

2012

# Development of solid-liquid separation technologies in bioprocessing

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# **Development of solid-liquid separation technologies in bioprocessing**

by

**Christopher Robert Koza**

A thesis submitted to the graduate faculty  
in partial fulfillment of the requirements for the degree of  
**MASTER OF SCIENCE**

Major: Civil Engineering (Environmental Engineering)

Program of Study Committee:  
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Iowa State University  
Ames, Iowa  
2012

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**ABSTRACT**

An innovative bioprocess utilizing thin stillage from dry-grind corn ethanol plants was used to produce a valuable filamentous fungus (*R. microsporus* var. *oligosporus*). A 1,500-L bioreactor was built to optimize operating parameters, produce fungal biomass for animal feed trials and develop equipment and processes for industrialization. Dewatering equipment was tested to determine proper solid-liquid separation equipment for the industrial process. Studies indicate that a two-step dewatering process is most effective utilizing a gravity fed, concave screen followed by a centrifuging filter. Solids content greater than 30 percent is not possible, as this value approaches theoretical limits due to intracellular water. This new fungal co-product has some superior feed properties in comparison to distillers grains. Selection of proper processing equipment minimizes capital and operating costs, making this process economically feasible for ethanol producers or other investors.

Methods to concentrate and remove phosphorus from a waste bioprocessing stream utilized as a fertilizer was investigated to produce two more desirable products, liquid fertilizer with a larger nitrogen to phosphorus (N:P) ratio and a “solid” phase with a lower N:P. A large percentage of phosphorus in this liquid slurry, left over from an industrial fermentation process, is tied up in the suspended solids. The study focuses on the use of coagulants and polymers to aid separation of suspended solids from the bulk liquid, ultimately producing a liquid fertilizer with a higher nitrogen to phosphorus ratio over the initial liquid slurry.

## CHAPTER 1. INTRODUCTION

### Introduction

The ethanol industry has seen marked growth in the past decade. Demand for transportation fuels keeps the industry producing record volumes of ethanol. Those knowledgeable in the field appreciate the benefits, however controversy persists. Among the various criticisms, the food-vs-fuel debate is a hot topic. The ethanol industry is using more corn than ever before, which could be used for livestock feeding.

Additionally, as commodity markets remain volatile, corn prices fluctuate and are undesirable for the ethanol industry. High corn or energy prices have resulted in low profit margins for the industry. As technology advances, ethanol plants will implement systems to increase profit margins, which could make the difference between profitability and bankruptcy.

For dry-grind ethanol plants, distillers grains has been the main co-product, sold as a livestock feed supplement. Large volumes are produced and sold, making this item essential towards ethanol profitability. However, the product has many limitations. The US market is nearly saturated, the nutritional profile is not favorable for many animals and ultimately distillers grains cannot be sold for a high value.

Research on new co-products for the ethanol industry strives to keep the industry profitable. Fungal cultivation on thin stillage, a low-value byproduct of dry-grind ethanol fermentation, is a new technology that can address the current issues in the ethanol industry. Fungal cultivation on thin stillage has the ability to create a new

value-added feed co-product (high-protein fungal biomass). This co-product has many nutritional benefits over distillers grains and could also have a higher market value. Installation of fungal cultivation equipment in a dry-grind ethanol plant could increase profit margins.

Disposal of waste products has always been a challenge for many industries. Land application, particularly farmland, is one solution for many organic waste products. This cost-effective disposal method has added value to many products destined for landfills or costly advanced treatment. Perspective leftover wastes, high in nutrients including nitrogen and phosphorus, can be considered as potential fertilizer sources. However, these sources may need treatment for individual application scenarios. Nitrogen to phosphorus ratios and nutrient concentrations play a critical role in determining the type and quantity of fertilizer used. Reformulation of the waste product allows farmers the ability to efficiently supply nutrients to their crops.

Separation of an organic waste stream into a concentrated phosphorus fertilizer and concentrated nitrogen fertilizer would give farmers control of nutrient loading on farmlands. Greater separation of the nutrients adds value to the product and justified research on this stream. The work supplements the research on separation and dewatering of biomass quite well.

## **Thesis Organization**

The thesis is organized into three main parts, a literature review and two research papers. The literature review outlines the motivation behind the start of the ethanol industry, current state of the ethanol industry, its benefits, challenges, production process and co-products. The detailed description of the ethanol industry illustrates the motivation behind this fungal biomass research project. Descriptions of solid-liquid separation technologies are outlined to indicate current technology and uses for these processes. Besides solid-liquid separation technologies, details on coagulants, polymers and flocculation are presented to provide insight for the process developed for Amana Farms, located in eastern Iowa, which operates/runs/manages 4,000 acres of corn, 2,500 acres of soybeans, 250 acres of wheat and 2,400-head cow/calf herd.

Chapter 2 investigates dewatering of fungal biomass grown on thin stillage. The study identified industrial equipment suitable for dewatering fungal biomass. Chapter 3 identifies a solid-liquid separation process for phosphorus removal and recovery from a bioprocessing waste.

## CHAPTER 2. LITERATURE REVIEW

### 1. US Ethanol Industry

Development of the US corn ethanol industry has shaped United States energy policies over the past four decades. The policies that promote increased domestic production of corn ethanol help to solve national, economic and environmental security concerns. Increased production of corn ethanol led to more US jobs, reduced reliance on foreign oil supplies and lowered release of greenhouse gas. This review demonstrates how and why the United States invested resources to promote the ethanol industry and the benefits of ethanol as part of the United States energy portfolio.

#### Current state of ethanol

American consumers purchase over 13 billion gallons of gasoline per year, since 2010. Approximately 95% of this fuel was blended with ethanol to make E10 fuel [1]. As the market approaches E10 saturation, policymakers work on approving an E15 fuel blend, containing 15% ethanol. This blend can create 5 billion gallons in ethanol demand. After almost constant production of 2 billion gallons between 1998 and 2003, US ethanol hit record production levels of almost 14 billion gallons in 2011 (Fig. 2.1).



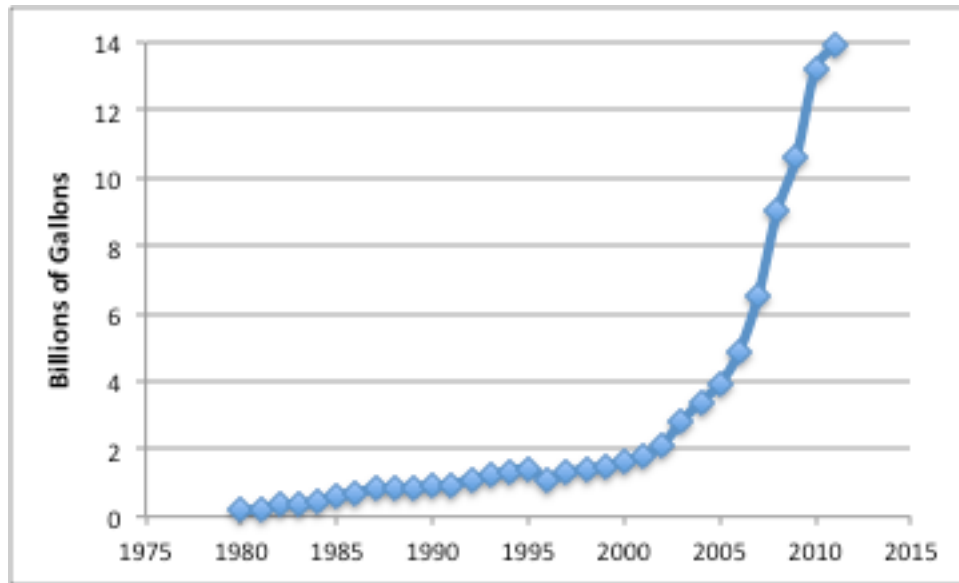


Figure 2.1: Annual US ethanol production [1]

In January 2012, 209 first generation ethanol facilities were online with a total capacity of 14.9 billion gallons ethanol per year and 2 new facilities were under construction that will add 140 million gallons ethanol per year [1]. Technological advancements, tax incentives and fuel mandates will help second-generation ethanol facilities. The Renewable Fuel Standard II mandate wants to bring cellulosic ethanol production to 16 billion gallons in 2022. The United States ethanol industry will continue to play an important role in energy markets for years to come.

### Benefits of ethanol production

As described above, the rise of ethanol production in the United States has economic, environmental and national security benefits. In 2011, 13.9 billion gallons of ethanol was produced and this production economically benefited the United States with 90,200 direct jobs, and 311,400 indirect and induced jobs, and an added \$29.9

billion in household income and \$42.4 billion to the national Gross Domestic Product [1]. As the US ethanol industry continues to expand and mature, it looks to new markets to sell its products. In 2011, more than one billion gallons of ethanol and eight million metric tons of distillers grains were exported [1]. Most ethanol exports, 78,000 barrels per day during 2011, went to Brazil [2]. However, ethanol exports averaged below 10 percent. Distillers grains exports account for over 20 percent of total production, with Mexico and Canada accounting for the majority of exports. In 2008, 4.5 million metric tons of distillers grains were valued close to \$1 billion [3]. International co-product demand will continue to rise over the next decade, as international buyers appreciate the benefits of products like distillers dried grains.

In respect to national security, the United States relies heavily on petroleum for products including gasoline, chemicals and plastics. The US consumes around 25 percent of the world's petroleum and have 3 percent of the world's reserves [4] With the majority (two thirds) of proven petroleum reserves located in the middle east, the United States and other countries rely on many unstable governments and regimes. This dependence on Middle Eastern oil keeps unwelcome US military forces tied up in the area and forces foreign policy compromises [4]. Ethanol provides the United States a domestically produced transportation fuel. Increased production of this domestic fuel could lead to a lower dependence on foreign oil, leading to a smaller US military presence in the Middle East, improving foreign relations and national security concerns.

### Criticism of ethanol production

Besides benefits attributed to the US ethanol industry, critics continue to address shortcomings with this biofuel. In recent years, the production of US ethanol has increased 800% since 2000 [1]. During this time, food prices have increased, creating controversy which links the increased corn demand necessary for ethanol production with food prices. Some critics state that the US farmers cannot produce enough corn to sufficiently supply energy for transportation fuels without starving the world's poor. Other critics directly blame the US ethanol industry for the rise in food prices. Additionally, after the US drought in Summer 2012, United Nations officials petitioned to drop US ethanol mandates in order to make more corn supplies available to global markets.

However, the debate has another side to the story. Over the years, as more corn has been utilized for ethanol, there have been increases in corn yields with the focus on productivity, not diverting supplies from other uses.

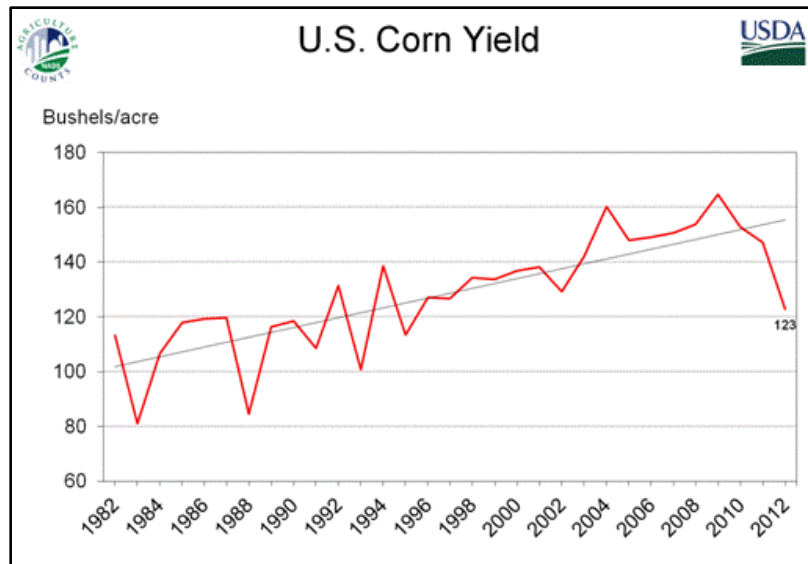


Figure 2.2: Annual US corn yields [5]

Increases in corn yields, shown in Fig. 2.2, are mainly due to stress tolerance and management practices. Genetic engineering of corn has allowed farmers to plant more seeds per acre while tolerating varying environmental factors [6]. The advances in corn genetics and management practices over the past 30 years have resulted in steadily increasing crop yields. Corn yields in the 1970's averaged 89.8 bushels per acre and has increased by more than 60% to 145.5 bushels per acre in the 2000's [7]. With programs like the National Corn Growers Association's Corn Yield Contest, farmers in the United States will continue to harvest more corn by learning best management practices from those who succeed [8]. In addition, seed companies project increases in corn yields to 250-300 bushels per acre by 2030 [8].

Buried in the discussion, animal feed co-products derived from corn ethanol production are part of many animal feed diets. Distillers grains, a major part of beef and dairy cattle diets, displace a large portion of corn utilized for ethanol. For every 56-

pound bushel of corn utilized for ethanol production, 2.8-gallons of ethanol is produced along with 18-pounds of animal feed. So, around 32% of corn can be utilized for ethanol production is placed back in the market as animal feed [9].

Domestic production of corn starch derived ethanol has increased and will continue to increase to 15 billion gallons a year in 2015 based on the Renewable Fuel Standard II. Additionally, from this mandate, cellulosic ethanol mandates will peak at 16 billion gallons per year in 2022 [10].

### Crude oil and ethanol

The United States has always had a large demand for transportation fuels with respect to other countries. In 2010, the United States consumed the most oil of any single country (Fig. 2.3).

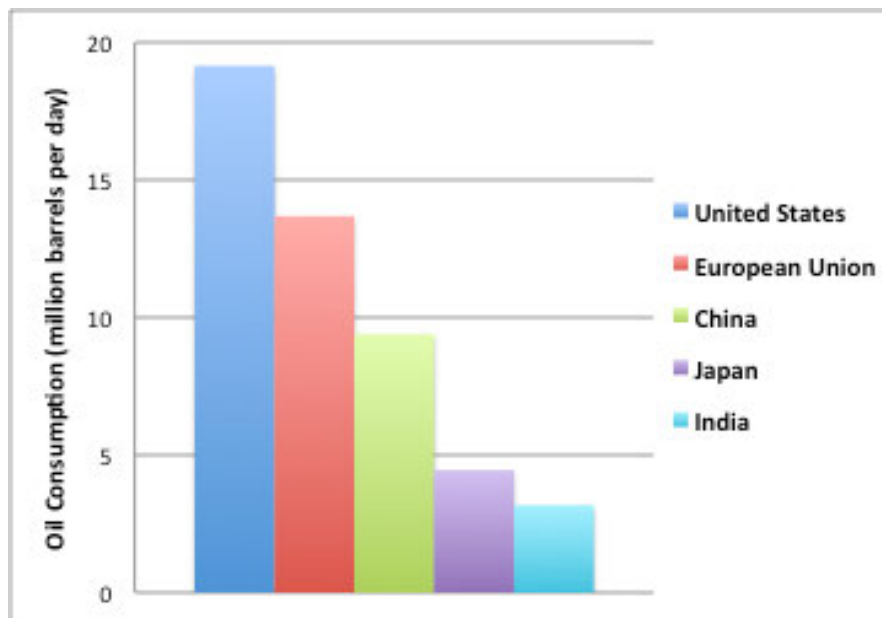


Figure 2.3: Top oil-consuming countries [11]

This demand, beginning in the 1920's, really began to cause problems in the 1970's. After peak US oil production, around 1970, the United States began importing more and more oil, demonstrated in Fig. 2.4. Additionally, as seen in Fig. 2.5, much of US oil comes from unstable political systems in areas of the Middle East or Venezuela. These unstable countries can create problems for the United States that include unsteady supplies and pricing.

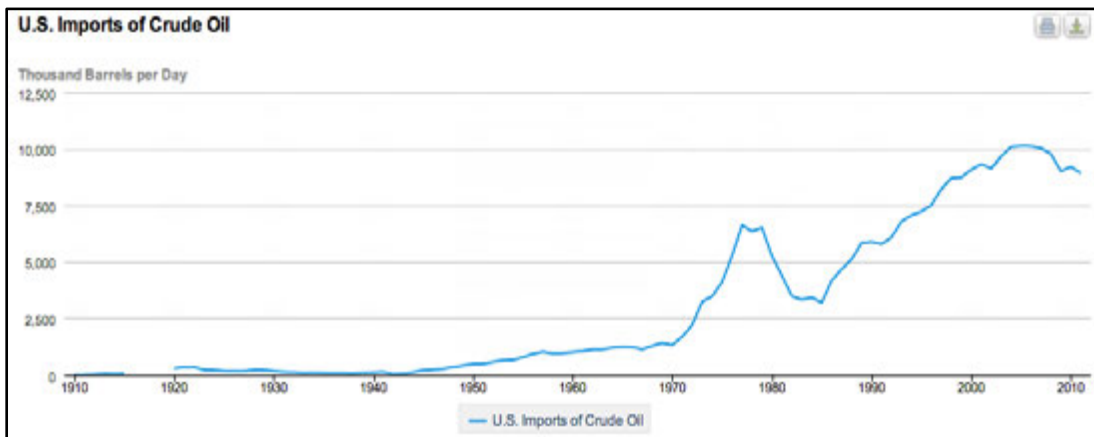


Figure 2.4: Historical US oil imports [12]

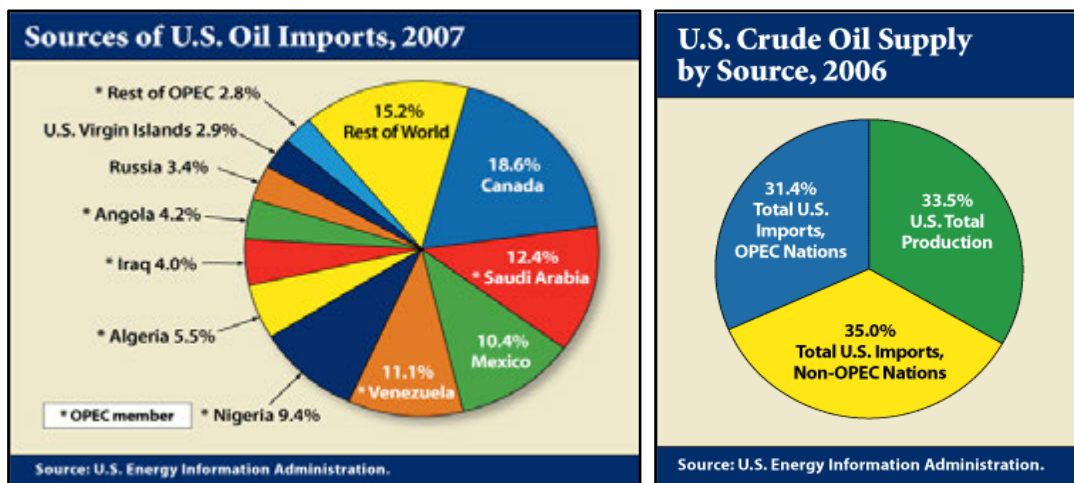


Figure 2.5: US oil supply breakdown, 2006/2007 [13]

\*OPEC: Organization of the Petroleum Exporting Countries

Economic and national security is threatened when supplies are disrupted or energy prices change rapidly. These security issues are also of importance in the long run when world oil demand continues to rise and eventually the production peaks. This peak production will fall and lead to increased prices. The United States needs to find solutions to these issues before they become a threat to national and economic security.

### *Increased Demand*

The oil demand for most industrialized countries has leveled off in recent years. Energy independence, energy conservation and increased public transportation have led to decreased oil demand for some countries. The same is not true for many developing countries, China and India in particular. Projected increases in oil consumption, illustrated in Fig. 2.6, may lead to higher gasoline prices.

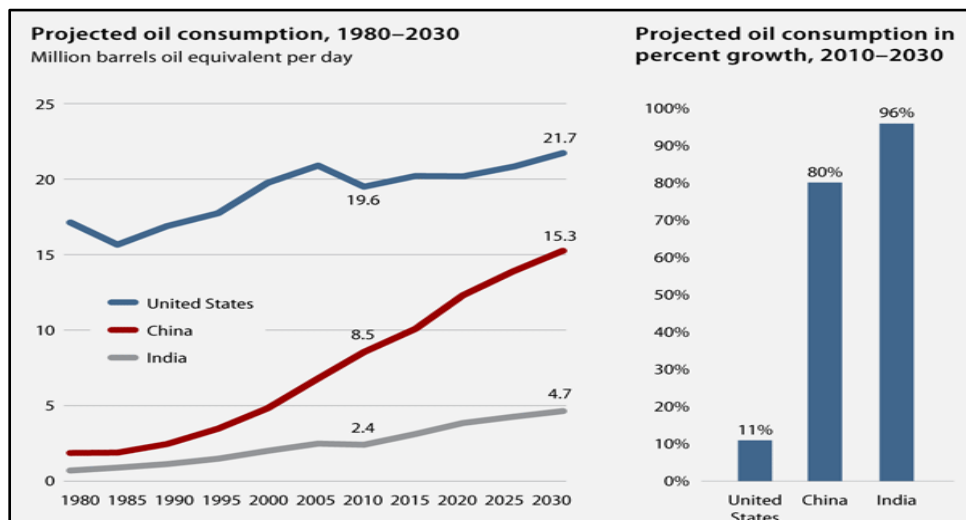


Figure 2.6: Projected Oil Consumption-China, India and USA [14]

## US government programs and ethanol

### *Federal Energy Tax Policies*

United States tax and energy policies were focused on domestic oil and gas production up to the oil shocks of the 1970's. These policies aimed at increasing domestic production and had no focus on energy conservation. Tax policies allowed intangible drilling costs, labor costs, materials and supplies associated with well drilling, to be deducted from tax returns. This increased incentives to find and utilize new oil reserves in the United States. Also, oil and gas percentage depletion allowance granted the oil and gas companies the ability to claim up to 27.5% of revenues as a deduction for the cost of exhausting or depleting an oil deposit [10]. These tax policies encouraged the extraction of oil and locating new reserves. Overall these tax policies kept oil prices low and consumption high. It inhibited the development of alternative fuels, such as ethanol.

Due to the energy crisis in the 1970's, politician and public opinions created an increased awareness for domestic and renewable sources of energy for the United States. This increased awareness resulted in a change in energy policies, research funding and tax restructuring. Federal budget revenue losses associated with the oil and gas industries became hard to justify. This came at a time when the federal government had budget deficits, so many politicians began to consider the preferential treatment they were giving the oil and gas industry. The Energy Tax Act of 1978 was created to put new taxes on conventional fossil fuels, taxes or penalties to promote fuel efficiency and promote alternative fuels [10]. The tax structure of earlier decades,



including the deductions for intangible drilling costs and percentage depletion were cut [10]. Also, increased environmental awareness resulted in additional taxes on oil. The funds from this tax were set aside for the “Superfund” program, which puts money into sites with hazardous waste problems. The program created a 40-cent per gallon of ethanol tax exemption on the 4-cent gasoline excise tax.

The Reagan Administration and their view of a “Free-Market Approach” dominated the 1980’s. The idea was that a neutral approach to the tax law would let the market guide the appropriate industries. His administration thought that high gas prices would allow private companies the incentive to create alternative energy technologies and conservation measures [10]. Overall, during the 1980’s, no significant legislation was created to provide additional incentive for energy industries. In 1980, an ethanol import tariff was created. It imposed a 54-cent/gallon charge on imported ethanol. This tariff kept cheaper, mainly Brazilian ethanol, out of the market to help domestic producers. Also, some small modifications were created to the taxing system. They included an increase to 54 cents per gallon ethanol tax exemption and 9 cents per gallon excise tax on gasoline in 1982 [15].

During the 1990’s, focus shifted back towards fuel conservation and alternative sources. Taxes were readjusted in 1990 under the Revenue Provisions of the Omnibus Reconciliation Act of 1990. This act increased the gasoline tax by 5 cents per gallon [13]. This helped increase government revenue and was ultimately used to reduce federal deficit. It also had the goal of reducing consumption due to higher costs. The Energy Policy Act of 1992 created tax deductions allowed on vehicles able to run on

85% ethanol (E85) and required specific vehicle fleets, primarily government, to purchase flex-fuel vehicles. The Transportation Efficiency Act of the 21<sup>st</sup> Century extended ethanol subsidies through 2007 but reduced them to 51 cents per gallon by 2005 [15].

Overall, during this time period, oil prices were very low. From 1986 to 1999, oil prices averaged \$17/ barrel [10]. Historical oil prices are illustrated below in Fig. 2.8. Low oil cost disincentivised conservation and alternative fuel technologies. In recent years, volatility in the energy market created more incentive to invest in the US homegrown fuel. Between 2008 and 2012, changes in crude oil prices were more volatile than in the previous four years as seen in Fig. 2.7 and 2.8.

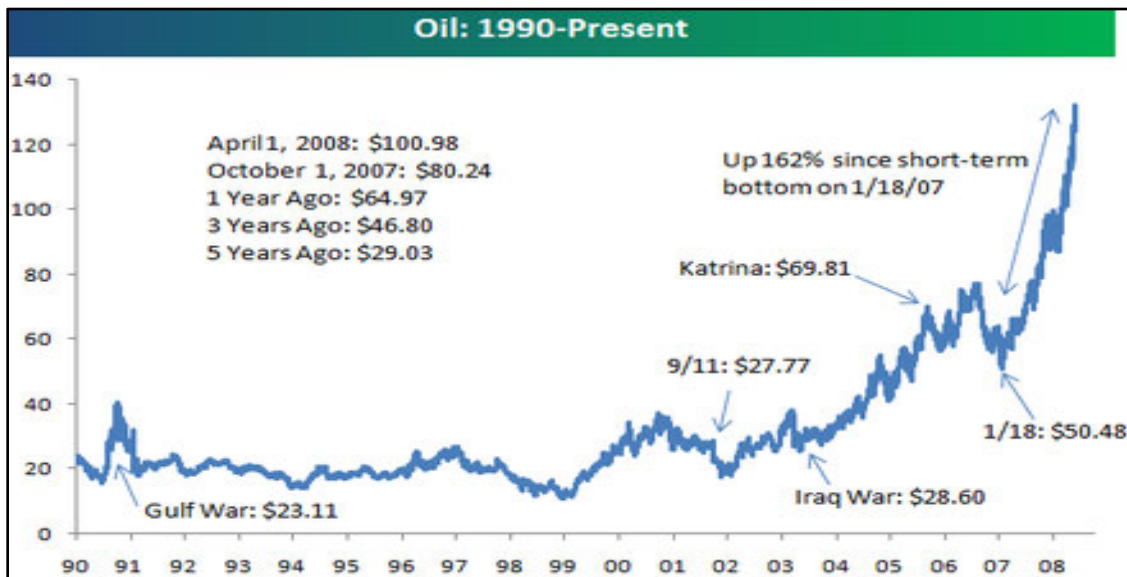


Figure 2.7: Crude oil prices: 1990-2008 [7]



Figure 2.8: Crude oil prices (US dollars): 2007- Present [16]

In 2004, as part of the American Jobs Creation Act, an excise tax credit was created and the mechanism of the old ethanol subsidy program was changed. This tax credit, Volumetric Ethanol Excise Tax Credit (VEETC), provided oil companies a tax incentive to blend ethanol with their fuels. The credit, 51 cents per gallon of ethanol for the E10 fuel blend was created. It was later reduced to 45 cents per gallon [17]. It extended the ethanol tax exemption until 2010. This change in the tax structure gave the oil companies a better incentive to blend ethanol into their fuels. Around the same time, the small ethanol producers tax credit was created. It allows ethanol producers who manufacture less than 60 million gallons of ethanol per year to qualify for a tax credit. The tax credit was 10 cents/gallon of ethanol up to 15 million gallons. So, a maximum of \$1.5 million/ annually was set [17].

In December 2011 the ethanol tax credit expired. The tax credit was in place over 30 years and cost the government nearly \$6 billion in 2011. Now the only tax credit in legislation for ethanol is the \$1.01/gallon cellulosic tax credit for producers.

Overall, policies encouraging domestic production of ethanol were successful in creating a thriving industry.

### *Environmental regulations*

In 1990, an amendment to the Clean Air Act was enacted to combat acid rain, urban air pollution and toxic air emissions. Much of urban air pollution is attributed to vehicle emissions. Problems of smog, carbon monoxide and particulate matter were the main problems tackled in this legislation. New control measures and technologies needed to be implemented to cut down pollution. A new program requiring cleaner “reformulated” fuel was required for the nine metropolitan areas with the worst ozone problems. These areas included Baltimore, Chicago, Hartford, Houston, Los Angeles, Milwaukee, New York City, Philadelphia and San Diego [18]. Other cities were able to opt into this program. Fuel oxygenates were blended in greater percentages to cut down pollution by promoting more complete combustion of hydrocarbons [16]. Methyl tert-butyl ether (MTBE) was the main fuel oxygenate of the time. California, in 1997, blended 11-15% volume MTBE into 90% of its gasoline supply [16]. As time passed, scientists found harmful effects of MTBE as it made its way to the environment via the atmosphere (precipitation) and leaking underground storage tanks [16]. Ethanol is another chemical that can be used as a fuel oxygenate. This made ethanol a prime chemical additive for gasoline [19]. MTBE was banned in many states across the US. These states accounted for 42% of national MTBE consumption, which now use ethanol as a fuel additive. Partial MTBE bans (<0.6% vol. in gasoline) have been enacted by

states including Iowa, South Dakota, Washington, Kansas, Nebraska, Illinois, Indiana, Wisconsin, Ohio, Missouri, Maine and New Hampshire. Complete MTBE bans have been enacted by New York, Connecticut, Michigan, California, Colorado and Minnesota [20].

### *Renewable fuel standard*

The renewable fuel standard was first created as an ethanol production goal under the Energy Policy Act of 2005. It went into effect in 2006. Its original goal was 4 billion gallons per year (BGY) and was increased to 7.5 BGY in 2012. However, as production levels quickly exceeded outdated legislation, the Renewable Fuel Standard 2 (RFS2) changed the policy under the Energy Independence and Security Act of 2007 [10]. This plan expanded the original plan to mandate 36 BGY by 2022. Of the 36 billion gallons, no more than 15 BGY can be from cornstarch based ethanol and no less than 16 BGY from cellulosic biofuels. Table 2.1 lays out all biofuel mandates until 2023.

This new regulation includes certain restrictions that include nested bio-based fuel categories, which include total renewable fuels, advanced biofuels, biomass-based diesel and cellulosic ethanol, which each have their own production volumes. The biofuels must also achieve certain minimum thresholds of lifecycle greenhouse gas (GHG) emission reductions.

Overall, this program is aimed at moving forward towards more advanced biofuels and is shown by the cap on cornstarch derived ethanol. This move away from cornstarch derived ethanol helps illustrate the current state of the ethanol industry and upcoming new technologies. The US corn based ethanol industry is not in its infancy

anymore. It is able to stand without any government aid and had time for R&D towards co-product and energy saving research.

Table 2.1: Renewable Fuel Standard (billion gallons)- Usage Volumes [10]

Year	RFS1 biofuel mandate in EPA Act of 2005	RFS2 biofuel mandate					
		Total renewable fuels	Cap on corn starch- derived ethanol	Portion to be from advanced biofuels			
				Total non- corn starch	Cellulosic	Biodiesel	Other
2006	4.0	—	—	—	—	—	—
2007	4.7	—	—	—	—	—	—
2008	5.4	9.00	9.0	0.00	0.00	0.00	0.00
2009	6.1	11.10	10.5	0.60	0.00	0.00	0.10
2010	6.8	12.95	12.0	0.95	0.0065 <sup>a</sup>	1.15 <sup>b</sup>	0.20
2011	7.4	13.95	12.6	1.35	0.0066 <sup>c</sup>	0.80	0.30
2012	7.5	15.20	13.2	2.00	0.00865 <sup>d</sup>	1.00	0.50
2013	7.6 (est.)	16.55	13.8	2.75	1.00	1.28 <sup>e</sup>	0.75
2014	7.7 (est.)	18.15	14.4	3.75	1.75	f	1.00
2015	7.8 (est.)	20.50	15.0	5.50	3.00	f	1.50
2016	7.9 (est.)	22.25	15.0	7.25	4.25	f	2.00
2017	8.1 (est.)	24.00	15.0	9.00	5.50	f	2.50
2018	8.2 (est.)	26.00	15.0	11.00	7.00	f	3.00
2019	8.3 (est.)	28.00	15.0	13.00	8.50	f	3.50
2020	8.4 (est.)	30.00	15.0	15.00	10.50	f	3.50
2021	8.5 (est.)	33.00	15.0	18.00	13.50	f	3.50
2022	8.6 (est.)	36.00	15.0	21.00	16.00	f	4.00
2023	—	g	g	g	g	g	g

### Dry-grind corn ethanol unit processes

Currently in the United States, the dry-grind corn ethanol process is most prevalent. As of January 2012, the industry has 209 ethanol plants with over 70 percent of ethanol production coming from dry mill plants [1,21]. Average production levels include 2.8 gallons of ethanol and 18 pounds of distillers grains and 18 pounds of CO<sub>2</sub> from every 56 pound bushel of corn [22]. Two thirds of corn is carbohydrates,

which are converted to fermentable sugars and ultimately to ethanol and CO<sub>2</sub>. The residual corn is processed into distillers grains; it contains protein, fibers, oils and leftover yeast cells.

### *Milling*

Corn kernels are milled without separating various components of the kernel (starch, protein, fat, fiber). Hammer mills grind the corn to a fine, grain meal consistency. This process allows efficient processing of starch in subsequent steps.

### *Slurry*

Milled corn is mixed with a combination of fresh and recycled process water, which is called thin stillage, at varying ratios. Ethanol plants can utilize up to 50% of thin stillage for this slurry [23]. Increasing concentrations of acetic acid, lactic acid and glycerol introduced from thin stillage limits its use. A heat stable enzyme ( $\alpha$ -amylase) is added to convert starch to dextrose [24,25]. Additionally ammonia is added to adjust the pH and as a nitrogen source.

### *Liquefaction*

Jet-cookers inject steam into the corn slurry, which increases the temperature to around 100°C. High temperatures and mechanical shearing from the steam process hydrolyzes starch granules present in the corn endosperm while enzymes break down starch polymers into smaller chains [24,25]. Additionally, the heating process

inactivates bacteria that would inhibit yeast fermentation. After cooking, the slurry is cooled and additional  $\alpha$ -amylase is added. The slurry is allowed to cool down to 30°C.

### *Saccharification*

Another enzyme (glucoamylase) is added to complete the breakdown of starch to glucose. This step is typically performed as the slurry is pumped into the fermenters [24,25].

### *Fermentation*

As the liquid slurry is pumped into fermentation tanks, yeast is propagated in a separate reactor. Yeast is added to convert sugar into ethanol with other contents remaining unchanged. This batch process typically runs for 48 h with multiple fermentation tanks to ensure a tank is always filling while another is emptying its contents. During fermentation, CO<sub>2</sub> is sometimes captured, compressed and sold for various industrial applications, although supply greatly exceeds demand. The finished product (beer) is sent to distillation columns for further processing.

### *Distillation and further processing*

The beer from fermentation typically contains 8-12% ethanol by volume [24,25]. Distillation utilizes boiling points to separate ethanol from the liquid slurry. Distillation columns heat the beer, evaporate ethanol and recondense ethanol (92-95%). Molecular sieves selectively remove water by adsorption to >99% [24,25]. This ethanol product



(~200 proof) is blended with a denaturing agent, typically gasoline, to render it undrinkable and not subject to alcohol excise tax [25].

After distillation, residual corn solids and water (whole stillage) remains and is processed further. Whole stillage is passed through centrifuges to separate solids from the remaining liquid. These solids, thick stillage, can be sold as wet distillers grains or processed to distillers dried grains. The liquid stream from centrifugation (thin stillage) contains solubles and some residual suspended solids. Thin stillage can be backset to fermentation to recycle water. Up to 50% of thin stillage can be backset to fermentation [23]. Additionally, thin stillage can be run through flash evaporators to obtain condensed distillers solubles (CDS) or syrup. Condensed distillers solubles (~30% solids content) can be added to distillers grains to create distillers dried grains with solubles (DDGS) after drying. Other variations of distillers grains can be produced and are categorized by moisture content and presence/ absence of solubles.

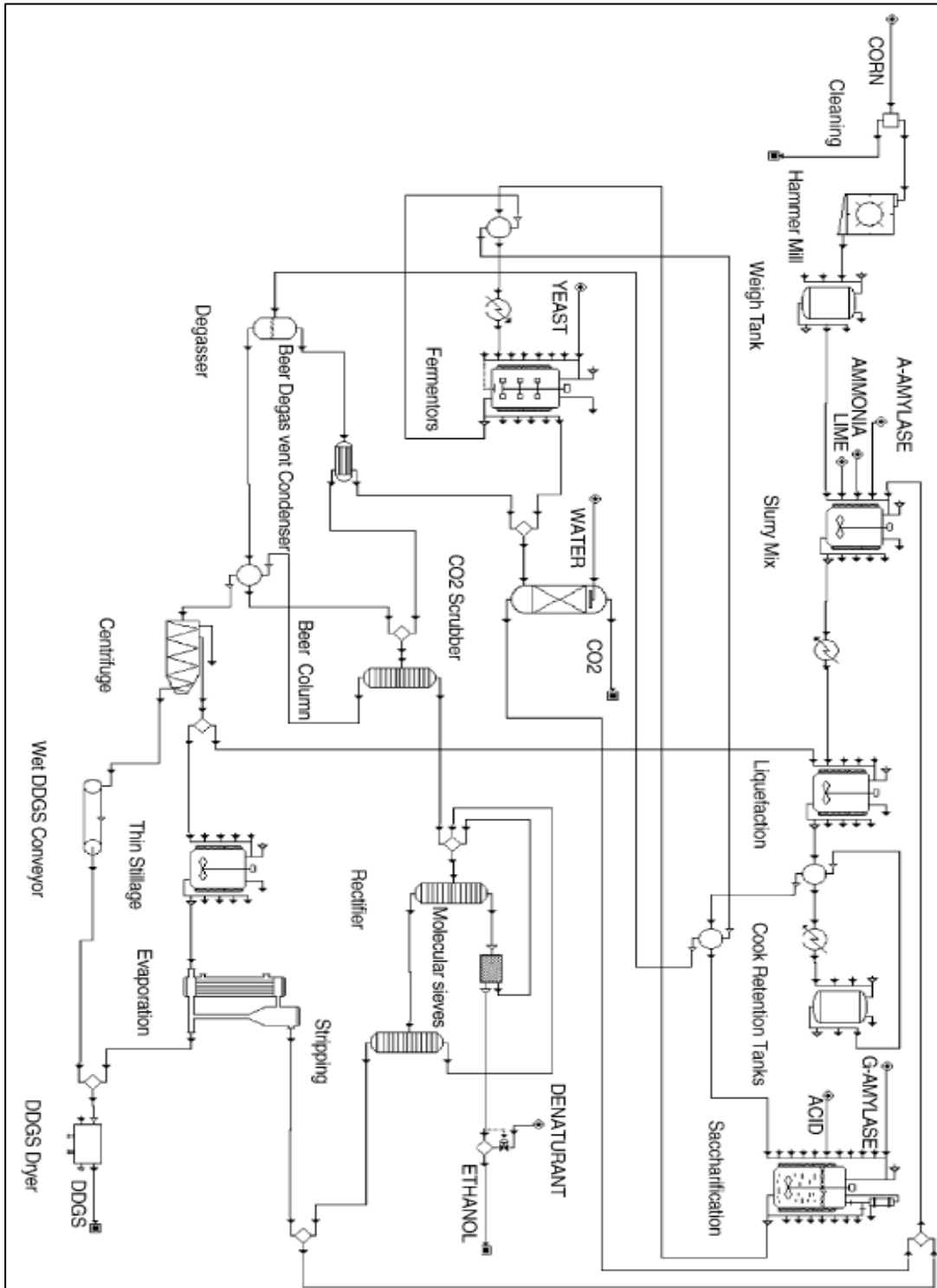


Figure 2.9: Dry-grind ethanol plant flow diagram [25]

### Distillers grains

The dry-grind ethanol production process is characterized by little or no fractionation of the corn kernel. Fiber, oil, protein and other components left after fermentation makes up whole stillage. This whole stillage is centrifuged to a liquid fraction (thin stillage, TS) and solid fraction (wet distillers grains, WDG).

Various products can be produced from the solid residue after centrifugation. Wet distillers grains (30-35% dry matter) are the initial residues left after separating whole stillage [26]. This co-product can be sold as-is with many benefits. Since the product does not require any additional processing, it is cheaper to produce. Also, WDG have higher energy concentrations; drying leads to Maillard reactions between carbohydrates and proteins that decrease digestibility. However, high moisture content limits shelf life to about 3 days, so the product must be sold to feedlots within a close proximity of the ethanol plant [26]. Lastly, high moisture content increases bulk weight that increases transportation and storage costs.

Drying of WDG to a higher solid content (45-50% dry matter) produces a more stable product, modified distillers grains (MDG). This additional processing increases shelf life to 3 weeks and opens sales up to regional markets [26]. Additionally, distillers grains can be dried to produce distillers dried grains (88-90% dry matter). This products shelf life is almost indefinite and can be sold to markets around the world.

Thin stillage, the liquid fraction after centrifuging whole stillage, is processed by flash-evaporation. This evaporation step condenses solubles and remaining particulates to condensed distillers solubles (35-40% dry matter). Condensed distillers

solubles can be added to all other distillers grains to produce a product with nutritional benefits [27]. Additionally, the solubles can be condensed further to distillers dried solubles (88-90% dry matter). The list of these products includes wet distillers grains with solubles, modified distillers grains with solubles (MDGS) and dried distillers grains with solubles (DDGS). Production and sale of these co-products is determined by various factors.

### *Nutritional information*

Distillers grains have become an integral part to many animal diets. Nutritional content of distillers grains is listed below in Tables 2.2 and 2.3.

Table 2.2: Distillers grain nutritional information [28]

Nutrients	Dry Matter	Crude Protein	Ether Extract	Crude Fiber	Calcium	Total Phosphorus	Ash
	- Percent -						
DDG	92	27	9	13	0.09	0.41	2.2
DDGS	92	27	9	8.5	0.14	0.89	4.5
CDS	92	27	9	4.0	0.35	1.3	8.2

\*DDG: distillers dried grains, DDGS: distillers dried grains with solubles, CDS: condensed distillers solubles

Table 2.3: Distillers grain nutritional information (continued) [28]

Nutrients	Ruminant Digestible Protein	Ruminant TDN	Methionine	Lysine	Poultry ME6	Swine ME6
	- Percent -				- Kcal / kg -	
DDG	19.3	79	0.45	0.9	2,000	3,200
DDGS	21.1	82	0.51	0.8	2,800	3,300
CDS	22.8	78	0.60	0.9	2,810	3,300

The nutritional value of distillers grains makes it advantageous for animal feed diets. However, the composition and digestibility of this feed product makes it better for certain animal diets or varying fractions of the diet. DDGS consumption varies with beef cattle (65.4%), dairy cattle (14.6%), swine (8.2%) and poultry (11.8%) of total US consumption [27]. Additionally, product quality varies with processing. Digestibility of amino acids in DDGS varies between ethanol facilities, due to varying drying techniques. Drying process (e.g. rotary kiln, steam tube, microwave) operate at different drying temperatures at times. Drying hotspots can result in heat damage. Maillard reactions (e.g. epsilon amino group on lysine reacting with reducing sugars), a product of heat damage, can decrease digestibility of amino acids [29]. These amino acids are still present in DDGS, however they are not available upon digestion.

### Conclusion

The US ethanol industry has made great strides in the past four decades with help from the government and a global shift in the energy markets. As time passes, ethanol will continue to be a part of a dynamic and diverse energy portfolio. Technological advancements will continue to make the production of ethanol more efficient and affordable. Co-product development must be pursued to keep the industry thriving. New co-products can create additional revenue streams, lowering ethanol costs. More specifically, certain co-products may have the ability to displace greater percentages of corn for feed as the industry consumes record volumes of the United State's largest agricultural commodity.

## 2. Solid-Liquid Separation

Separation of solids from liquid slurry is a challenging task in many industrial processes. The wastewater, drinking water, pharmaceutical, chemical, food, agriculture, and mining industry all have processes involving solid-liquid separation technology. All variations of solid-liquid separation technologies utilize one of two distinct processes, i.e. density or size based separation. Density differences allow materials to separate by gravity, flotation or added gravitational forces. Filtration traps solids as liquids pass through a membrane of granular medium, where the solids have to be larger than the liquid molecules. Both of these separation techniques have advantages and disadvantages. Understanding of the product or prior testing can help determine which method is correct for a particular process.

Liquid-solid separation technology and methodology has been of much importance for decades. Publicly owned waste treatment plants (POTW's) set up for the treatment of municipal and industrial wastewater generate huge quantities of solids from a variety of separation techniques. In addition to biosolids, large quantities of inorganic solids are removed from water and wastewater treatment plants all over the world, requiring effective solid-liquid separation as disposal of the solids is typically quite costly.

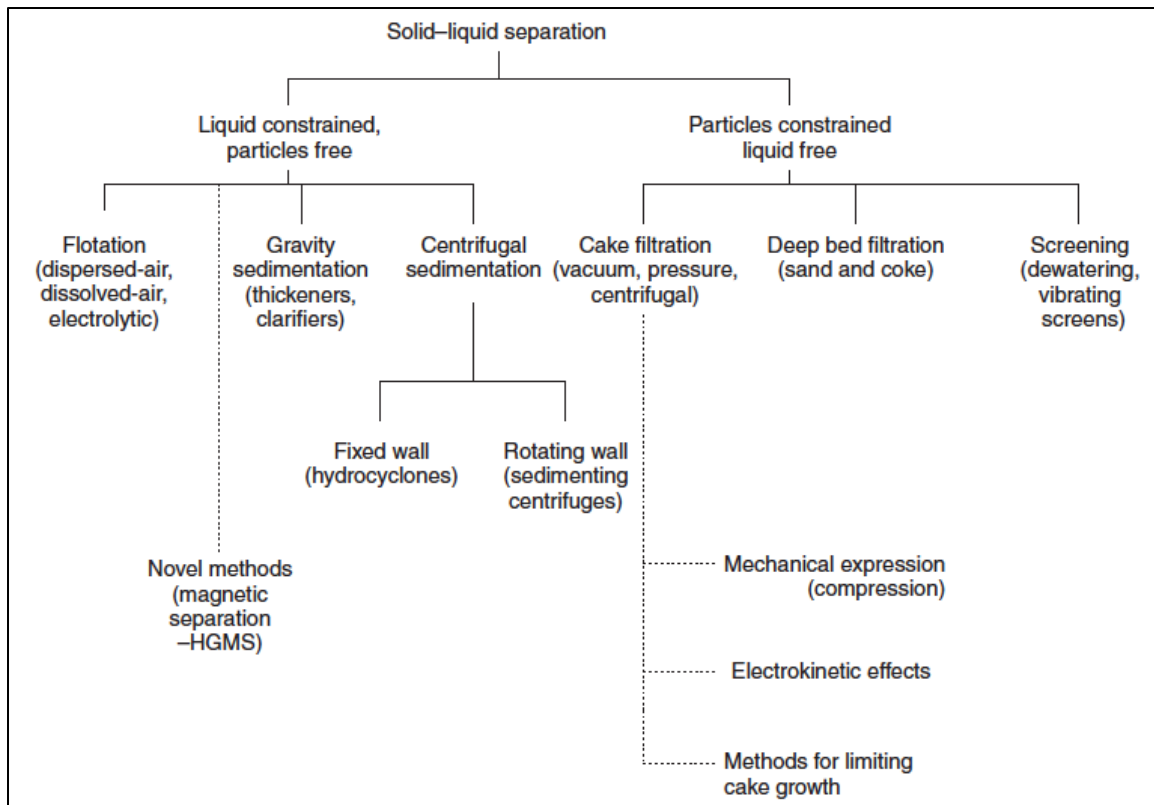


Figure 2.10: Solid-liquid separation flow diagram [30]

## Filtration

Filtration uses one of four driving forces to separate solids and liquids. They include gravity, vacuum, pressure or centrifugal forces. There are different filtration methods that utilize these forces. Additionally, various filtration media are used for separation. They include cartridge, rigid porous media, non-woven media, woven wire, woven fabric filters [30] and granular media. The choice of filter material is made from the various physical, chemical and operating parameters as well as cost efficiency and footprint.

### *Screening*

An influent stream introduced to a screen with a given aperture size can act as a filter. The majority of screens are used for size grading but dewatering applications are common in various industries. Solids have the ability to pass through or be retained on the screen material if the aperture size is smaller than the particle size. Screen designs include the DSM gravity-fed screen. This stationary screen is curved with a wedge wire deck or woven wire cloth. Feed suspension is fed along the top and allowed to dewater by gravity as it is washed and pushed along the screen [30].

Bar screening is the first separation step at a wastewater treatment plant. The coarse screens remove large debris from influent water that may damage process equipment, reduce treatment effectiveness or contaminate waterways [31]. Additionally, fine screens may be used to protect process equipment and eliminate materials inhibitory to biosolids reuse. Coarse screens can be classified by design of parallel bars, rods or wires, grating, wire mesh or perforated plates. Openings are typically circular or rectangular and have openings between 6 to 150 mm. Fine screens are typically constructed of perforated plates, wedge-wire elements or wire cloth with openings less than 6 mm across [31].

Coarse screens in wastewater treatment can be broken down into chain-driven, reciprocating rakes, catenary and belt designs. All designs have advantages and disadvantages. Selection of the proper equipment can be determined by the solids loading and concentrations, plant size, need for manual operation, capital and operations costs associated with the equipment. Fine screen equipment includes static,



rotary drum and step type screens. These screen applications include primary treatment in replace of primary clarifiers, improved solids removal before primary treatment or additional solids removal after primary treatment. However, increased head loss through fine screens may limit uses [31].

### *Deep bed filtration*

Water treatment is one process that utilizes deep-bed granular filtration for solid-liquid removal. Sand filters or dual-media filters are utilized to capture solids in water treatment. Filter beds typically consist of silica-sand, anthracite coal, garnet or other granular materials, which make up the filter media. Pretreated water is passed through these filter beds at 5-30 m/h. Typical downward flow through the bed operates under gravity and performance is rated by effluent quality and head loss through the filter [32].

Solid particles get trapped within the filter media. As more liquid passes through the filter, trapping more solid particles, head loss increases and the filter clogs. As operation continues, filtrate quality will deteriorate and indicate a need for cleaning. Lastly, a filter run may have a maximum run time. These three cases indicate the need for periodic washing of the filter. Backwashing runs clean water, backwards through the filter to removes solids from the filter bed. Air is often used to increase turbulence during backwashing while saving on backwash water needs.

### *Cake filtration - pressure*

Cake filtration can be broken down based on the driving force utilized. They include vacuum, pressure and centrifugal forces. A cake builds up on the up-stream side of the initial filter media. The filter cake turns into a filter, functioning as the filter medium. Pressure filtration utilizes hydraulic or mechanical pressure to direct liquid through a membrane while maintaining solids on or above the membrane. Plate in frame filter is a batch operation, pressure filter that utilizes a sequence of plates with supported hollow frames. Plates are covered with filter cloth, which forms the septum and sealing gasket. Liquid slurry is pumped into the frames and retained while liquid filtrate is drained from the plates [30].

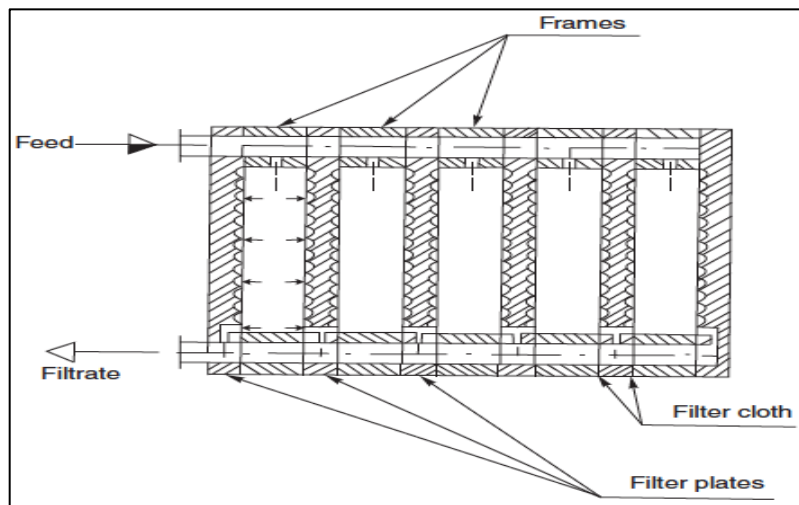


Figure 2.11: Plate-in-frame filter diagram [30]

Pressure vessel filters utilize leaves that form filtration surfaces. The filtration leaf can be set up horizontally or vertically. The slurry is pumped onto the filter leaves, pressurized, and dewatered to form a compressible filter cake. These filters include

cylindrical element filters, vertical-tank and leaf filters, horizontal-vessel and vertical-leaf filters, and other combinations [30].

Compression filters utilize mechanical forces to force filtrate through the filter. As opposed to typical cake filtration where hydraulic pressure in a fixed volume chamber provides necessary forces for filtration, compression filters reduce the volume of the liquid chamber. This method is advantageous to slurries with compressible cakes. A typical cake formation has a pressure gradient. The use of mechanical forces distributes pressures evenly to produce a drier cake. This type of cake filtration can be broken down into four categories including membrane plate presses, tube presses, belt presses and screw presses [30].

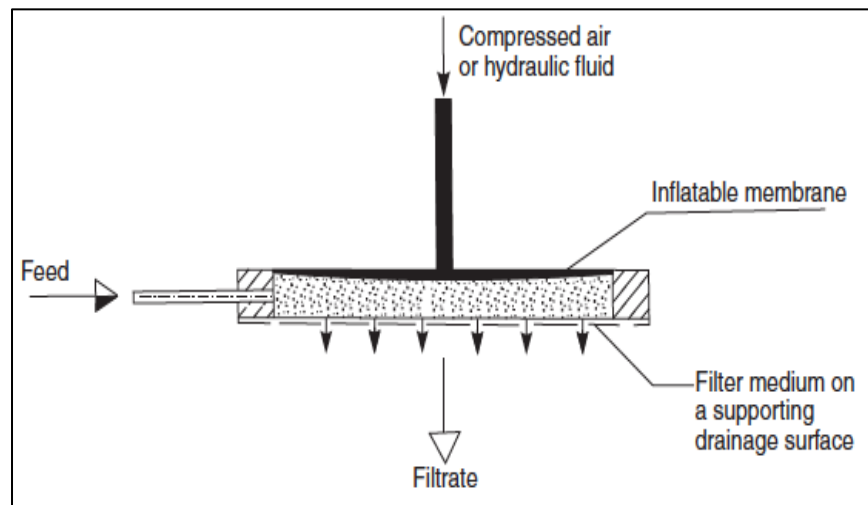


Figure 2.12: Pressure filter diagram [30]

A belt press is another pressure filter that presses two porous cloths together to provide pressure. A cake forms as liquid passes through the porous cloth. Higher pressures are introduced as the belts pass through a series of rollers. The majority of

belt presses are utilized in wastewater treatment for sludge thickening. Gravity drainage typically starts the process to remove free water with the addition of vacuum pressure to remove additional water and odors. Pressure is applied and scraper blades remove the final cake.

Unlike deep-bed sand filters utilized for water treatment, membrane filtration utilizes hydraulic pressure for water treatment and other applications. Microfiltration (MF) and ultrafiltration (UF) membranes have the largest pore sizes of typical membrane filters. MF membranes can remove particles in different size ranges, but greater than 0.2 micron and UF membranes can remove organic substances with a molecular weight greater than 10,000 Da, colloids and some viruses [33]. These membranes are configured in one of three ways:

- 1) Inside to outside flow with positive pressure,
- 2) Outside to inside flow with positive pressure and
- 3) Outside to inside flow with negative pressure.

MF and UF membranes have the ability to run in a dead end mode of operation, where the contaminants are retained on the entire filter surface. These retained contaminants form a filter cake, which causes membrane fouling and decreased productivity. Cross-flow design utilizes only a portion of the filter capacity available for filtration. This may seem wasteful, but the tangential flow lowers fouling potential due to the shear forces on the filter cake [32].

Nano-filtration (NF) and reverse osmosis (RO) run on similar principles to UF and MF but have smaller pore sizes and different filter configurations. Typical configurations include spiral wound and hollow fine fiber membranes [32]. NF has the ability to remove organics with molecular weights greater than 400 Da. The membranes can also remove hardness ions much as anything MF and UF membranes remove. This includes viruses, bacteria and many other substances greater than 0.01 microns. RO is able to remove particles as small as salt ions [33].

#### *Cake Filtration – Centrifugation*

A centrifuge filter incorporates two processes into a single unit. The process is similar to gravity filtration, which utilizes the hydrostatic head of liquid as the driving force for filtration. However, the centripetal forces placed on the liquid increases the driving force for filtration. In the process, filtrate is driven through the filter media by a force of 100 to 2500 times that of gravity [34]. With the range in g-forces and physical characteristics of the solid phase, solid compaction is variable.

A filter centrifuge is constructed with a perforated or slotted wall basket. The basket is covered with filter medium, restricting passage of solids and allowing the liquid phase to pass through freely [34]. Two main centrifuge filters exist, a fixed-bed design and moving-bed design. Fixed-bed designs hold solids on the filter until discharge, in a batch system. Moving-bed designs allow solids to move across the filter medium continuously until they reach the discharge point, a continuous process [34]. Fixed bed designs can be separated into two subcategories, manual or automatic

discharge. The fixed-bed centrifuge filters include basket, inverted basket and peeler centrifuges. Moving-bed centrifuge filters include the directed-flow cone screen centrifuge, vibrating centrifuge and tumbling centrifuge [34]. All of these devices are designed to move solids to a discharge point under continuous operation.

## Sedimentation

### *Gravity Sedimentation*

Grit removal is another unit process for wastewater treatment to separate solids from the influent wastewater stream utilizing sedimentation. This unit process, typically located after screening and before primary treatment, removes sand, gravel and other inorganic solids with specific gravities substantially higher than organic solids in the water. In addition to improve water quality, grit chambers protect equipment from abrasion and abnormal wear. The process reduces grit deposits in pipelines, tanks and digesters. Horizontal flow grit chambers utilize discrete particle settling (gravity settling), allowing the lightest grit particles to settle to the bottom of the basin before water reaches the outlet. Aerated grit chambers, rectangular in design, introduce air on one side parallel to the liquid flow. This air creates a spiral flow pattern, perpendicular to liquid flow, which increases settling rates. A change in airflow allows plant operators the ability to determine which particles are removed based on settling velocities.

Primary sedimentation is another unit process utilizing gravity to remove solids from the liquid waste stream. This process is particularly involved with the removal of

readily settleable solids. Effective primary sedimentation will remove 50-70 percent of suspended solids (TSS) and 25-40 percent of biological oxygen demand (BOD) from the wastewater [31]. Rectangular settling tanks have a linear flow pattern. Water enters one side, flows while solids settle to the bottom, and exits over weirs on the opposite side of the basin. Tank dimensions are primarily determined by wastewater flow rates and overflow rates. For circular tanks, the flow pattern is radial. Water enters in the center and flows over baffles along the perimeter of the tank. The area, depth and weir length are important to design a system with liquid overflow rates low enough to allow settling of solids. In general, velocity is the most important factor for continuous sedimentation. Downward velocities of the solids being removed must be large enough to overcome the upflow velocity of the waste stream. Secondary sedimentation, clarification, is a similar process to primary sedimentation. However, solids removal is slower because organic solids from biological treatment have less dense. The lighter, organic material requires lower overflow rates and ultimately larger tanks.

#### *Advanced Gravity Sedimentation*

Vortex-type grit chambers utilize centrifugal and gravitational forces to settle grit particles. This compact design is beneficial for facilities with limited space [27]. For this system, the g-forces are produced by the motion of the slurry. The material is introduced tangentially to create gravitational forces, not a mechanical action by the equipment [35]. The thickened material forms on the outer wall and is removed

through a discharge nozzle. Dewatering efficiency is determined by shape of the equipment and feed rate.

The decanter centrifuge is another piece of equipment utilized to mechanically dewater many products including environmental and biosolids sludges [35]. The ethanol industry utilizes centrifuges to separate solids from the residual process stream (whole stillage) after distillation. In this process, the g-forces are created by mechanical action from a rotating bowl. Operating g-forces produced from this rotating bowl typically range from 2000 to 4000 g's. Material enters one end of the machine and the rotating action forces denser particles outward against the rotating bowl. At the same time, less dense liquids form at the inner layer. As more material enters the device a screw conveyer, rotating at a different speed than the bowl, continuously removes solids. Input flow rate, bowl and conveyer speeds all contribute to the efficiency of the process. Advantages to this process include high forces applied directly to the feed, handle ability of high solid liquid slurries and continuous operation [36].

### Coagulation and Flocculation

Typically, various process slurries and wastewater sludges are made up of a stable suspension of solids dispersed throughout the bulk liquid. Applications to remove solids can be successful without additional material. However, in many cases, this suspension of solids is very difficult to remove. Addition of various chemicals may be necessary to help the sludge particles to agglomerate into larger particles or flocs prior to separation [37].



### *Destabilization*

Coagulation is the destabilization of colloids by chemicals that neutralize the electrical charge of the dispersed solids. Before addition, colloids remain in suspension due to the similar, repulsive forces. Colloids, typically organic solids for most wastewater and bioprocessing sludges are negatively charged. Addition of higher valence cationic salts ( $\text{Al}^{3+}$ ,  $\text{Fe}^{3+}$ , etc.) lowers interparticle repulsion [38]. Additionally, polymers may be utilized to destabilize colloids. Localized charges of the long polymer chain allow colloids to adsorb onto oppositely charged surfaces. Typically, coagulants are vigorously mixed into the bulk liquid to ensure complete dispersion.

### *Floc development*

Destabilization of colloids is only the first step to remove solids from the bulk suspension. If dosed correctly, most surface charges have been neutralized, which allow these fine-particle suspensions to adhere to one another [39]. Flocs grow as the particles interact and adhere upon contact. Polymers aid flocculation by bridging multiple floc formations. Mixing is a critical step to good floc formation, which depends on shear forces within a certain range. High mixing speeds produce high shear forces, breaking apart floc formations. In comparison, a low mixing speed doesn't allow adequate mixing for enough interparticle contact.

### *Coagulants*

There are many different coagulants and many properties determine which should be used. Alkalinity is particularly important for metal salt coagulants. Alkalinity is needed to drive the hydrolysis reactions. The pH is another factor that should be determined to select the correct coagulant. Additionally, temperature can be an important factor. Inorganic metal salts that rely on chemical reactors may not work properly at low temperatures ( $<5^{\circ}\text{C}$ ) [40].

Table 2.4: Typical Chemical Coagulants

Chemical Name	Chemical Formula
Aluminum sulfate (Alum)	$\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{H}_2\text{O}$
Ferrous sulfate	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$
Ferric sulfate	$\text{Fe}_2(\text{SO}_4)_3 \cdot 9\text{H}_2\text{O}$
Ferric chloride	$\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$
Calcium hydroxide	$\text{Ca}(\text{OH})_2$
Calcium oxide	$\text{CaO}$
Sodium aluminate	$\text{Na}_2\text{Al}_2\text{O}_4$
Calcium carbonate	$\text{CaCO}_3$

### *Polymers*

Polymers are used in many different industries including mineral processing, papermaking, water/ wastewater treatment and biotechnology [40]. Most polymers are synthetic but some natural polymers, including chitin, have become increasingly common. Most polymers are constructed of linear chains but some branched or cross-linked chains are utilized. Below is a description of various non-ionic, cationic and

anionic polymers. Construction of the polymer types is based on various amides, carboxylic acids and other chemical groups that ionize and produce surface charges.

- Non-ionic: polyethylene oxide (PEO), polyacrylamide (PAM), polyvinyl alcohol (PVA), polyvinyl pyrrolidone (PVP)
- Anionic: hydrolyzed polyacrylamide, polyacrylic acid (PAA), polyvinyl sulfate
- Cationic: poly(dimethylammonium chloride) (PDADMAC), diallyl dimethylammonium chloride (DDMAC), cationic polyacrylamide (CPAM), polyethylene imine (PEI), polyvinyl pyridine (PVP)

### Sludge disposal

Land application of biomass sludges, including wastewater treatment biosolids and bioprocessing sludges, have become increasingly popular due to large concentrations of nutrients (nitrogen and phosphorus). Currently, around 50% of all wastewater biosolids are recycled to agricultural land. This makes up one percent of the United States' agricultural land [35]. The Environmental Protection Agency works to regulate land application of biosolids. Rules for biosolids are located in the US Code of Federal Regulations, Title 40 (protection of environment), and part 503. This rule regulates various items including pathogens and metal addition. Besides land application, other sludges can be used for soil amendment, sent to landfills or incinerated.

## Conclusion

Over the past 100 years, numerous researchers have developed methods and equipment for solid-liquid separation. Mechanisms, operating conditions and various other parameters separate the equipment types from one another. Understanding of these mechanisms will help in the selection of the proper equipment for a specific product. This review illustrates methods of solid-liquid separation that can be applied to a variety of applications.

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## **Chapter 3. A Pilot Study on Dewatering Fungal Biomass From a Novel Corn Ethanol Bioprocess**

A paper prepared for submission to Bioresource Technology  
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### **Abstract**

An innovative bioprocess utilizing thin stillage from dry-grind corn ethanol plants was used to produce a valuable filamentous fungus (*R. microsporus* var. *oligosporus*). A 1,500-L bioreactor was built to optimize operating parameters, produce fungal biomass for animal feed trials and develop equipment and processes for industrialization. Dewatering equipment was tested to determine proper solid-liquid separation equipment for the industrial process. Studies indicate that a two-step dewatering process is most effective utilizing a gravity fed, concave screen followed by a centrifuging filter. Solids content greater than 30 percent is not possible, as this value approaches theoretical limits due to intracellular water. Thermal drying, final processing step to obtain a dried feed product, takes the biomass to 90 percent solids. Extensive mechanical dewatering is critical, as higher energy inputs are associated with thermal drying.

This new fungal co-product has some superior feed properties in comparison to distillers grains. It is known to be high in oils and protein. Key nutritional values include 35% protein, 1.4% lysine and 1% methionine on a dry basis. Overall, this innovative fungal process is able to produce a high-value co-product at a dry-grind

ethanol facility, while facilitating higher water recycling rates. Selection of proper processing equipment minimizes capital and operating costs, making this process economically feasible for ethanol producers or other investors.

## **1. Introduction**

The United States ethanol industry has grown in response to various factors including increasing fuel demands and the Renewable Fuel Standard II (RFSII). Historic US corn-ethanol production has increased from 1.77 billion gallons in 2001 to 13.9 billion gallons in 2011, a 685% increase in 10 years [1]. Ethanol, the largest piece of a diverse renewables portfolio, has helped to alleviate oil dependence. As this industry matures and its input costs including corn and energy remain variable, development of new co-products may keep ethanol prices competitive and increase revenues for the industry.

The most common method to produce ethanol from corn is the dry-grind process. Out of the 209 current ethanol plants in the United States, dry-mill plants account for over 82 percent of the production capacity [2]. These plants continue to be the more attractive process for investors due to lower capital costs compared to wet-mill plants. The dry-grind process is characterized by minimal fractionation of the corn kernel before fermentation. Two thirds of the corn kernel is made up of starches, which are converted to ethanol and carbon dioxide and the last third is left as stillage [3]. The residual corn biomass and yeast cells left over after fermentation constitutes the make-up of whole stillage, a crude by-product of the dry-grind process.

Stillage contains fiber, oil, protein and other unfermented components [2]. Whole stillage, the direct by-product of fermentation, is typically processed by centrifugation into two products. These products include the solid fraction (wet distillers grains - WDG) and the liquid fraction (thin stillage - TS). TS can be concentrated in an energy intensive step to produce condensed distillers solubles (CDS), also known as syrup that may be sold as is or added to other distillers grains. The solid fraction can be sold directly as WDG or as wet distillers grains with solubles (WDGS: 30-35% dry matter), modified distillers grains with solubles (MDGS: 45-50% dry matter), distillers dried grains or distillers dried grains with solubles (DDG/DDGS: 88-90% dry matter) [4]. Of all the distillers grains, DDGS has the largest market share. Approximately 64 percent of distillers grains with solubles were fed dry in the United States, compared to 36 percent of wet distillers grains [4]. Distillers grains have always been the largest co-products from dry-grind ethanol plants.

Thin stillage, the centrate produced from the whole stillage separation process, is currently used as liquid backset for fermentation and its solids as an additive to distillers grains after an energy intensive evaporation process. The fraction of thin stillage, recyclable to the fermentation process, is limited by its lactic acid, acetic acid and glycerol concentrations. These fermentation byproducts build up in ethanol fermentation tanks, inhibiting ethanol production [5]. Also, the large energy input necessary to concentrate thin stillage by flash-evaporation into CDS is not advantageous for ethanol plants because of its relatively low market value. A treatment process

capable of utilizing these organic compounds in thin stillage, while producing a higher value co-product would be very beneficial to the ethanol industry.

Development of a new fungal treatment process at Iowa State University has led to advances in water reclamation of thin stillage and production of a new animal feed co-product, MycoMeal, derived from the cultivation of the fungus *R. oligosporus* on thin stillage. Utilization of many organic compounds, lactic acid, acetic acid, glycerol and others, during the fungal treatment process increases the volume of backset thin stillage to fermentation, which decreases fresh water usage. Another benefit of this fungal process is the production of an animal feed with superior feed properties. Large concentrations of the essential amino acids lysine and methionine, compared to corn and DDGS, make this product an ideal candidate for swine and poultry nutrition. This highly nutritional animal feed allows ethanol facilities the ability to sell the product to many animal-feed markets, including swine and poultry production facilities. Creation of additional revenue, while diversifying a co-product portfolio, should make this process advantageous to any dry-grind ethanol plant.

The fungal process has progressed with investigations on various aspects of the process. Currently a 1,500-L pilot-scale plant is running to understand challenges in industrial-scale production. The goal of this study was to determine the best way to process the filamentous fungus, *Rhizopus oligosporus*, and utilize the information for industrial scale-up. Processing of the material to an acceptable 85 to 90 percent dry matter requires dewatering and drying equipment. This study utilized small-scale lab

tests and various pilot tests to develop the best method to remove free water from the fungal biomass.

## **2. Methods and Materials**

### 2.1 Fungal cultivation

#### *Seed culture*

The fungal seed culture for the pilot bioreactor cultivation and harvest was carried out in a two-step process. First, *Rhizopus microsporus var. oligosporus* was obtained, grown and stored according to Ozsoy et al. [6]. Next, eight 2-L Erlenmeyer flasks were prepared with 1 L of yeast malt (YM) broth per flask. The YM broth pH was adjusted to 4.0 with hydrochloric acid, capped with filter paper, autoclaved at 121°C for 15 minutes and cooled to room temperature. One 2-ml vial of spore suspension was added to each flask in a bio-hood under aseptic conditions. This seed culture was incubated in a shaker at 180 rpm, 37°C for 24 h.

#### *Thin stillage*

Thin stillage, substrate for fungal growth, was obtained from a local dry-grind ethanol plant, Lincolnway Energy (Nevada, IA). Properties of thin stillage from Lincolnway Energy include solids concentrations (total solids = 7%, dissolved solids = 4%) and a chemical oxygen demand (COD) of 100 g/L. Additionally, temperature of receiving thin stillage measured 80-85°C.

A 1000-L liquid storage tote, disinfected with steam and hypochlorite solution (500 ppm), was used to transport thin stillage from Lincolnway Energy to the pilot plant at BECON (Biomass Energy Conversion Center - Iowa Energy Center). An FDA approved, 1" ID smooth bore, hose was autoclaved prior to operation. This hose was utilized to fill the storage tote. While obtaining thin stillage at the ethanol plant, aseptic techniques were applied to minimize risk of contamination from unwanted microbes. These techniques included wearing nitrile gloves and wiping them with 70% ethanol prior to connecting the hose or operating valves. Also, the ethanol plant's and storage tote's hose adapters were disinfected with 70% ethanol prior to connecting the autoclaved hose. At BECON, the same aseptic techniques were utilized while filling thin stillage into the bioreactor. One additional step of attaching an air filter to the top the storage tote prevented airborne microbes from contaminating the thin stillage while being pumped into the bioreactor.

#### *Pilot bioreactor cultivation*

The pilot bioreactor, nominal working volume of 1500 L, was utilized for the large-scale cultivation of *R. oligosporus*. Pumping in 1500 L of the substrate, thin stillage, was the first step of the process. Aeration, introduced through fine-bubble ceramic diffusers, was maintained between 0.1 and 0.2 v/v/min to provide dissolved oxygen and mixing. Temperature was adjusted with an externally looped heat exchanger. Once the thin stillage reached 37°C, the bioreactor was inoculated with the seed culture and carried out for 48 h under batch mode of operation.

## 2.2 Analytical procedures

### *Moisture content*

During most dewatering experiments, moisture content of the fungal matrix was critical to determine dewatering effectiveness. Samples of the dewatered fungal biomass were analyzed for moisture content according to Mitra et al. [7].

### *Solids analysis*

Total solids, total suspended solids and total dissolved solids were determined by the procedures in Standard Methods for the Examination of Water and Wastewater [8]. Also, screened solids were calculated by passing material over a screen (20 mesh, 0.841 mm pore size). Biomass retained on the screen was washed with deionized water to remove solids held in free liquid for purposes of determining actual fungal biomass yield.

## 2.3 Biomass dewatering

Samples of cultivated fungal biomass suspended in residual thin stillage from the pilot bioreactor after a 48-h retention period was used to investigate solid-liquid separation techniques.

### 2.3.1 Gravity sedimentation

#### *Sludge volume index*

Common in wastewater treatment, the sludge volume index (SVI) is utilized to assess the settling characteristics of sludges. It was used to assess the settling characteristics of this particular fungal suspension. The SVI method in Standard Methods for the Examination of Water and Wastewater [8] defines SVI by Eq. (3.1):

$$SVI = \frac{SV_{30}}{X} \quad (3.1)$$

as the volume of sludge occupied by one gram of suspended solids (mL/g),  $SV_{30}$  is the volume of settled sludge after 30 minutes in a 1-L graduated cylinder (mL/L), and  $X$  is the suspended solids concentration of the sludge being tested (g/L).

Dilute sludge volume index (DSVI), a test used to determine settling characteristics of sludges with high solids concentrations, beyond the hindered settling phenomenon [9]. In this procedure, a series of twofold dilutions ( $n$ ) were prepared using the fungal suspension and water. After dilution, all samples were placed into 1-L graduated cylinders and mixed to uniformly distribute the fungal biomass. The fungal suspension was settled for 30 minutes to calculate the settled sludge volume ( $SV_{30}$ , mL/L) [10]. Suspended solids concentration of the fungal suspension ( $X$ , g/L) was calculated, utilizing the procedure in Methods for the Examination of Water and Wastewater [10]. The DSVI was calculated from Eq. (3.2).

$$DSVI = \frac{SV_{30}2^n}{X} \quad (3.2)$$



### *Centrifugation*

A lab centrifuge was utilized to determine the solid-liquid separation potential of the fungal suspension at various g-forces. Centrifuge bottles were filled with 250 ml of fungal suspension, placed in the centrifuge and run (2000-5000 g's) for 10 minutes. After running fungal samples in the centrifuge, sludge volumes were calculated as the percent of settled fungal biomass. Next, centrate liquid was removed and the moisture content of this fungal biomass was analyzed.

In addition to lab tests, 200 L of fungal suspension was run through a pilot-scale horizontal decanter centrifuge (Centrisys, Kenosha, WI, USA). During the investigation, the centrifuge speed was adjusted up to its upper limit (3500 g's) while adjusting the screw conveyer speed.

### 2.3.2 Filtration

#### *Specific resistance to filtration*

Specific resistance to filtration (SRF), a filtration property, provides insight into the dewaterability of solid-liquid slurries. SRF was introduced in 1956 by Coackley and Jones, part of a vacuum filtration model [11]. However, since its inception, this test has been extensively utilized as a general model for sludge dewaterability in the wastewater industry. Specific resistance to filtration results for the fungal biomass has been utilized to set a general benchmark against other sludges.

For this test, a Buchner filter was attached to a modified graduated cylinder with a pump to create vacuum pressure. Whatman, grade-4 disk filter (Whatman, Maidstone,

UK) was used; properties include 11 cm diameter, 20-25  $\mu\text{m}$  pore size and  $9.5 \times 10^{-3} \text{ m}^2$  effective filtration area. A 200 ml fungal suspension was filtered with 30 kPa of transmembrane pressure. Cumulative volume of filtrate was plotted vs. time. The plot, time/cumulative volume of filtrate (y-axis) and cumulative volume of filtrate (x-axis), was created and the slope value was used to calculate SRF.

After collecting data from filtration, cake solid concentration (mass of cake per unit volume of filtrate) was obtained. Overall, all of these steps were needed to calculate  $r$ , the specific resistance to filtration ( $\text{m/kg}$ ) by Eq. (3.3):

$$r = \frac{2\Delta P A^2 b}{\mu w} \quad (3.3)$$

where  $\Delta P$  is the transmembrane pressure (Pa),  $A$  is the filter area ( $\text{m}^2$ ),  $b$  is the slope of time/cumulative volume vs. cumulative volume ( $\text{s/m}^6$ ),  $\mu$  is dynamic viscosity of filtrate ( $\text{kg/m s}$ ) and  $w$  is mass of cake per unit volume filtrate ( $\text{kg/m}^3$ ).

### *Belt Press*

A screened sample of fungal biomass (10% suspended solids) was run through the belt press. Second, a sample of fungal biomass (3.1% suspended solids) from the bioreactor was mixed with a cationic polymer (200 mg/L) and run through belt press. Observations were noted and biomass samples were taken for moisture content analysis.

### *Filter press*

A Bench top pressure filter with a 3-inch diameter filter apparatus was utilized for the experiment. The fungal suspension was placed into the sample reservoir and pressurized, utilizing compressed air. Initial feed pressure of 25 psig was increased to 100 and 225 psig. The filtration cycle ended when filtrate flow reached 0.01 gallon per minute per ft<sup>2</sup> of filtration area. Cake air blow down was performed prior to discharge of the filter cake.

### *Simple screening*

Screening, with gravity drainage, was utilized as a method to remove free liquid from the fungal suspension. A coarse metal mesh screen (20 mesh, 0.841 mm pore size) seen in Fig. 3.1 was used as the filter media with dimensions of 55-cm wide by 120-cm long.



Figure 3.1: Metal mesh screen

The volume of screen filtrate and screened fungal biomass were calculated to perform a mass balance on all bioreactor materials. Screen filtrate was collected underneath screen in a sump and discharged through a mechanical flow meter. The

volume (L) and mass (kg) of screened fungal biomass was measured. Design of the screen is shown in Fig. 3.2.

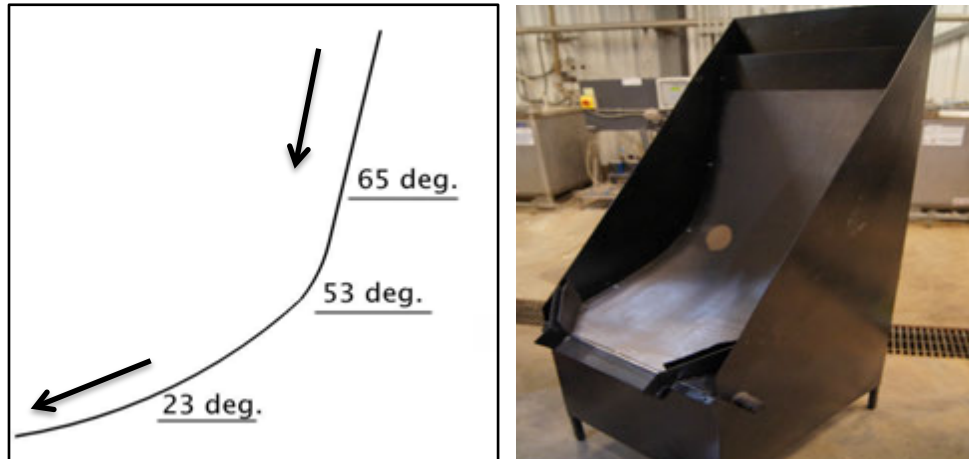


Figure 3.2: Simple gravity screen and angles

### *Filter centrifuge*

A domestic washing machine was utilized as a centrifuge filter. Fungal biomass was placed into woven polypropylene bags. These bags acted as the filter medium, while spinning in the washer's drum, during the spin cycle, provided centripetal forces found in a centrifuge filter. The machine's drum radius ( $r$ ) is 26 cm and spins at 800 rpm. The g-force produced by the equipment is calculated by Eq. (3.5):

$$\text{RCF} = \frac{4\pi^2 r}{T^2 g} \quad (3.5)$$

with additional units: gravitational acceleration ( $g, \text{m/s}^2$ ) and frequency ( $T, \text{s/rev}$ ).

Fungal biomass was placed into two woven bags to balance the centrifuge. Variables for the experiments include mass of biomass (kg) and filtration time (min). Cake thickness (cm) and cake area ( $\text{m}^2$ ) were recorded. The test demonstrated the

extent of dewatering and cake compression. Like the previous experiment, screened fungal biomass was placed into the centrifuge filter. Samples were taken at five-minute intervals to track moisture and cake thickness (cm).

### 3. Results and Discussion

#### 3.1 Solids Analysis

##### *Thin Stillage*

A solid matrix of thin stillage, presented below in Table 3.1, includes total, suspended, dissolved and screened solids (20 mesh, 0.841 mm pore size).

Table 3.1: Thin stillage solid matrix

Constituent	Concentration (g/L)
Total Solids	74.5
Dissolved Solids	44
Suspended Solids	30.5
Screened Solids	1.2

The initial liquid substrate for fungal growth (thin stillage) had large suspended solid concentrations, approximately 3% (w/v). By comparison, domestic wastewater treatment bioreactors operate at 0.2 to 0.5% (w/v) suspended solids concentration [9]. This order of magnitude higher solids concentration over domestic wastewater treatment systems will prove to limit methods of dewatering fungal biomass. However, dewatering fungal biomass was a selective process. Goals for the study were to separate fungal biomass from the bulk suspension in order to harvest nutrient rich fungal biomass. Total solids removal from the liquid growth medium would increase

operating costs associated with dewatering and decrease product value, as the properties would more closely represent condensed distillers solubles.

### 3.2 Gravity settling

#### *Sludge volume index*

Gravity and increased gravity sedimentation tests were conducted to investigate separation technologies. Sludge volume index (SVI) tests indicate that gravity sedimentation is not a feasible method of solid-liquid separation for the fungal effluent. No settling was achieved in multiple sludge volume index tests. The study demonstrates that this fungal slurry is beyond hindered settling, describing the phenomenon in which interparticle forces are sufficient to hinder the settling of neighboring particles [9]. However, a dilute sludge volume index was determined to investigate the settling characteristics of the biomass at lower concentrations. The fungal effluent had a DSVI of 52 mL/g. This value indicates that sedimentation is feasible if not inhibited by high solid concentrations. The specific gravity of the fungal biomass is 1.03, not unusual for microbial biomass. The small density differential results in low settling velocities, another reason gravity sedimentation is not feasible.

#### *Centrifugation*

Operation of the pilot-scale horizontal decanter centrifuge, operated at 1000 to 3500 g's, was unsuccessful in separating fungal biomass from the bulk liquid. At 3,500 g's, the maximum acceleration for this particular industrial centrifuge, observations indicated breakdown of the filamentous structure of the biomass. High g-forces acting

on the organic material, fungal mycelia, turn it to a gelatinous paste with no fibrous structure. Ultimately sedimentation technologies are not feasible for dewatering this fungal biomass.

### 3.3 Filtration

#### *Specific resistance to filtration*

The results in Table 2.2 illustrate the filterability of this fungal biomass in comparison to water and wastewater sludges. Numerous filtration studies have been conducted on these waste streams, which make this comparison a good benchmark for further investigation.

Table 3.2: Specific resistance to filtration; filterability of fungal biomass (*R. oligosporus*) in comparison to typical wastewater/ water sludges

Material	Specific resistance to filtration (m/kg)
Fungal biomass <sup>1</sup>	$2.0 \times 10^{13}$
Raw waste activated sludge <sup>2</sup>	$4.8 \times 10^{14}$
Anaerobically digested primary & waste activated sludge <sup>2</sup>	$9.3 \times 10^{14}$
Readily dewaterable (water treatment) <sup>2</sup>	$1.0 \times 10^{11}$
Poorly dewaterable (water treatment) <sup>2</sup>	$5.0 \times 10^{13}$

<sup>1</sup> Lab testing at Iowa State University

<sup>2</sup> Typical values [12]

The fungal biomass has less resistance, by an order of magnitude, to filtration than sludge from a wastewater treatment plant, but more than the typical chemical sludges from water treatment. Lower filtration resistance rates in comparison to

wastewater sludges, coupled with ready available filtration equipment used in the water and wastewater industry for sludge dewatering, warranted further investigation.

### *Gravity screening*

Table 3.3 presents a mass balance for bioreactor effluent, screen filtrate, screen solids and biomass retained on the screen. Fungal solids make up one percent of the reactor content. An order of magnitude reduction in volume from reactor effluent to gravity screen effluent was achieved.

Table 3.3: Gravity screen mass balance

Constituent	Total Volume (L)	Solids Concentration (% w/w)
Bioreactor effluent	1500	1
Screen cake solids	100	15

The pilot-scale curved metal woven screen (1.2 m long, 20 mesh) had a variable slope: top of the screen, 65° from horizontal, curved to 20° from horizontal. Most separation occurred at the beginning, 65° section of screen, as biomass continuously poured down, removing free liquid. As biomass reached the end of the screen, a hand scraper was used to move biomass to a hopper. Observations of filtrate liquid indicate no visible loss of fungal biomass. The filamentous nature of the material creates a matrix from numerous mycelia. This fungal matrix prevents biomass from passing through the relatively coarse screen. Ultimately, biomass is thickened on the screen up to 15% solids.



This value (15% solids) for screening is quite useful, but insufficient before drying and other methodology needed to be investigated. However, this system does not require any power inputs besides a scraping mechanism, minimizing operating costs for the primary and main water removal. No complicated machinery, which minimize capital costs. Overall, this process is a cost-effective way to remove most free liquid from the fungal biomass. With a ten-fold reduction in process volume, size of subsequent dewatering equipment can be significantly reduced. As stated by Sveegaard et al. [13], drainage or low-pressure filtration is often an effective and cheap method to reduce the water content of highly compressible cake sludges [13]. This gravity screening method has proved to be an advantageous method to remove fungal solids from the bioreactor effluent.

#### *Belt filter press*

The initial sample for testing on the belt filter press was dewatered fungal biomass (15% suspended solids) by gravity screening. However, the solid content was too high and could not be fed into the machine. At (3% suspended solids) the sample could be filtered, but only with the addition of a cationic polymer (200 mg/L). This test removed most free liquid from the biomass cake. The cake (20-25% dry matter) varied under different operating conditions. However, these results were achieved with polymer doses (4-8 lbs./dry ton solids).

Belt filter dewatering appeared to be undesirable for two reasons: a) Fungal biomass after initial gravity screening (15% dry matter) was advantageous for

industrial processing, as decreased process volumes would decrease equipment sizes. However, the higher viscosity of the dewatered fungal biomass hindered its even placement onto the belt. b) The addition of any chemical to an animal feed product could prove to be problematic when the product is introduced to the market, in addition to higher operational costs for the polymer.

### *Filter press*

A 3-inch lab filter press was used to assess the ability to dewater this fungal slurry. Tests at different operating conditions show how this fungal slurry is not amenable to pressure filtration. The testing proceeded until the filtrate flow rate went down to 0.01-gallons per minute per square foot filter area. During filtration, a 25-mm thick cake layer was formed and most free liquid was not removed. Also, release of the biomass cake from the filtration unit was a challenging task. Based on observations from this filtration test and specific resistance to filtration tests, thin stillage particulates foul the membrane before adequate dewatering can be achieved. Also, the highly compressible organic solids, fungal and residual thin stillage solids, were compressed under relatively high feed pressures (100 and 225 psig) compared to other dewatering equipment (Table 3.4). This lowered permeability and increased specific resistance to filtration.

Table 3.4: Operating pressures of filtration equipment

Equipment	Operating Pressure (psi)
Low pressure belt filter	58
Medium pressure belt filter	72.5
High pressure belt filter	101.5
Filter press	225
Rotary pressure filter	85

Biological sludges are known to express high compressibility, low permeability and high specific resistance to filtration, at moderate to high pressure [13]. This information provides insight to the failed attempt of the filter press to dewater this fungal slurry. The cake solids compression decreased permeability, which ultimately led to an increase in resistance to filtration and filtration effectiveness.

#### *Filter centrifuge*

This process utilizes an advanced gravitational field, which removes free liquid from the fungal matrix. Various fungal biomass loading rates, at 15 percent solids, were run up to 25 minutes in a centrifuge filter. Fig. 3.3 illustrates the solids content possible from this filtration process. The centrifuge filter was able to increase solids content to 25-30 percent solids from an input solids content of 10-15 percent. Solids content greater than 31 percent was never achieved.

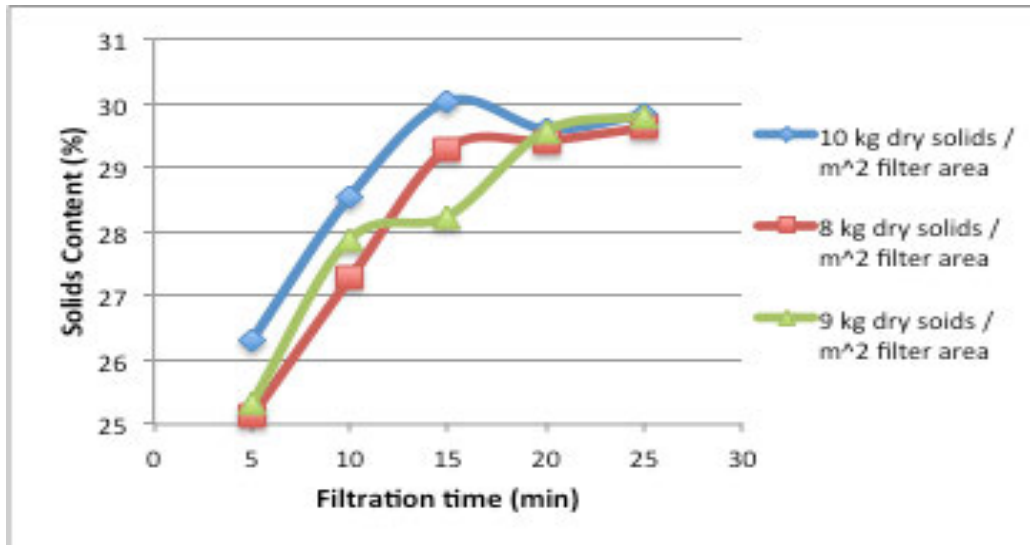


Figure 3.3: Centrifuge filter – fungal dewatering

In addition to solids content, cake thickness was measured to determine the compressibility of the cake in this centripetal field. Screened fungal biomass, 15 percent solids, was input into the centrifuge filter for this study (Table 3.5).

Table 3.5: Centrifuge filter cake compressibility

Fungal Biomass Loading (kg)	Initial Cake Thickness (mm)	Final Cake Thickness (cm)	Change in Cake Thickness (%)
10	38	27	28.9
15	42	30	28.6
20	51	38	25.5

Filter centrifuges provide remarkable results in dewatering this fungal biomass. Utilizing a washing machine as a pilot-scale fixed bed centrifuge filter produced a dewatered fungal biomass, utilizing relatively low g-force (200 g's), in respect to sedimentation centrifuges (1,000 to 20,000 g's) [14]. Additionally, transmembrane

pressure across the filter septum ranged from 10-20 psi as calculated from acceleration forces, mass and filtration area.

A key factor contributing to the success of this equipment is the g-force and pressure induced on the filter cake. The relatively low g-force provided the driving force to remove free liquid from the cake matrix, while its structure remained intact. In other words, the low-pressure filtration minimizes cake compression. The filamentous fungal biomass forms a porous, fibrous structure. As centripetal forces act on this structure, free liquid is filtered out through the filter membrane. Because of minimal cake compression, there are no significant increases in specific resistance to filtration; high solids content can be achieved.

Time plays a critical role in the centrifuge filter efficiency. Based on experimental data, cake solids content plateaus after about 20 minutes. Based on solids loading rates and g-forces, this value may change for industrial equipment. Investigations on industrial equipment should be conducted to maximize efficiency.

#### **4. Conclusion**

Gravity screening coupled with centrifuge filters proved to be efficient and practical processes to remove free liquid from the fungal biomass. The high solids content (25-30% dry matter) of the processed biomass over a short time period makes this equipment an attractive option for industrial application. Filament interactions create a meshed biomass matrix, which allow the use of coarse screens (1mm opening) without fungal biomass loss. The low g-force/ pressure applied to this fungal biomass

matrix limits cake compression, which maintains a lower resistance to filtration for proper drainage of free liquid from the biomass cake. Almost all of the free liquid is removed by this method. Any further mechanical dewatering (>30% dry matter) is not possible without lysing the cells. Ultimately, the remaining liquid will be removed by thermal drying to 85-90% solids but the dewatering process removes most free liquid, which substantially cuts down energy inputs required to evaporate water from fungal biomass.

Collaboration with gravity screen and centrifuge filter manufactures should be initiated. Testing of the material on industrial equipment can be conducted to optimize loading rates and other operating parameters for industrialization of the bioprocess.

## **Acknowledgements**

This research was supported financially by the Iowa Energy Center. I would like to extend my gratitude and appreciation to Norm Olson, Linda Hintch, Siphon Ndlela and other Iowa Energy Center staff members for their support of the project. Thank you Dan Erickson for being a great lab partner throughout graduate school. Additionally, I would like to thank everyone at Lincolnway Energy (Nevada, IA).

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## **CHAPTER 4. PHOSPHORUS REMOVAL FROM A BIOPROCESSING WASTE WITH SOLID-LIQUID SEPARATION TECHNOLOGIES**

A paper prepared for submission to Bioresource Technology

Christopher R. Koza and J. (Hans) van Leeuwen

### **Abstract**

Methods to concentrate and remove phosphorus from a waste bioprocessing stream utilized as a fertilizer was investigated to produce two more desirable products, liquid fertilizer with a larger nitrogen to phosphorus (N:P) ratio and a “solid” phase with a lower N:P. A large percentage of phosphorus in this liquid slurry, left over from an industrial fermentation process, is tied up in the suspended solids. The study focuses on the use of coagulants and polymers to aid separation of suspended solids from the bulk liquid, ultimately producing a liquid fertilizer with a higher nitrogen to phosphorus ratio over the initial liquid slurry.

## 1. Introduction

Waste sludges from many processes, including industrial fermentation, wastewater treatment, food processing and livestock confinement operations, can be utilized as organic fertilizers, due mainly to useful nitrogen (N), phosphorus (P) and potassium (K) concentrations [1]. The increased demand for agricultural products due to global population growth, livestock production and biofuels leads to increased demand for fertilizers. To meet these demands, nutrients in agriculture lands need to be replenished. Fertilizers accomplish this task; they play a critical role in sustainable production of biomass [2]. The industries mentioned above are capable of providing low-value waste products, high in nutrients, for use as organic fertilizers.

Existing soil conditions, crop type, crop rotation and other management practices are factors that lead to varying quantities and nutrient formulations of fertilizers applied to agricultural land. The NPK rating placed on commercial fertilizers is used to indicate quantities of the three primary macronutrients needed for plant growth. This rating system lists nitrogen (N), phosphorus ( $P_2O_5$ ) and potassium ( $K_2O$ ). A wide variety of synthetic fertilizers are available with varying NPK ratings. However, the variety of nutrient ratios in organic waste fertilizers is limited. In many cases farmers have a limited selection of products; they obtain organic waste fertilizers from industries near their fields. The nutrient ratio within these waste products may be unsuitable for certain crops or existing soil conditions. Control over the nutrient ratios by treatment of the waste product can make the fertilizer more desirable.

Amana Farms, located in eastern Iowa, which operates 4,000 acres of corn, 2,500 acres of soybeans, 250 acres of wheat and 2,400-head cow/ calf herd, uses a waste product from an industrial fermentation process. This product is utilized as an organic fertilizer for corn crops. With application rates suitable for corn nitrogen needs, more phosphorus is applied than needed for the given season. Consequently, the product cannot be used the following season. In addition to the nitrogen to phosphorus ratio concerns, this waste stream is produced year around but not continuously land applied, so it must be stored. The dilute nature of this liquid fertilizer (1% total nitrogen) results in a large volume of the material (4,000-5,000 gal/acre) needed to deliver an ample nitrogen supply for the corn crop. A treated product with a higher nitrogen concentration could significantly reduce storage requirements.

This study aimed at concentrating phosphorus with solid-liquid separation techniques. The resulting liquid fraction (higher N:P ratio) and sludge fraction (lower N:P ratio) can be utilized for different fertilizer applications. Physical separation technologies with chemical aids (coagulants and polymers) were investigated to determine equipment, chemical selection and doses for treatment of this waste stream at Amana Farms. It has to be pointed out that the management of Amana Farms had certain stipulations with any chemical aids including Generally Regarded as Safe (GRAS) certification and no metal salt addition (aluminum and Iron).

## **2. Methods and Materials**

### 2.1 Material

The nutrient rich liquid slurry produced by DuPont, Genencor (Cedar Rapids, IA, USA) is currently used at Amana Farms (Amana, IA, USA) as a liquid fertilizer. Amana Farms provided the samples, which were delivered to the laboratory and stored (4°C) to limit microbial growth. The soybean-based liquid slurry is the residual growth substrate of an industrial fermentation. The fermentation process was not disclosed.

### 2.2 Material analysis

Samples were analyzed for total solids (TS), total suspended solids (TSS) and total dissolved solids (TDS) according to the Standard Method for the Examination of Water and Wastewater [3]. Samples were analyzed for total phosphorus by USEPA's Methods for Chemical Analysis of Waters and Wastes [5].

### 2.3 Jar Test

A jar tester (Phipps & Bird, Richmond, VA, USA) was used to coagulate/flocculate 500-ml samples of liquid slurry in 1-L beakers. Stirring speed was adjusted to 175 rpm, chemicals added and mixed for 2 minutes. Next, stirring speed was reduced to 50 rpm for 15 minutes. This flocculated suspension was used for subsequent testing.

## 2.4 Coagulants

Coagulants including calcium hydroxide, calcium carbonate, ferric chloride and Nalco 410 (GRAS certified) were tested by the ability to flocculate solids during jar testing. This visual investigation was a precursor to in depth analysis of solid-liquid separation techniques and N:P ratio changes. The management of Amana Farms had certain stipulations with any chemical aids including Generally Regarded as Safe (GRAS) certification and no metal salt addition (aluminum and iron).

## 2.5 Polymers

Polymers have different chemical and physical characteristics. Cationic, anionic and nonionic polymers are the three broad categories with multiple subsections based on chemical structures. Visual investigations were conducted to investigate which polymers flocculated solids in this waste slurry. Polymer samples (liquid and solid) were diluted to a 2% stock solution. Stock solutions were used to dose the waste slurry during jar tests.

In addition to the preliminary investigation, determining which polymers flocculated solids, a detailed solid-liquid separation study with nitrogen and phosphorus analysis was conducted utilizing waste sludge treated with polymers. All waste sludge samples treated with polymers were dosed with 2% stock polymer solutions and run through jar tester procedure.

## 2.6 Physical solid-liquid separation

Common in wastewater treatment, the sludge volume index (SVI) is used to assess the settling characteristics of sludges such as this particular waste sludge. The SVI method in Standard Methods for the Examination of Water and Wastewater [33] defines SVI by Eq. (4.1):

$$SVI = \frac{SV_{30}}{X} \quad (4.1)$$

as the volume of sludge occupied by one gram of suspended solids (mL/g),  $SV_{30}$  is the volume of settled sludge after 30 minutes in a 1-L graduated cylinder (mL/L), and  $X$  is the suspended solids concentration of the sludge being tested (g/L).

Dilute sludge volume index (DSVI), a test used to determine settling characteristics of sludges with high solids concentrations, beyond the hindered settling phenomenon [6]. In this procedure, a series of twofold dilutions ( $n$ ) were prepared using the waste sludge and water. After dilution, all samples were placed into 1-L graduated cylinders and mixed to uniformly distribute the solids. The sludge settled for 30 minutes to calculate the settled sludge volume ( $SV_{30}$ , mL/L) [34]. Suspended solids concentration of the fungal suspension ( $X$ , g/L) was calculated, utilizing the procedure in Methods for the Examination of Water and Wastewater [3]. The DSVI was calculated from Eq. (4.2).

$$DSVI = \frac{SV_{30}2^n}{X} \quad (4.2)$$

A lab centrifuge was utilized to determine the solid-liquid separation potential of the waste sludge at various g-forces. Centrifuge bottles were filled with 250 ml of waste sludge, placed in the centrifuge and run (2000-5000 g's) for 1 or 2 minutes. After

running samples in the centrifuge, sludge volumes were calculated as the percent of settled suspended solids.

### 3. Results and Discussion

#### 3.1 Solids matrix

Solid content of the waste slurry is presented in Table 4.1. Due to the high solids content, in respect to other waste sludge, solid-liquid separation becomes a challenging task. High total solids content (10% w/v), total suspended solids (4.5% w/v) and dissolved solid content (5.5% w/v) creates problems for solid-liquid separation.

Table 4.1: Solids analysis

Constituent	Concentration (g/L)
Total solids (TS)	100
Total suspended solids (TSS)	45
Total dissolved solids (TDS)	55
Total volatile solids (TVS)	85
Total fixed solids (TFS)	15

#### 3.2 Coagulation and flocculation

##### *Calcium hydroxide*

Initially, calcium hydroxide was added to the entire liquid slurry at various doses to precipitate phosphorus. Visual inspection of the coagulation and flocculation of solids was observed. Upon completion at various doses, it was concluded that calcium

hydroxide is not applicable due to large sludge volumes and poor settling characteristics of the resulting sludge.

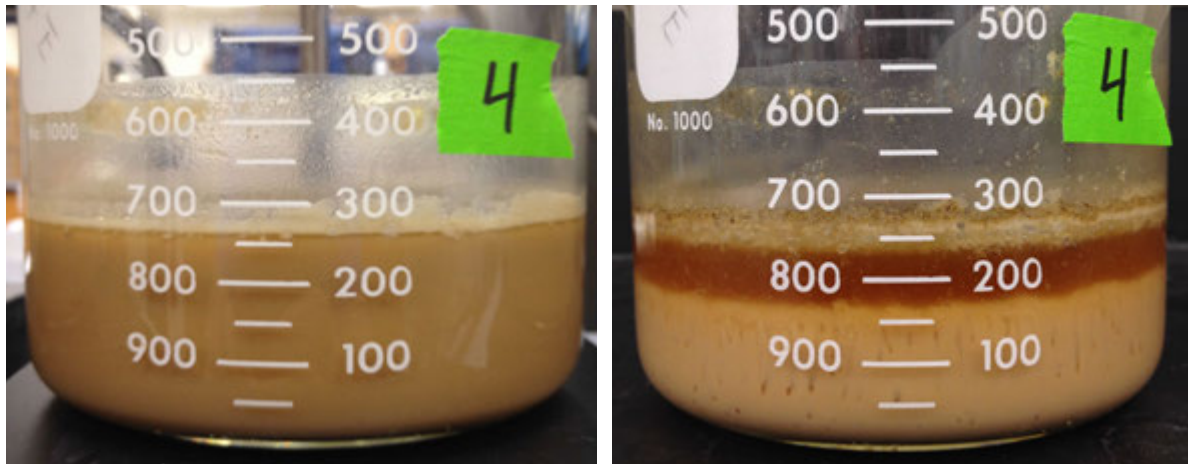


Figure 4.1: Calcium hydroxide jar test (1.5 g/L dose)  
(Left to right: flocculation, 6-h settling)

Due to the high solid content of the liquid slurry, the liquid slurry was separated and tested. Supernatant liquid was dosed with calcium hydroxide after gravity settling. Observations indicate the process was successful in flocculating solids, however the calcium hydroxide produces a large secondary sludge volume. Ultimately, no further tests were conducted because of the large volumes of sludge produced.

#### *Coagulants and polymers*

Ferric chloride, aluminum sulfate and other metal-based coagulants were not investigated for this liquid slurry. Applying metals to Amana Farms land were undesirable, so these products were not tested.



In addition to metals loading, polymers needed to meet GRAS (generally regarded as safe) standards, which limited the selection of polymers. However, a series of GRAS polymers were obtained from Nalco (Naperville, IL, USA). Visual inspection during jar tests concluded that one of Nalco's GRAS polymers was suitable for flocculation. Many polymer doses were tested and at minimum, a 800-ppm dose was required for flocculation.

### 3.2 Initial Nitrogen and Phosphorus Analysis

Nutrient (total nitrogen and phosphorus) testing was conducted on fractions of waste slurry after settling, filtration and centrifugation. Methods that separated solid particulates from the bulk slurry were conducted to investigate the link between solids removal and nitrogen/ phosphorus removal.

Table 4.2: Initial Nitrogen and Phosphorus Analysis

Separation Method	Material	Total Nitrogen (mg/L)	Total Phosphorus (mg/L)
None	Whole sample	10,000	2,000
Gravity sedimentation	Liquid supernatant	7,200	980
Centrifugation	Liquid supernatant	6,000	850
Filtration (1 micron)	Liquid filtrate	4,400	560

From these initial results, it was shown that a large percentage of phosphorus is tied up in solid particulates. The larger concentration of phosphorus, opposed to

nitrogen, in the suspended solids makes solid-liquid separation a viable method to concentrate and remove phosphorus from the bulk waste sludge. The concentrated phosphorus sludge can be removed, as the remaining liquid would have a higher nitrogen to phosphorus ratio. Additional tests were needed to optimize the process. These tests were conducted utilizing coagulants and polymers in parallel with solid-liquid separation studies.

### 3.4 Solid-Liquid Separation

#### *Dilute Sludge Volume Index*

The sludge volume index, commonly used in domestic wastewater treatment to assess the settling characteristics of waste activated sludge, was used to understand the settling characteristics of this liquid slurry. Sludge samples did not settle in 30 minutes, so a dilute sludge volume index was calculated. These values (Fig. 4.2) indicate that gravity sedimentation is feasible if not inhibited by high solid concentrations.

This value indicates that sedimentation is feasible if not inhibited by the large solid content; however advanced gravity settling may be an option at current solid concentrations.

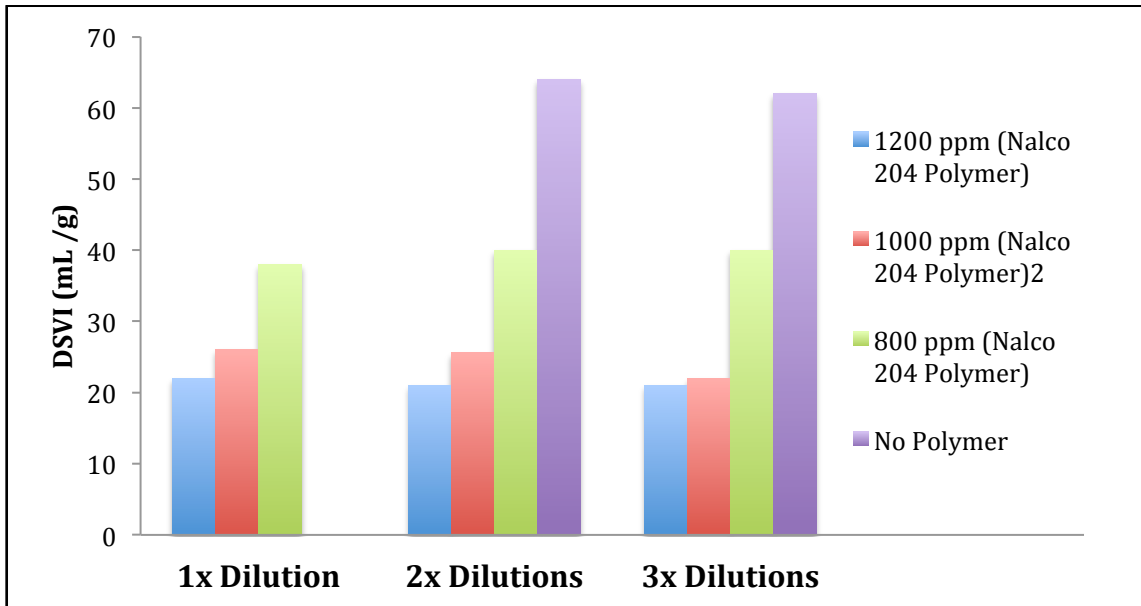


Figure 4.2: Dilute Sludge Volume Index

Based on these results, there is a noticeable change in settling behavior with the samples flocculated with 1000-parts per million (ppm) of Nalco's 204 GRAS polymer in comparison to 800-ppm or no polymer addition. Additionally, no significant improvement in settling was obtained at 1200-ppm. Ultimately, additional solid-liquid separation techniques were investigated with addition of 1000-ppm of Nalco's 204 GRAS polymer.

### *Centrifugation*

Based on results from the dilute sludge volume index tests, the waste sludge was dosed (1000 ppm, Nalco 204) and run through jar tester to flocculated solids. This treated waste sludge was compared against non-treated waste sludge in a lab centrifuge for solid-liquid separation. More specifically, for centrifugation, g-forces play

a critical role in the separation of solids from the bulk liquid. Larger g-forces produce more compact sludge, ultimately improving the product quality by concentrating phosphorus into a smaller volume sludge.

Operating conditions including runtime and g-forces were selected based on typical g-forces found in industrial equipment and the treatment rates at Amana Farms and available centrifuge size. Settled sludge volumes from the centrifuge study are presented below in Fig. 4.3.

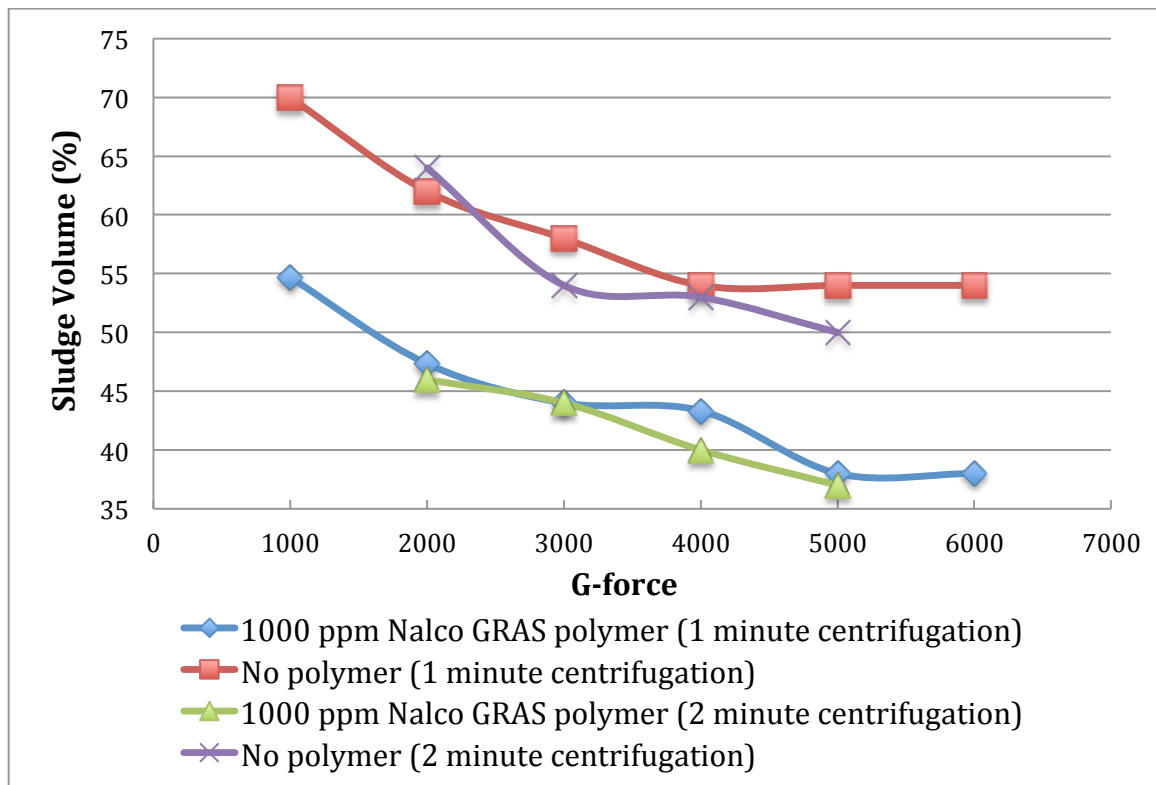


Figure 4.3: Lab centrifugation study

Impact of centrifugation time is illustrated above. Negligible changes in sludge volumes were observed between samples run for one or two minutes. Short operating

time results in less equipment. This is advantageous for industrial implementation because of the high capital costs associated with equipment.

A horizontal decanter centrifuge is an ideal piece of industrial equipment to separate solids from the bulk waste product by advanced gravity sedimentation. The equipment can be run continuously without the need to periodically stop and clean like most filtration equipment.

### 3.5 Nitrogen and Phosphorus Analyses

Methods to separate particulates from bulk sludge were investigated. Upon completion of this solid-liquid separation procedure, the fractions were analyzed for total nitrogen and phosphorus. The figure below (Fig. 4.5) presents the nitrogen to phosphorus (N:P) ratio of the original sludge, centrate (with and without polymer) and filtrate.

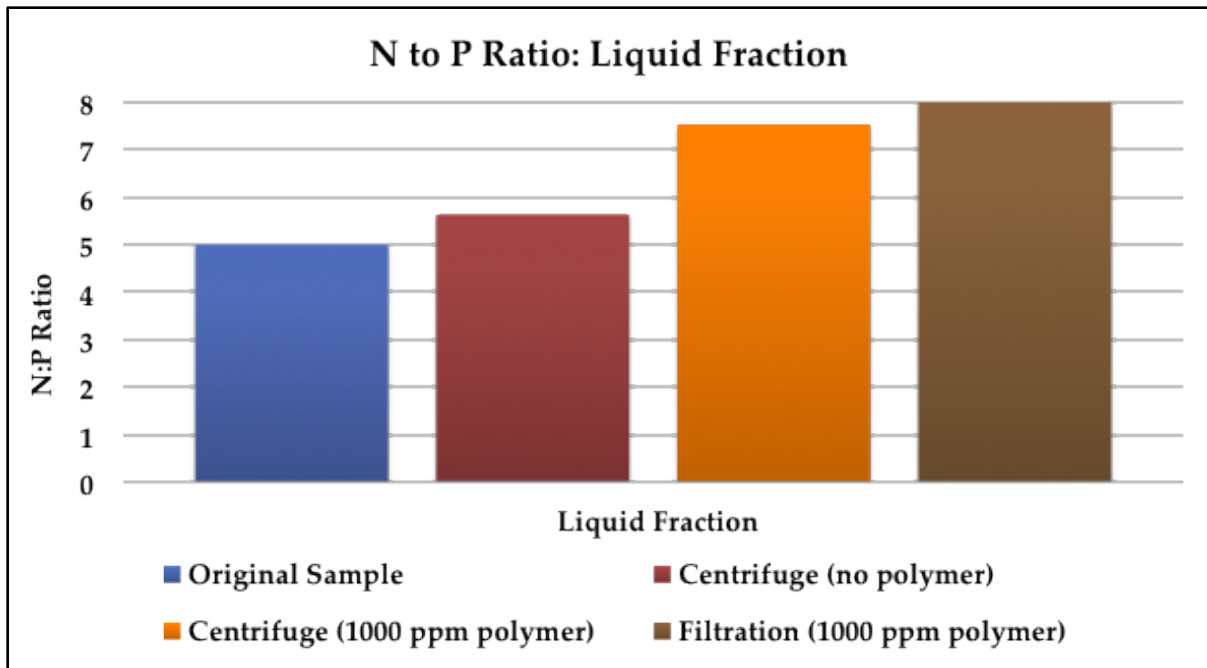


Figure 4.5: Nitrogen to phosphorus ratios - solid liquid separation procedures

Shown in Fig. 4.5, removing solid particulates from the sludge increases the N:P ratio of the remaining liquid. Centrifugation aided by a polymer is an advantageous method to concentrate solids from the bulk sludge, obtain a higher N:P ratio in the liquid fraction and lower N:P ratio in the sludge fraction. Filtration of the polymer treated sludge is yet another way to obtain a liquid sludge with a large N:P ratio. However, it is not a method suited for Amana Farms due to high capital and operating costs associated with filtration equipment and cleaning of fouled membranes.

Overall, 1 min centrifugation (3,000-5,000 g's) of polymer (1,000 ppm Nalco 204) treated sludge can produce a liquid concentrate with a higher N:P ratio. Fig. 4.6 illustrates the separation and concentration procedure.

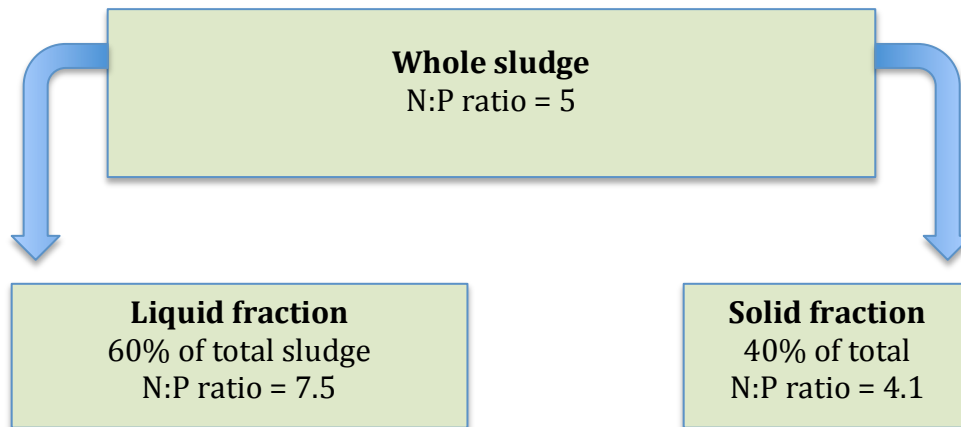


Figure 4.6: Centrifuge solid-liquid separation flow diagram

#### 4. Conclusion

A solid-liquid separation procedure utilizing 1,000 ppm (Nalco 204 polymer) along with centrifugation (3,000 to 4,000 g's) for one minute will separate a bioprocessing waste sludge into two products. The liquid product, with a 50% increase in N:P ratio, can be utilized as a nitrogen fertilizer on Amana Farm's corn fields. The sludge fraction can be used or sold as a phosphorus-rich fertilizer product.

#### Acknowledgements

The research was financially supported from Amana Farms (Amana, IA) and the Iowa Institute for Physical Research and Technology. I would like to thank John McGrath and the Amana Farm's staff for their collaboration. I would like to thank Shane Vonnahme and Nalco, Ecolab for providing polymer samples and technical assistance.

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## CHAPTER 5. GENERAL SUMMARY AND CONCLUSION

### Summary

Over the past 100 years, researchers have developed methods and equipment for solid-liquid separation. Mechanisms, operating conditions and various other parameters separate the equipment types from one another. Understanding of these mechanisms will help in the selection of the proper equipment for a specific product. Work on solid-liquid separation of biological waste materials described above combines the understanding of fundamental principles and available technology to develop an application for industry.

### Conclusions

#### A Pilot Study on Dewatering Fungal Biomass From a Novel Corn Ethanol Bioprocess

Gravity screening coupled with centrifuge filters proved to be efficient and practical processes to remove free liquid from the fungal biomass. The high solids content (25-30% dry matter) of the processed biomass over a short time period makes this equipment an attractive option for industrial application.

- Filament interactions create a meshed biomass matrix, which allow the use of coarse screens (1mm opening) without fungal biomass loss.
- The low g-force/ pressure applied to this fungal biomass matrix limits cake compression, which maintains a lower resistance to filtration for proper drainage of free liquid from the biomass cake.

- Further mechanical dewatering (>30% dry matter) is not possible without lysing the cells. Remaining liquid will be removed by thermal drying to 85-90% solids.
- Collaboration with screen and centrifuge filter manufactures should be initiated to test material for optimized loading rates and other operating parameters.

#### Phosphorus removal from a bioprocessing waste with solid-liquid separation technologies

A solid-liquid separation procedure utilizing 1,000 ppm (Nalco 204 polymer) along with centrifugation (3,000 to 4,000 g's) for one minute will separate a bioprocessing waste sludge into two products. The liquid product, with a 50% increase in N:P ratio, can be utilized as a nitrogen fertilizer on Amana Farm's corn fields. The sludge fraction can be used or sold as a phosphorus-rich fertilizer product.

## **CHAPTER 6. ACKNOWLEDGEMENTS**

I would like to extend my appreciation to those who made this research possible and who helped me throughout graduate school. Thank you Dr. van Leeuwen for his support throughout this project. His dedication to education and research is remarkable. I was truly blessed to have such a wonderful major professor, who took a personal interest in the research as well as my own personal development throughout graduate school. Also, thanks to my co-major professor Dr. Stephanie Jung and committee member Dr. Shihwu Sung for serving on my committee, making suggestions and taking interest in my project.