dodecahydrate

Alum

Potassium alum

Formula: $AlKO_8S_2 \cdot 12H_2O$

Molecular Weight: 474.39 g/mol

No ingredients are hazardous according to OSHA criteria.

4. FIRST AID MEASURES

If inhaled

If breathed in, move person into fresh air. If not breathing, give artificial respiration.

In case of skin contact

Wash off with soap and plenty of water.

In case of eye contact

Flush eyes with water as a precaution.

If swallowed

Never give anything by mouth to an unconscious person. Rinse mouth with water.

5. FIREFIGHTING MEASURES

Suitable extinguishing media

Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

Special protective equipment for firefighters

Wear self-contained breathing apparatus for firefighting if necessary.

Hazardous combustion products

Hazardous decomposition products formed under fire conditions. - Sulphur oxides, Potassium oxides, Aluminum oxide

6. ACCIDENTAL RELEASE MEASURES

Personal precautions

Avoid dust formation. Avoid breathing vapors, mist or gas.

Environmental precautions

Do not let product enter drains.

Methods and materials for containment and cleaning up

Sweep up and shovel. Keep in suitable, closed containers for disposal.

7. HANDLING AND STORAGE

Precautions for safe handling

Provide appropriate exhaust ventilation at places where dust is formed. Normal measures for preventive fire protection.

Conditions for safe storage

Keep container tightly closed in a dry and well-ventilated place.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Contains no substances with occupational exposure limit values.

Personal protective equipment

Respiratory protection

Respiratory protection is not required. Where protection from nuisance levels of dusts are desired, use type N95

(US) or type P1 (EN 143) dust masks. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Hand protection

Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Eye protection

Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH

(US) or EN 166(EU).

Skin and body protection

Choose body protection in relation to its type, to the concentration and amount of dangerous substances, and to the specific work-place. The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Hygiene measures

General industrial hygiene practice.

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance

Form solid

Color no data available

Safety data

pH 3.3 at 94.88 g/l

Melting point/freezing point

Melting point/range: 92 °C (198 °F) - lit.

Boiling point no data available

Flash point not applicable

Ignition temperature no data available

Auto-ignition temperature: no data available

Lower explosion limit: no data available

Upper explosion limit: no data available

Vapor pressure: no data available

Density 1.757 g/mL at 25 °C (77 °F)

Water solubility no data available

Partition coefficient:

N-octanol/water: no data available

Relative vapor density: no data available

Odor no data available

Odor Threshold no data available

Evaporation rate no data available

10. STABILITY AND REACTIVITY

Chemical stability

Stable under recommended storage conditions.

Possibility of hazardous reactions

No data available

Conditions to avoid

No data available

Materials to avoid

Strong oxidizing agents, Bases, Steel (all types and surface treatments), Aluminum, Copper, Zinc

Hazardous decomposition products

Hazardous decomposition products formed under fire conditions. - Sulphur oxides, Potassium oxides, Aluminum oxide

Other decomposition products - no data available

11. TOXICOLOGICAL INFORMATION

Acute toxicity

Oral LD50

No data available

Inhalation LC50

No data available

Dermal LD50

No data available

Other information on acute toxicity

No data available

Skin corrosion/irritation

No data available

Serious eye damage/eye irritation

No data available

Respiratory or skin sensitization

No data available

Germ cell mutagenicity

No data available

Carcinogenicity

IARC: No components of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.

ACGIH: No components of this product present at levels greater than or equal to 0.1% is identified as a carcinogen or potential carcinogen by ACGIH.

NTP: No components of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.

OSHA: No components of this product present at levels greater than or equal to 0.1% is identified as a carcinogen or potential carcinogen by OSHA.

Reproductive toxicity

No data available

Teratogenicity

Developmental Toxicity - rat - Oral

Effects on Embryo or Fetus: Fetotoxicity (except death, e.g., stunted fetus).

No data available

Specific target organ toxicity - single exposure (Globally Harmonized System)

No data available

Specific target organ toxicity - repeated exposure (Globally Harmonized System)

No data available

Aspiration hazard

No data available

Potential health effects

Inhalation May be harmful if inhaled. May cause respiratory tract irritation.

Ingestion May be harmful if swallowed.

Skin May be harmful if absorbed through skin. May cause skin irritation.

Eyes May cause eye irritation.

Signs and Symptoms of Exposure

Gastrointestinal disturbance: To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

Synergistic effects

No data available

Additional Information

RTECS: WS5690000

12. ECOLOGICAL INFORMATION

Toxicity

No data available

Persistence and degradability

No data available

Bioaccumulative potential

No data available

Mobility in soil

No data available

PBT and vPvB assessment

No data available

Other adverse effects

No data available

13. DISPOSAL CONSIDERATIONS

Product

Offer surplus and non-recyclable solutions to a licensed disposal company.

Contaminated packaging

Dispose of as unused product.

14. TRANSPORT INFORMATION

DOT (US)

Not dangerous goods

IMDG

Not dangerous goods

IATA

Not dangerous goods

15. REGULATORY INFORMATION

OSHA Hazards

No known OSHA hazards

SARA 302 Components

SARA 302: No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

SARA 313 Components

SARA 313: This material does not contain any chemical components with known CAS numbers that exceed the threshold

(De Minimis) reporting levels established by SARA Title III, Section 313.

SARA 311/312 Hazards

No SARA Hazards

Massachusetts Right to Know Components

No components are subject to the Massachusetts Right to Know Act.

Pennsylvania Right to Know Components

Aluminum potassium bis (sulphate)

CAS-No.

7784-24-9

Revision Date

New Jersey Right to Know Components

Aluminum potassium bis (sulphate)

CAS-No.

7784-24-9

Revision Date

California Prop. 65 Components

This product does not contain any chemicals known to State of California to cause cancer, birth defects, or any other reproductive harm.

16. OTHER INFORMATION

Further information

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our

knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

Chitosan Material Safety Data Sheet

SIGMA-ALDRICH *sigma-aldrich.com*

Version 4.4

Revision Date 08/19/2013

Print Date 12/23/2013

1. PRODUCT AND COMPANY IDENTIFICATION

Product name: Chitosan

Product Number: 448869

Brand: Aldrich

Supplier: Sigma-Aldrich

3050 Spruce Street

SAINT LOUIS MO 63103

USA

Telephone: +1 800-325-5832

Fax: +1 800-325-5052

Emergency Phone # (For both supplier and manufacturer): (314) 776-6555

Preparation Information: Sigma-Aldrich Corporation

Product Safety - Americas Region

1-800-521-8956

2. HAZARDS IDENTIFICATION

Emergency Overview

OSHA Hazards

No known OSHA hazards

GHS Classification

Acute aquatic toxicity (Category 2)

GHS Label elements, including precautionary statements

Pictogram none

Signal word none

Hazard statement(s)

H401 Toxic to aquatic life.

Precautionary statement(s): none

HMIS Classification

Health hazard: 0

Flammability: 0

Physical hazards: 0

NFPA Rating

Health hazard: 0

Fire: 0

Reactivity Hazard: 0

Potential Health Effects

Inhalation May be harmful if inhaled. May cause respiratory tract irritation.

Skin May be harmful if absorbed through skin. May cause skin irritation.

Eyes May cause eye irritation.

Ingestion May be harmful if swallowed.

3. COMPOSITION/INFORMATION ON INGREDIENTS

Synonyms: Poly (D-glucosamine)

Deacetylated chitin

No ingredients are hazardous according to OSHA criteria.

4. FIRST AID MEASURES

General advice

Consult a physician. Show this safety data sheet to the doctor in attendance.

If inhaled

If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact

Wash off with soap and plenty of water. Consult a physician.

In case of eye contact

Flush eyes with water as a precaution.

If swallowed

Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

5. FIREFIGHTING MEASURES

Conditions of flammability

Not flammable or combustible.

Suitable extinguishing media

Use water spray, alcohol-resistant foam, dry chemical or carbon dioxide.

Special protective equipment for firefighters

Wear self-contained breathing apparatus for firefighting if necessary.

Hazardous combustion products

Hazardous decomposition products formed under fire conditions. - Carbon oxides, nitrogen oxides (NOx)

6. ACCIDENTAL RELEASE MEASURES

Personal precautions

Avoid dust formation. Avoid breathing vapors, mist or gas. Ensure adequate ventilation.

Environmental precautions

Prevent further leakage or spillage if safe to do so. Do not let product enter drains. Discharge into the environment must be avoided.

Methods and materials for containment and cleaning up

Pick up and arrange disposal without creating dust. Sweep up and shovel. Keep in suitable, closed containers for disposal.

7. HANDLING AND STORAGE

Precautions for safe handling

Provide appropriate exhaust ventilation at places where dust is formed.

Conditions for safe storage

Keep container tightly closed in a dry and well-ventilated place.

Keep in a dry place.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Contains no substances with occupational exposure limit values.

Personal protective equipment

Respiratory protection

Respiratory protection is not required. Where protection from nuisance levels of dusts are desired, use type N95 (US) or type P1 (EN 143) dust masks. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Hand protection

Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Full contact

Material: Nitrile rubber

Minimum layer thickness: 0.11 mm

Break through time: 480 min

Material tested: Dermatril® (KCL 740 / Aldrich Z677272, Size M)

Splash contact

Material: Nitrile rubber

Minimum layer thickness: 0.11 mm

Break through time: 480 min

Material tested: Dermatril® (KCL 740 / Aldrich Z677272, Size M)

Data source: KCL GmbH, D-36124 Eichenzell, phone +49 (0)6659 87300, e-mail sales@kcl.de, test method: EN374

If used in solution, or mixed with other substances, and under conditions which differ from EN 374, contact the supplier of the CE approved gloves. This recommendation is advisory only and must be evaluated by an industrial hygienist and safety officer familiar with the specific situation of anticipated use by our customers. It should not be construed as offering an approval for any specific use scenario.

Eye protection

Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin and body protection

Choose body protection in relation to its type, to the concentration and amount of dangerous substances, and to the specific work-place. The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Hygiene measures

Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance

Form powder

Color beige

Safety data

pH no data available

Melting point/freezing point: no data available

Boiling point: no data available

Flash point: no data available

Ignition temperature no data available

Auto-ignition temperature: no data available

Lower explosion limit: no data available

Upper explosion limit: no data available

Vapor pressure: no data available

Density: no data available

Water solubility: no data available

Partition coefficient: n-octanol/water: no data available

Relative vapor density: no data available

Odor: no data available

Odor Threshold: no data available

Evaporation rate: no data available

10. STABILITY AND REACTIVITY

Chemical stability

Stable under recommended storage conditions.

Possibility of hazardous reactions

No data available

Conditions to avoid

No data available

Materials to avoid

Strong oxidizing agents

Hazardous decomposition products

Hazardous decomposition products formed under fire conditions. - Carbon oxides, nitrogen oxides (NO_x).

Other decomposition products - no data available

11. TOXICOLOGICAL INFORMATION

Acute toxicity

Oral LD50

LD50 Oral - rat - > 10,000 mg/kg

Inhalation LC50

No data available

Dermal LD50

No data available

Other information on acute toxicity

No data available

Skin corrosion/irritation

No data available

Serious eye damage/eye irritation

No data available

Respiratory or skin sensitization

No data available

Germ cell mutagenicity

No data available

Carcinogenicity

IARC: No components of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.

ACGIH: No components of this product present at levels greater than or equal to 0.1% is identified as a carcinogen or potential carcinogen by ACGIH.

NTP: No components of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.

OSHA: No components of this product present at levels greater than or equal to 0.1% is identified as a carcinogen or potential carcinogen by OSHA.

Reproductive toxicity

No data available

Teratogenicity

No data available

Specific target organ toxicity - single exposure (Globally Harmonized System)

No data available

Specific target organ toxicity - repeated exposure (Globally Harmonized System)

No data available

Aspiration hazard

No data available

Potential health effects

Inhalation May be harmful if inhaled. May cause respiratory tract irritation.

Ingestion May be harmful if swallowed.

Skin May be harmful if absorbed through skin. May cause skin irritation.

Eyes May cause eye irritation.

Signs and Symptoms of Exposure

To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

Synergistic effects

No data available

Additional Information

RTECS: Not available

12. ECOLOGICAL INFORMATION

Toxicity

Toxicity to fish LC50 - Oncorhynchus mykiss (rainbow trout) - 1.73 mg/l - 96 h

Toxicity to daphnia and other aquatic invertebrates

EC50 - Daphnia pulex (Water flea) - 13.69 mg/l - 48 h

Persistence and degradability

No data available

Bioaccumulative potential

No data available

Mobility in soil

No data available

PBT and vPvB assessment

No data available

Other adverse effects

An environmental hazard cannot be excluded in the event of unprofessional handling or disposal.

Toxic to aquatic life.

No data available

13. DISPOSAL CONSIDERATIONS

Product

Offer surplus and non-recyclable solutions to a licensed disposal company.

Contaminated packaging

Dispose of as unused product.

14. TRANSPORT INFORMATION

DOT (US)

Not dangerous goods

IMDG

Not dangerous goods

IATA

Not dangerous goods

15. REGULATORY INFORMATION

OSHA Hazards

No known OSHA hazards

SARA 302 Components

SARA 302: No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

SARA 313 Components

SARA 313: This material does not contain any chemical components with known CAS numbers that exceed the threshold

(De Minimis) reporting levels established by SARA Title III, Section 313.

SARA 311/312 Hazards

No SARA Hazards

Massachusetts Right to Know Components

No components are subject to the Massachusetts Right to Know Act.

Pennsylvania Right to Know Components

Chitosan

CAS-No.

9012-76-4

Revision Date

New Jersey Right to Know Components

Chitosan

CAS-No.

9012-76-4

Revision Date

California Prop. 65 Components

This product does not contain any chemicals known to State of California to cause cancer, birth defects, or any other reproductive harm.

16. OTHER INFORMATION

Further information

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

Zetag 7650 Material Safety Data Sheet

BASF

Revision date: 2011/06/27 Page: 1/7

Version: 2.0 (30482601/SDS_GEN_US/EN)

1. Product and Company Identification

Use: flocculation agent

24 Hour Company Emergency Response Information

BASF CORPORATION

100 Park Avenue

Florham Park, NJ 07932, USA

CHEMTREC: 1-800-424-9300

BASF HOTLINE: 1-800-832-HELP (4357)

2. Hazards Identification

Emergency overview

CAUTION:

The product can cause skin and eye irritation.

May cause some irritation to the respiratory system if dust is inhaled.

Avoid the formation and deposition of dust.

Avoid sources of ignition.

Refer to MSDS Section 7 for Dust Explosion information.

Caution - Slippery when wet!

Combustible organic powder.

Avoid creating dusty conditions, dust build-up or formation of dust clouds.

Avoid all sources of ignition: heat, sparks, and open flame.

State of matter: solid

Color: off-white

Odor: odorless

Potential health effects

Primary routes of exposure:

Routes of entry for solids and liquids include eye and skin contact, ingestion and inhalation. Routes of entry for gases include inhalation and eye contact. Skin contact may be a route of entry for liquefied gases.

Chronic toxicity:

Carcinogenicity: None of the components in this product at concentrations greater than 0.1% are listed by

IARC; NTP, OSHA or ACGIH as a carcinogen.

Reproductive toxicity: No data for product. No effects anticipated

Genotoxicity: The chemical structure does not suggest such an effect.

Signs and symptoms of overexposure:

No significant symptoms are expected due to the non-classification of the product.

Potential environmental effects

Aquatic toxicity:

Fish toxicity and aquatic toxicity are drastically reduced by rapid irreversible adsorption onto suspended and/or dissolved organic matter. Acute effects on aquatic organisms are due to the cationic charge of the polymer, which is quickly neutralized in natural water courses by irreversible adsorption onto particles, hydrolysis and dissolved organic carbon. The hydrolysis products are not acutely harmful to aquatic organisms.

3. Composition / Information on Ingredients

CAS Number Content (W/W) Chemical name

124-04-9 3.0 - 7.0 % adipic acid

69418-26-4 85.0 - 90.0 % Ethanaminium, N, N, N-trimethyl-2-[(1-oxo-2-propenyl) oxy]-chloride, polymer with 2-propenamide

4. First-Aid Measures

General advice:

Remove contaminated clothing.

If inhaled:

If difficulties occur after dust has been inhaled, remove to fresh air and seek medical attention.

If on skin:

Wash thoroughly with soap and water.

If irritation develops, seek medical attention.

If in eyes:

Wash affected eyes for at least 15 minutes under running water with eyelids held open.

Seek medical attention.

If swallowed:

Rinse mouth and then drink plenty of water. Do not induce vomiting. Immediate medical attention required.

Note to physician

Treatment: Treat according to symptoms (decontamination, vital functions), no known specific antidote.

5. Fire-Fighting Measures

Flash point: not applicable

Self-ignition temperature: > 530 °C Data for powdery solid.

Suitable extinguishing media:

Dry powder, foam

Unsuitable extinguishing media for safety reasons:

Water jet, carbon dioxide

Additional information:

If water is used, restrict pedestrian and vehicular traffic in areas where slip hazard may exist.

Hazards during fire-fighting:

Carbon oxides, nitrogen oxides

The substances/groups of substances mentioned can be released in case of fire. Very slippery when wet.

Protective equipment for fire-fighting:

Wear a self-contained breathing apparatus.

Further information:

The degree of risk is governed by the burning substance and the fire conditions. Contaminated extinguishing water must be disposed of in accordance with official regulations.

6. Accidental release measures

Personal precautions:

Use personal protective clothing.

Environmental precautions:

Do not discharge into drains/surface waters/groundwater.

Cleanup:

Spilled product which becomes wet or spilled aqueous solution creates a hazard because of their slippery nature.

Avoid raising dust.

For small amounts: Pick up with suitable appliance and dispose of.

For large amounts: Contain with dust binding material and dispose of.

7. Handling and Storage

Handling

General advice:

Breathing must be protected when large quantities are decanted without local exhaust ventilation. Handle in accordance with good industrial hygiene and safety practice. Forms slippery surfaces with water.

Storage

General advice:

Store in unopened original containers in a cool and dry place. Avoid wet, damp or humid conditions, temperature extremes and ignition sources.

Storage stability:

Avoid extreme heat.

8. Exposure Controls and Personal Protection

Personal protective equipment

Respiratory protection:

Wear a NIOSH-certified (or equivalent) organic vapor/particulate respirator.

Hand protection:

Chemical resistant protective gloves

Eye protection:

Safety glasses with side-shields.

General safety and hygiene measures:

Wear protective clothing as necessary to minimize contact. Handle in accordance with good industrial hygiene and safety practice.

9. Physical and Chemical Properties

Form: powder

Odor: odorless

Color: off-white

pH value: 3.5 - 4.5 (10 g/l)

Melting point: The substance / product decomposes therefore not determined.

Boiling point: not applicable

Bulk density: approx. 750 kg/m^3

Partitioning coefficient

N-octanol/water (log Pow): not applicable

% volatiles: 0 %

Solubility in water: Forms a viscous solution.

Other Information: If necessary, information on other physical and chemical parameters is indicated in this section.

10. Stability and Reactivity

Conditions to avoid:

Avoid extreme temperatures. Avoid humidity.

Substances to avoid:

strong acids, strong bases, strong oxidizing agents

Hazardous reactions:

The product is not a dust explosion risk as supplied; however the build-up of fine dust can lead to a risk of dust explosions.

Decomposition products:

No hazardous decomposition products if stored and handled as prescribed/indicated.

Corrosion to metals:

No corrosive effect on metal.

11. Toxicological information

Acute toxicity

Oral:

Type of value: LD50

Species: rat

Value: > 5,000 mg/kg (OECD Guideline 401)

Irritation / corrosion

Skin:

Species: rabbit

Result: non-irritant

Method: OECD Guideline 404

Eye:

Species: rabbit

Result: non-irritant

Information on: adipic acid

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Species: rabbit

Result: Risk of serious damage to eyes.

Method: OECD Guideline 405

Other Information:

The product has not been tested. The statements on toxicology have been derived from products of a similar

Structure and composition.

12. Ecological Information

Fish

Acute: static

LC50 (96 h): 10 - 100 mg/l

Aquatic invertebrates

Acute: EC50 (48 h): 10 - 100 mg/l

Degradability / Persistence

Biological / Abiological Degradation

Evaluation: Not readily biodegradable (by OECD criteria).

Hydrolysis

In contact with water the substance will hydrolyze rapidly.

Environmental mobility:

Information on: cationic polyacrylamide

Assessment transport between environmental compartments:

Adsorption to solid soil phase is expected.

Other adverse effects:

The product has not been tested. The statement has been derived from products of a similar structure or composition.

13. Disposal considerations

Waste disposal of substance:

Dispose of in accordance with national, state and local regulations.

Container disposal:

Dispose of in a licensed facility. Recommend crushing, puncturing or other means to prevent unauthorized use of used containers.

RCRA:

Not a hazardous waste under RCRA (40 CFR 261).

14. Transport Information

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Land transport

USDOT

Not classified as a dangerous good under transport regulations

Sea transport

IMDG

Not classified as a dangerous good under transport regulations

Air transport

IATA/ICAO

Not classified as a dangerous good under transport regulations

15. Regulatory Information

Federal Regulations

Registration status:

Chemical TSCA, US released / listed

OSHA hazard category: Chronic target organ effects reported; ACGIH TLV established

EPCRA 311/312 (Hazard categories): Not hazardous;

State regulations

State RTK CAS Number Chemical name

MA, NJ, PA 124-04-9 adipic acid

CA Prop. 65:

THIS PRODUCT CONTAINS A CHEMICAL(S) KNOWN TO THE STATE OF CALIFORNIA TO CAUSE

CANCER AND BIRTH DEFECTS OR OTHER REPRODUCTIVE HARM.

16. Other Information

NFPA Hazard codes:

Health: 2 Fires: 1 Reactivity: 0 Special: -

HMIS III rating

Health: 2 Flammability: 1 Physical hazard: 0

NFPA and HMIS use a numbering scale ranging from 0 to 4 to indicate the degree of hazard. A value of zero means that the substance possesses essentially no hazard; a rating of four indicates extreme danger. Although similar, the two rating systems are intended for different purposes, and

use different criteria. The NFPA system was developed to provide an on-the-spot alert to the hazards of a material, and their severity, to emergency responders. The HMIS system was designed to communicate workplace hazard information to employees who handle hazardous chemicals.

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MSDS Prepared by:

BASF NA Product Regulations

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MSDS Prepared on: 2011/06/27

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