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Recycling effect in expanded granular sludge bed reactor and implementation of Six Sigma in a methane generation process

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RECYCLING EFFECT IN EXPANDED GRANULAR SLUDGE BED REACTOR & IMPLEMENTATION OF SIX SIGMA IN A METHANE GENERATION PROCESS

by

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Joseph Smith, Advisor
Douglas Ludlow
Peter Ryan

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This thesis consists of the following three articles which will be submitted for publications as follows:

Paper I: Pages 03–23 will be submitted to Taylor and Francis

Paper II: Pages 24–55 will be submitted to Institute of Industrial and Systems Engineers

ABSTRACT

Brewery industries are the largest consumers of water among several production industries. Despite consuming these huge amounts of water and electricity, they generate by-products that are harmful to the environment. These by-products contain organic, inorganic, and solid wastes with high chemical oxygen demand (COD) strength. The anaerobic digestion (AD) process plays an important role in treating this wastewater. This study investigates the design and development of an expanded granular sludge bed reactor (EGSB) effluent recirculation, which can achieve high COD removal efficiency of the wastewater and enhance the efficiency of generating biogas with high yields and increases in the concentration of methane in biogas. The recirculation of effluent for different organic loading rates was studied and investigated.

The EGSB system was improved by applying Six Sigma methodology, which followed the DMAIC (Define Measure Analyze Improve Control) process to achieve the goal. By applying this methodology, the production of biogas was improved, process defects were identified and corrected, and significant improvements in the methane composition of the biogas were achieved.

ACKNOWLEDGMENTS

This thesis would not have been complete without the support of Dr. Joseph Smith. I want to offer my thanks to Dr. Smith for his endless tolerance and direction at each progression of this work. There are no words to describe his character, which has had an unmeasurable bearing on me. I am perpetually thankful to Dr. Douglas Ludlow and Dr. Dwindle Ryan for their direction in composing the thesis. I am always thankful to them for willing to be on my committee advisory group, and for giving recommendations to the advancement of my thesis. I am much obliged to Haider Al-Rubaye for preparing me on test methodology, digestion project, and other projects. I am appreciative to Dean for his assistance amid the mechanical activity, and to Shadha Jebur for helping me with FTIR. I express gratitude toward Nathan Leigh, Dave Satterfield and Jonathon Sidwell. I should thank my lab mates for making lab a moment home especially on account of Haider, Shadha, Anand, Vivek, Aso, Jia, Han, and Jeremy. I am much obliged to Dr. Darrell Ownby for teaching me to fly a plane, and for the fun part.

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NOMENCLATURE

Symbol	Description
mm	Millimeter
°C	Degree Celsius
gal	Gallons
g	Gram
L	Liter
mg	Milligram
mL	Milliliter
CH3COOH	Acetic acid
N	Nitrogen
$CaCO_3$	Calcium carbonate
PO_4^{3-}	Phosphate
SO_4^{2-}	Sulfate
NH_3-N	Ammonia
OLR	Organic Loading Rate
HRT	Hydraulic Retention Time

SECTION

1. INTRODUCTION

This thesis is presented as two papers describing the work related to the treatment of wastewater from brewery industries, the study of effluent recirculation in an expanded granular sludge bed reactor (EGSB), and the implementation of Six Sigma methodology on improving biogas production rates and methane composition in biogas.

Brewery industries consume huge amounts of water for production. To produce one liter of beer, almost 8–10 L of wastewater is generated. This wastewater contains chemical oxygen demand (COD) in higher concentrations, along with organic, inorganic, and solid compounds. Untreated brewery wastewater may be discharged in many ways such as into water bodies like (oceans, lakes, and rivers), and municipal sewer systems where it should be pre-treated in brewery water treatment plants before being discharged. Still this wastewater can cause significant potential effects on the environment. The well-known method from the past century for treating this kind of wastewater is the anaerobic digestion (AD) process. The anaerobic digestion process involves the degradation of this wastewater by a series of steps by groups of anaerobic bacterium. The degradation steps are hydrolysis, acidogenesis, acetogenesis, and methanogenesis by the bacterium groups called hydrolysis bacterium, acidogenesis bacterium, acetogenesis bacterium, and methanogenic bacterium. These bacteria are the reason for the degradation of this wastewater and the liberation of biogas. The anaerobic digestion process helps industries to meet the regulatory requirements with minimal post treatment. The biogas produced during this process contains 50–75% methane, 50–25% carbon dioxide, and less than 1% nitrogen, hydrogen, and hydrogen sulfide. Bioenergy generated from this AD process in the form of biogas can be used in several daily activities like domestic heating purposes, generating electricity, and heating boilers in industries, which can support the world energy demand.

The EGSB reactor was designed and developed for carrying out anaerobic digestion process. The reactor was fed with different concentrations of feed from Square One Brewery. The first paper deals with the study of recirculation effect of effluent in this reactor. The study was conducted for different organic loading rates with different recirculation rates, and the performance for these different scenarios was analyzed.

The second paper deals with the study and implementation of Six Sigma methodology for the EGSB reactor system. The possible failures and defects were identified towards the improvement of the overall process, yield of the system, and methane composition in biogas.

PAPER

I. RECYCLING EFFECT IN EXPANDED GRANULAR SLUDGE BED REACTOR

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ABSTRACT

The effects of effluent wastewater recirculation from a two-stage expanded granular sludge bed reactor of distillery wastewater were studied. The reactor was fed with different ranges of organic loading rates (OLRs) starting from 2 g COD/L/day, 4 g COD/L/day, and 6 g COD/L/day. For COD concentration of the substrate 20 gCOD/L, varying recirculation rates at 20%, 30%, and 40% of OLRs were used. Results showed the biogas production rate was increased to 51.41% for 30% recirculation rate at 6 gCOD/L/day OLR. Where the introduction of a high of pH 7.15–7.25 effluent into the expanded granular sludge bed reactor helped to create the suitable conditions for generating high biogas production in the system. The chemical oxygen demand value decreased from 20 gCOD/L to 955.67 gCOD/L, which shows that significant improvement and recycling of the effluent decreases the amount of fresh feed required and alkalinity required to maintain the pH concentration of the feed.

Keywords: Distillery wastewater, Anaerobic Digestion, Organic Loading rate, Chemical Oxygen demand

1. INTRODUCTION

Process wastewater from brewery industries must be treated before discharging it into the environment because of its high organic, inorganic, chemical oxygen demand content, and solid content [1]. Brewery companies have to pay the local municipal authorities for further treatment of this wastewater. Burning fossil fuels has several negative effects on the environment and population [2], but it is one of the easiest and most available forms of energy. But in the future, most of these fossil fuels will be depleted and due to ever-growing population [8][10], the energy is always in demand. To overcome these energy demand issues, people have come up with renewable energy sources, and one of them is the generation of biofuels from waste [3]. The wastewater from the brewery industries can be further treated an with anaerobic digestion process to generate energy, which can be a small addition to the world energy demand and helps to treat wastewater from impurities [2][4]. The biofuel generated from the anaerobic digestion process is called biogas which contains 50–70% methane, 30–50% carbon dioxide, and about 1% nitrogen, hydrogen, and hydrogen sulfate [11]. The anaerobic digestion process has been used as a form of wastewater for the past few centuries. This is a biological degradation process with the help of anaerobic microorganisms, where the microorganisms consume the organic compounds in wastewater and release methane and carbon dioxide as a by-product. These by-products can be further used for generating electricity, domestic purposes, heating, and vehicular fuel [2]. Besides getting energy from the wastewater, it also reduces the amount of greenhouse gases and water pollution levels [4]. An expanded granular sludge bed (EGSB) reactor is used in our study with a design change that allows it to separate solid, liquid, and gas from the reactor with no effort. The bed expansion provides a favorable atmosphere for interaction between biomass and substrates. One of the advantages of using an EGSB is that, its recycle stream helps during a high organic loading rate in case of a continuous process by recirculating part of the effluent to enrich the biogas production. Zuo investigated the effect of recirculation of effluent in two-stage anaerobic digestion process and discovered that it helped in mitigating the inhibition of volatile fatty acids, and improved the biogas production rate [5]. Zuo experimented on the methane production rate, which was affected by the recirculation of effluent in a positive way and hydrolysis was enhanced by a recirculation rate of 0.6, which caused the efficient removal of COD [6]. The overall biogas yield was increased from 0.5 L/g to 0.66 L/g by enhancing the recirculation rate from 0 to 1.4 [6]. Zuo concluded that the recirculation rate improved the decomposition of vegetable waste in the acidogenic stage by transferring huge amounts of volatile fatty acids to the system by shortening the hydraulic retention time (HRT), which results in a higher composition of methane [7]. Al-Rubaye conducted the studies on anaerobic digestion (AD) by developing an Aspen Plus model for different substrates with varying HRTs, temperature, and pressure of the system [3][9]. The anaerobic digestion process consists of four main steps: hydrolysis, acidogenesis, acetogenesis, and methanogenesis, as shown in Figure 1.

Hydrolysis is a process where the addition of water breaks the chemical bonds between the large polymers (carbohydrates, proteins, and fats) to form simpler monomers (sugar, amino acids, and fatty acids). It is a primary stage in the anaerobic digestion process. By adding water, the cations and anions of the water react with large polymer molecules, which helps in unbinding the bonds between them due to changes in pH. The

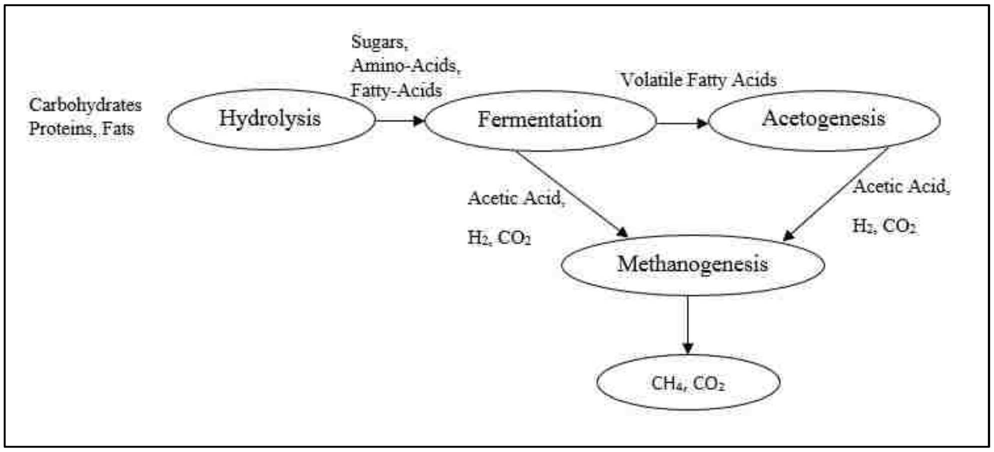

Figure 1. Anaerobic Digestion Degradation Process Flow

degradation of substances to simpler monomer molecules takes place in the process by extracellular enzymes.

Acidogenesis is a secondary stage in the AD process. Here, simple monomers are broken down to volatile fatty acids by fermentative bacteria. It is a biological process where acidogenic bacteria break the larger chain monomers into shorter chain volatile fatty acids, ketones, alcohols, carbon dioxide, and hydrogen.

Acetogenesis is a tertiary stage in the AD process. It is a biological process where acetogenic bacteria groups convert volatile acid groups into acetic acid, carbon dioxide, and hydrogen. These three bacteria groups produce acetic acid: clostridium aceticum, acetobacter woodii, and clostridium termoautotrophicum. Additional hydrogen and carbon dioxide is produced from the following bacteria groups: homoacetogens, syntrophes, and sulphoreductors.

Methanogenesis is the last stage in the AD process. Biological reaction take place to form methane and carbon dioxide from acetic acid, carbon dioxide, and hydrogen with the help of anaerobic methanogens bacterium groups.

2. EXPERIMENTAL SETUP AND PROCEDURE

The anaerobic digestion process was studied in the Aspen Plus model, and built in solid works and analyzed with Start CCM+ for compatibility and for different process cases by Al-Rubaye [3]. The process and instrumentation diagram for the process that was designed is shown in Figure 2. The two-stage anaerobic digestion system was built in lab as shown in Figure 3.

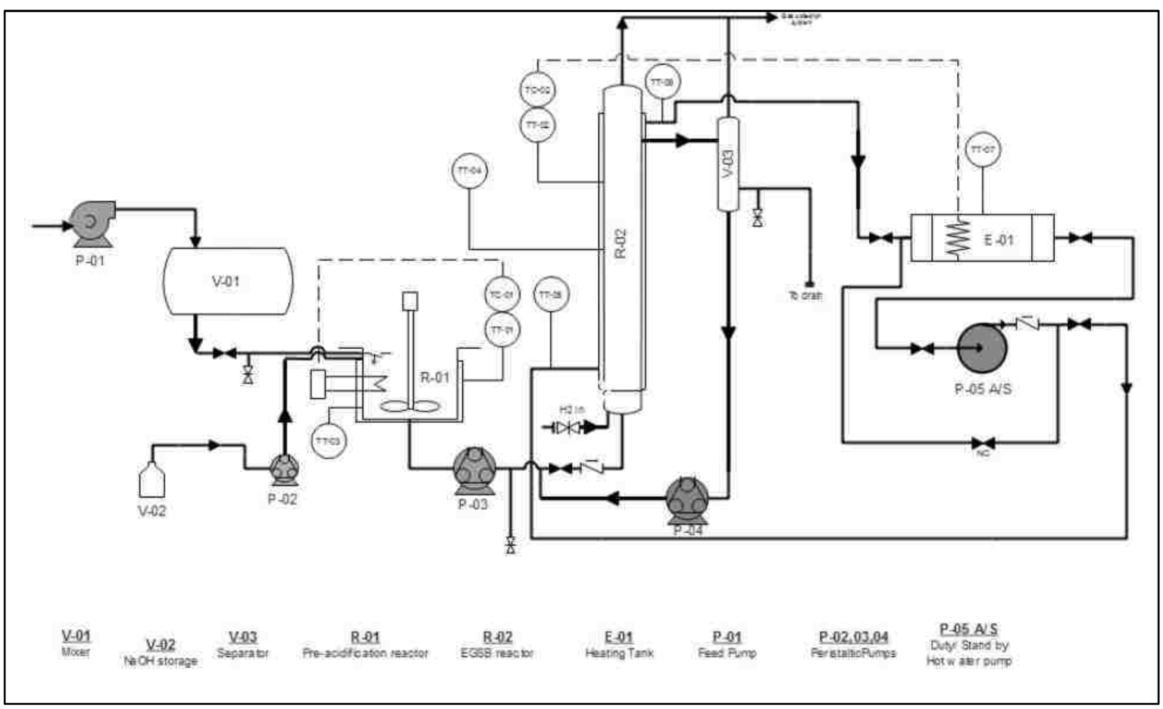

Figure 2. Process and Instrumentation Diagram

Figure 3. Two-Stages Expanded Granular Sludge Bed Reactor System

The first two-stages of the AD process took place in the pre-acidification reactor, and the last two stages occurred in the main reactor generating biogas. Brewery process wastewater treatment took place in a high-rate anaerobic digestion process called an EGSB. The AD system was split into four units: process wastewater storage unit, pre-acidification (PA) reactor unit, main reactor unit, and hot water system. The first unit of the system was the 55 gal horizontal plastic tank V-01 used to store process stillage beer wastewater brought from Square One Brewery & distillery. In this tank, the required COD concentrations for the process were prepared. This analysis was conducted at the department of chemistry and material research center of Missouri University of Science and Technology as per standard methods provided by the United States Geological Survey and United States Department of Environmental Protection. The wastewater from the storage tank was transferred to the next process through gravity flow. The second unit consists of the pre-acidification reactor R-01, which was 33 gal stainless steel tank with an agitator. This helps to maintain uniformity inside the reactor and to maintain a uniform temperature of 34ºC–35ºC, heated from a direct contact heating element inside the reactor. The temperature was controlled using the temperature controller TC-01, which was integrated with a heating element. The pH change in the PA reactor launches the hydrolysis step of the process where large polymer chain molecules will break down to small monomers. The pH was maintained by adding a sodium hydroxide (NaOH) solution stored in a V-02 container using Milwaukee MC122 pH meter with peristaltic pump P-02 to achieve the pH range of 4.5–5.0 during the operation. Alkalinity was maintained through the regular manual addition of sodium bicarbonate ($NaHCO_3$), which was also responsible for minute changes in the pH of the wastewater. After this stage where second stage acidogenesis of the AD process took place, the simple monomers were broken down to form volatile fatty acids by acidogenic bacterium. A TT-03 thermocouple was provided to monitor and collect the temperature data from the PA reactor. At this point the wastewater was rich in volatile fatty acids, which led to the acetogenesis step where most of the volatile fatty acids were converted into acetic acid, carbon dioxide and hydrogen. Produced carbon dioxide and hydrogen were stored in an air tank using Focal-Flux vacuum pumps (Model no. VAC-100) for future experimental studies on hydrogen injection to an EGSB reactor at different organic loading rates. The unreacted volatile fatty acids and acetic acids were pumped to R-02 EGSB reactor from PA reactor using P-03 variable frequency drive peristaltic pump (Model no. BT100S) from Golander based on different organic loading rates (OLR) defined for the study. Here, OLR 2 gCOD/L/day, 4 g COD/L/day and 6 g COD/L/day were investigated. A nutrient medium was injected periodically to the reactor through a septum port. The nutrient medium contained the mineral base I, mineral base II, nutrient base, and a buffer base required for the process as shown in Table 1.

Table 1. Nutrient Medium Composition

	Component	Amount of Component (mg/mL)
Mineral Base I	Cobalt (Co)	0.062
	Iron (Fe)	1.126
	Manganese (Mn)	0.0139
	Boron (B)	0.0044
	Zinc (Zn)	0.0119
	Molybdenum (Mo)	0.0020
	Nickel (Ni)	0.0062
	Selenium (Se)	0.0104
	Copper (Cu)	0.0026
Mineral Base II	Calcium (Ca)	5.4
	Magnesium (Mg)	2.36
Nutrient Base	Nitrogen (N)	13.9
	Phosphorus (P)	11.4
	Sulphur (S)	6.76
Buffer Base	Sodium Bicarbonate ($NaHCO_3$)	40

The EGSB reactor was divided into three regions. The lower part was aluminum plenum, where it had two nozzles, one for gas injection and another for liquid injection. The gas sparger was installed for the gas injection into the reactor. The gas injection study will be conducted in future. The liquid port was T-shaped distributor where the wastewater will be discharged evenly into the reactor. Above this, a liquid distributor was installed consisting of 171 holes 2 mm in diameter to distribute wastewater and to support the biomass particles. The second part of the reactor was jacketed and made of acrylic material. The reactor length is about 63in with a diameter of 7.5in and a working volume of 12 gal. Hot water from the hot water system was made to recirculate through this jacket to maintain the temperature of the reactor. A TC-02 temperature controller was used to control the temperature of the reactor by maintaining the hot water temperature. This was the part where the biomass was loaded with the acetic acids and unreacted volatile fatty acids, which were consumed by methanogenic bacterium inside this biomass, generating methane gas and carbon-dioxide by majority. The upper body was a special design for the reactor, which separates gas, liquid, and solid biomass. The produced gas was collected in a glass tank filled with water, where the gas displaces the water into another container, which shows the amount of gas generated. The water displaced will be measured by a pre-calibrated marking on the water collection tank. The pressure inside the reactor was monitored using an Omega pressure transducer and the indicator was about 14.9~15.9 psig. The solid biomass stayed inside the reactor to further carry out the digestion process. The effluent from the reactor will be discharged into the buffer recycle tank V-03. It is constructed of acrylic material with a length of 25in and a 4.5in diameter with full a volume size of 6.8 gal. It consists four ports: one for effluent inlet from the reactor, one for gas outlet generated inside the recirculation reactor, one for effluent discharge to the sewer system, and one for recirculation of the effluent to the main reactor using a P-04 variable frequency drive peristaltic pump (Model no. BT100S) from Golander. The recirculation rates varied to 20%, 30%, and 40% based on different organic loading rates. The last part of the system is the hot water system, E-01. It is made of stainless steel with a working volume of 23 gal. The water in the tank was heated through a direct heating element, and it was connected to the main reactor controller TC-02 for maintaining the temperature of the water. The hot water was circulated using centrifugal pumps P-05 A/S. These pumps were connected to a time

controller set for 30 min, where it switched the pumps every 30 min. The thermocouples TT-04, TT-05, and TT-06 were inserted at different spots of the reactor to monitor the temperature inside the main reactor. The TT-07 was placed in a hot water system for monitoring the temperature. These thermocouples were connected using a Pico TC-08 data logger system for recording and monitoring purposes.

3. CHARACTERIZATION

The wastewater brought from Square One Brewery was analyzed for total solids (TS), total volatile solid (TVS), total suspended solid (TSS), and total dissolved solids (TDS), as shown in Table 2.

The protocols followed for these tests were taken from the U. S. Geological Survey. The wastewater collected from distillery vessels contains a high chemical oxygen demand value of 90 g COD/L. This wastewater was diluted to the required concentration of 20g COD/L. During the operations, the COD and VFA form reactor were regularly collected and analyzed. The effluent was analyzed for volatile acids, COD (HACH model no. DRB 200 was used for digestion of COD vials), phosphate, sulfate, total alkalinity, total ammonia, and total nitrogen.

Table 2. Characterization of Wastewater

Characterization of Wastewater	
VSS (mg/L)	23
TSS (mg/L)	1,542.0
TDS (mg/L)	80,266.0
pH	3-4

These were measured using spectrometer from HACH (Model no. DR3900) and reagents provided by HACH (TNT vails: 872, 823, 845, 865, 870, 833, and 828 respectively). Values are shown in Table 3 and Table 4.

The biomass was also characterized for TS, TVS, TSS, TDS and pH, which was obtained from a local company called Anheuser Busch Beverage. The characteristic values are shown in Table 5.

4. RESULTS AND DISCUSSION

Wastewater from 90 g COD/L was diluted to 20 g COD/L by adding tap water and stored in a storage tank. Then, it was sent to a PA reactor where the wastewater was treated with NaOH solution to maintain a pH of 4.5~5.0 and a temperature of about 35°C. The wastewater was charged to the EGSB reactor based on an organic loading rate starting from 2 g COD/L/day, 4 g COD/L/day, and 6 g COD/L/day. The EGSB reactor was operated at a mesophilic temperature of about 37°C and was maintained throughout the reactor. The effluent from the reactor was recirculated based on OLR rates (i.e., 20%, 30%, and 40% of OLR were recirculated) to study the effects of COD, VFA, and biogas production rate and methane composition.

The effluent from the main reactor was analyzed and found to contain a significant amount of COD and VFA along with methanogenic bacterium group. A recirculation experiment was conducted to improve these issues. The methane composition was improved to 73.24%.

The Figure 4 shows the variation of methane composition percentage during different OLRs and different recirculation rates. It indicates that the lower the OLR the higher the composition of methane in biogas, and it also shows that the highest recirculation leads to a high percentage of methane in the gas stream (i.e., 40% recirculation rate at OLR 2 g COD/L/day has the maximum methane percentage of 73.2%). However, the COD removal efficiency works quite the opposite way to methane composition percentage.

The COD removal efficiency was improved to 96.84%, Figure 5 shows the COD removal efficiency for different OLR ranges at different recirculation rates. It shows higher that the recirculation rate lowers the efficiency of COD removal (i.e., the 20% recirculation at OLR 2 g COD/L/day has the maximum COD removal capacity). The overall biogas production rate was found to be 19.45 gal/day.

Table 3. Recirculation Rates for Each Organic Loading Rate and Hydraulic Retention Rate

No.	(OLR)	HRT, Day	Recycle, %	COD Influent, mg COD/L	COD pre-acidification, mg COD/L	COD Effluent, mg COD/L	VFA Influent, mg CH3COOH/L	VFA Pre-acidification, mg CH3COOH/L	VFA Effluent, mg CH3COOH/L
1	2	10	20	20,000.00	15575.67	631.67	2493.34	4112.34	146.67
2			30		13103.67	647.34	2508.67	3030.67	151.67
3			40		15234.00	776.00	2186.00	4057.67	150.67
4	4	5	20		14416.00	709.34	2373.34	4024.00	162.67
5			30		15557.34	955.34	3057	5044.00	204.67
6			40		15162.67	1134.34	2340	4227.34	174.00
7	6	3.34	20		14767.67	1292.34	2466.34	3680.00	290.67
8			30		15360.00	1213.34	2941.34	4950.34	223.67
9			40		15001.34	1375.00	2547.67	3543.34	198.66

Table 4. Characterization of Effluent at Different Recirculation Rates

No.	(OLR)	Recycle, %	Total Nitrogen, N mg/L	Total Alkalinity, $CaCO_3$ mg/L	Phosphorous, PO_4^{3-} mg/L	Sulfate, SO42- mg/L	Total Ammonia, NH3-N mg/L	Phenol, mg/L
1		20	125.00	1350.33	214.00	87.56	187.00	4.63
2	2	30	62.33	1430.33	242.00	82.96	233.66	-
3		40	57.03	1356.67	227.00	93.60	163.00	4.83
4		20	66.53	1071.00	239.00	93.93	182.66	4.86
5	4	30	52.96	1316.00	302.00	99.06	232.33	-
6		40	53.06	716.34	245.00	105.67	110	6.37
7		20	32.50	1025.34	269.00	108.00	133.00	11.10
8	6	30	64.23	960.00	279.34	118.00	185.34	-
9		40	28.76	981.00	265.34	104.34	205.00	10.36

Table 5. Characterization of Granular Biomass

Characterization of Granular Biomass	
VSS (mg/L)	60,914.66
TSS (mg/L)	422
TDS (mg/L)	5832
Particle size (mm)	2-5
pH	6.9-7.2

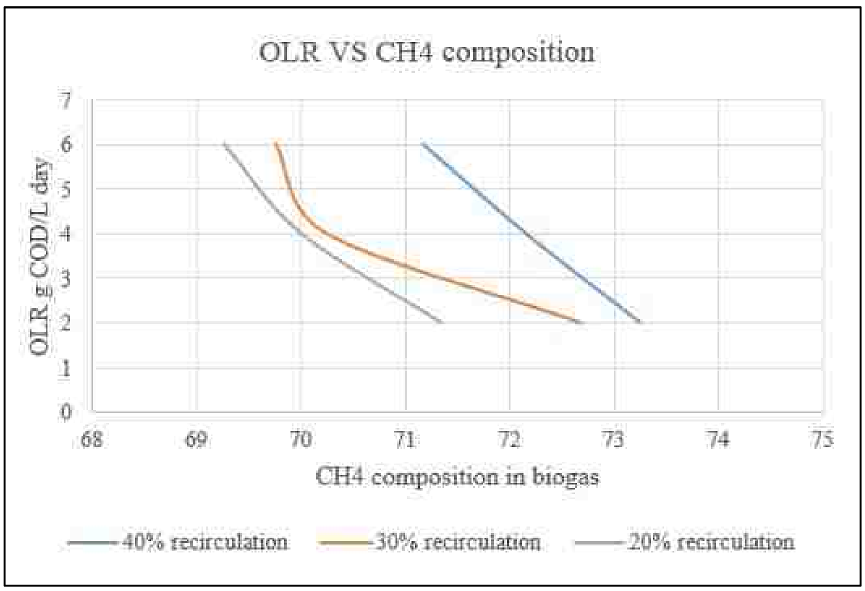

Figure 4. Variation of Methane Composition for Different OLRs at Different Recirculation Rates

Figure 6 shows the biogas production rate for different OLRs at different recirculation rates. The biogas production increases with an increase in OLR and an increase in the recirculation rate. The highest recirculation rate was 40%, but the maximum biogas production rate shows for 30% because the reactor was inhibited during the process.

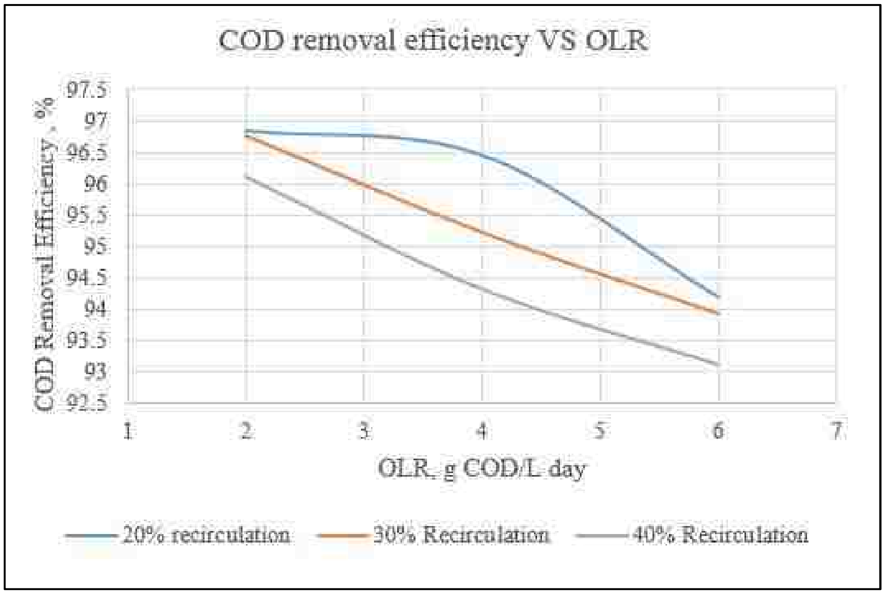

Figure 5. COD Removal Efficiency for Different OLRs at Different Recirculation Rates

Figure 7 and Figure 8 shows the different scenarios conducted for COD analysis. The pre-acidification COD remains almost constant during ORLs values ranging between 14000 g COD/L to 15600 g COD/L. The COD for the effluent shows significant results (i.e., it has decreased from 20000 g COD/L to 631.66 g COD/L). The pH and alkalinity for the effluent at different OLRs and different recirculation rates were analyzed.

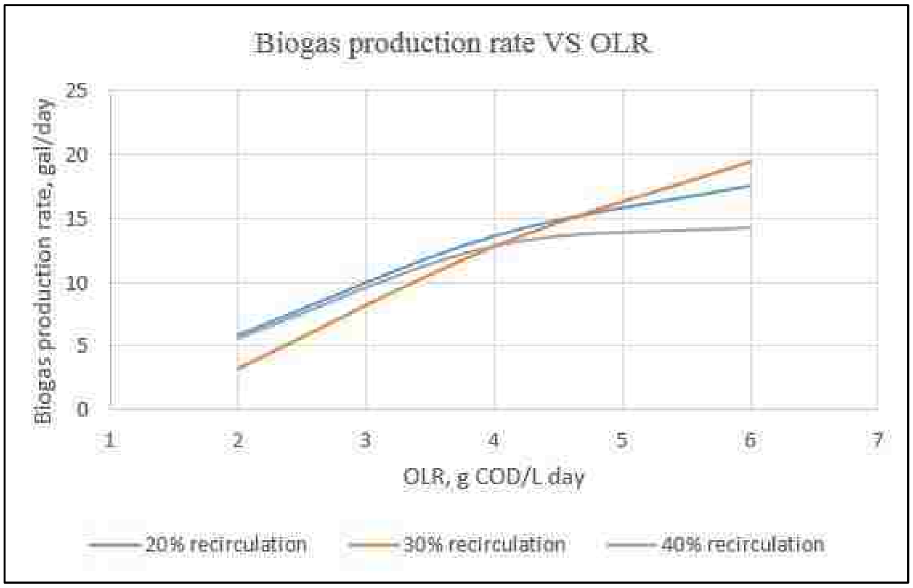

Figure 6. Biogas Production Rate for Different OLRs at Different Recirculation Rates

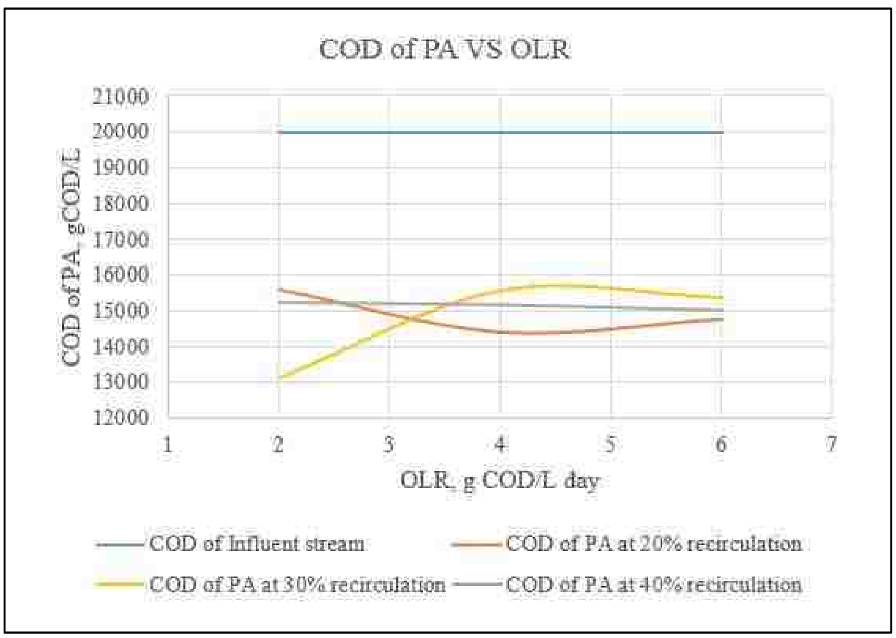

Figure 7. COD from Pre-Acidification for Different OLRs at Different Recirculation Rates

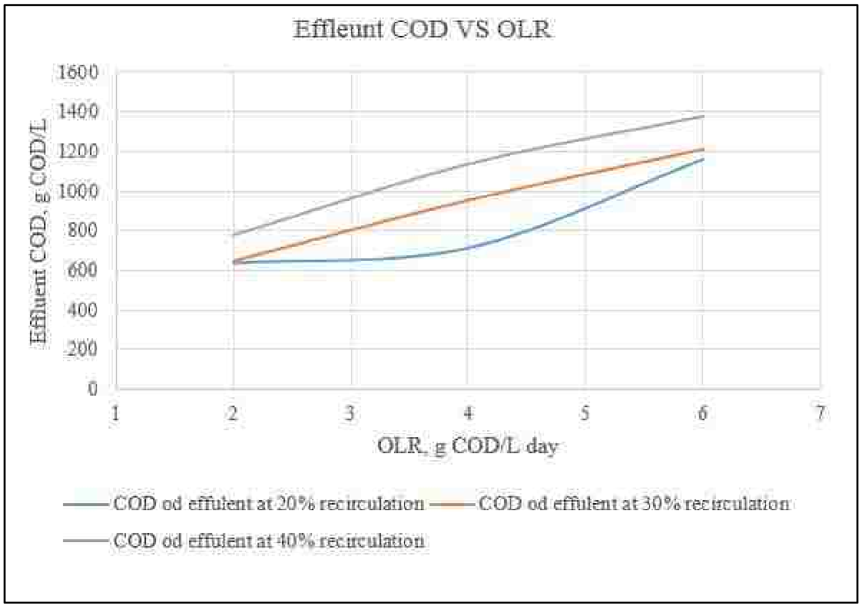

Figure 8. Effluent COD Variation for Different OLRs at Different Recirculation Rates

Figure 9 indicates the values of pH and alkalinity remains almost similar values. Except for OLR 6 g COD/L/day at 40% recirculation value, the alkalinity for this was lower because of inhibition to the reactor, where it suppressed the activity of the process.

Volatile fatty acids at different stages of the process were examined, as shown in Figure 10. The samples were taken from the influent stream after pre-acidification and the effluent stream for different OLRs at different recirculation rates. The values of the VFA from the influent for different cases are similar, the same case with VFA of all the PA values, the effluent VFA show good results, and almost all the VFA was consumed in the main reactor (i.e., VFA from 5044 g CH_3COOH/L decreased to 146.66 g CH_3COOH/L).

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5. CONCLUSION

The effluent recirculation study was conducted on an expanded granular sludge bed reactor for brewery distillery wastewater. The substrate concentration was maintained at 20 g COD/L for six different scenarios, starting from OLR 2 g COD/L/day, 4 g COD/L/day, and 6 g COD/L/day at 20%, 30%, and 40% recirculation rates. The results show that the higher the recirculation rate the higher the methane composition in the biogas, and as the OLRs increases with increases in the recirculation production rate of biogas also increases. The COD removal efficiency increases as the OLR decreases, and the lower value of OLR will have the maximum removal efficiency of COD.

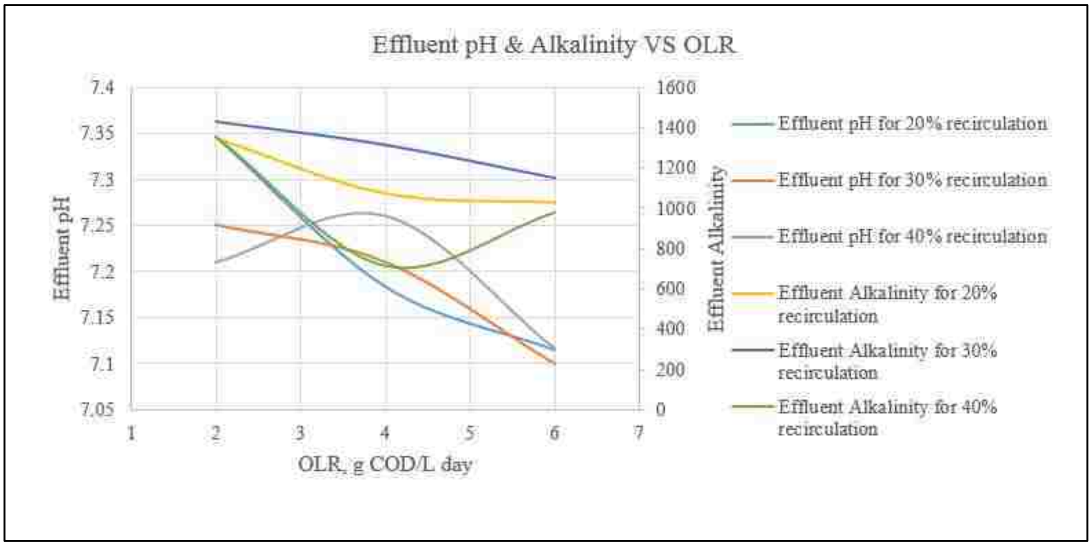

Figure 9. Variation of pH, Alkalinity of Effluent for Different OLRs at Different Recirculation Rates

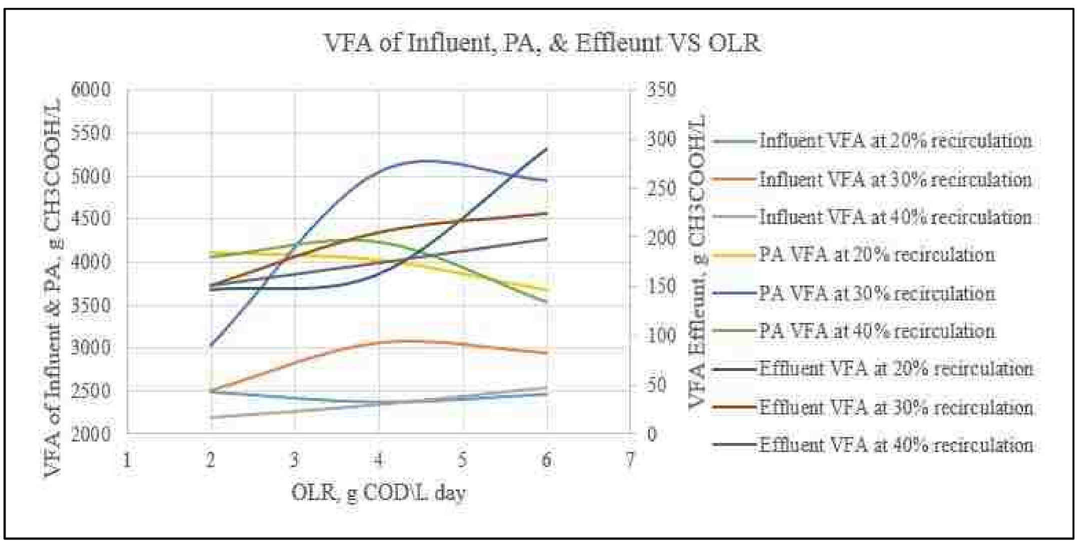

Figure 10. Variations of Volatile Fatty Acids of Influent, PA, & Effluent for Different OLRs at Different Recirculation Rates

REFERENCES

[1] M. Ghorbanian, R. M. Lupitskyy, J. V Satyavolu, and R. E. Berson, “Impact of Hydraulic Retention Time at Constant Organic Loading Rate in a Two-Stage Expanded Granular Sludge Bed Reactor,” *Environ. Eng. Sci.*, vol. 31, no. 6, pp. 317–323, 2014.

[2] K. Moriarty, “Feasibility Study of Anaerobic Digestion of Food Waste in St . Bernard , Louisiana,” Natl. Renew. Energy Lab., no. January, 2013.

[3] H. Al-Rubaye, S. Karambelkar, M. M. Shivashankaraiah, and J. D. Smith, “Process Simulation of Two-Stage Anaerobic Digestion for Methane Production,” Biofuels, pp. 1–11, Apr. 2017.

[4] N. J. Themelis, “Anaerobic Digestion of Biodegradable Organics in Municipal Solid Wastes,” Found. Sch. Eng. Appl. Sci. Columbia Univ., no. May, pp. 1–56, 2002.

[5] Zhuang Zuo, Shubiao Wub, Wanqin Zhang, and Renjie Dong, “Effects of organic loading rate and effluent recirculation on the performance of two-stage anaerobic digestion of vegetable waste,” Bioresource Technology, 556–561, 146 (2013).

[6] Zhuang Zuo, Shubiao Wub, Wanqin Zhang, and Renjie Dong, “Performance of two-stage vegetable waste anaerobic digestion depending on varying recirculation rates,” Bioresource Technology, 266-272, 162 (2014).

[7] Zhuang Zuo, Shubiao Wub, Wanqin Zhang, and Renjie Dong, “Performance enhancement of leaf vegetable waste in two-stage anaerobic systems under high organic loading rate: Role of recirculation and hydraulic retention time,” Applied Energy, 279-286, 147 (2015).

[8] Yebo Li, Stephen Y. Park, Jiying Zhu, “Solid-state anaerobic digestion for methane production from organic waste,” Renewable and Sustainable Energy Reviews, 821–826, 15 (2011).

[9] R. S. Peris, “Biogas Process Simulation using Aspen Plus,” 2011.

[10] A. S. Dieter Deublin, Biogas from Waste and Renewable Resources. Weinheim, 2008.

[11] A. Dhir and C. Ram, “Design of an Anaerobic Digester for Wastewater Treatment,” Int. J. Adv. Res. Eng. Appl. Sci., vol. 1, no. 5, pp. 56–66, 2012.

II. IMPLEMENTATION OF SIX SIGMA IN A METHANE GENERATION PROCESS

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ABSTRACT

Distillery processed wastewater contains organic and inorganic compounds with high chemical oxygen demand (COD) strength, which causes negative impacts on the environment. Instead of discharging this wastewater to sewer systems, it can be pre-treated and used for energy generation before discharging it into the environment. The pre-treatment that could possibly be used is the anaerobic digestion process. Several groups of bacteria will feed on this wastewater to generate methane gas. An expanded granular sludge bed reactor has been utilized as the main reactor for this process. The biogas produced will mainly consist of methane and carbon dioxide. This paper investigates, how to improve the biogas production and how to improve the methane composition in the generated gas using Six Sigma methodology. The DMAIC (Define Measure Analyze Improve Control) method has been implemented in this biogas production process so that process failures could be identified to improve the gages used in measurement and to enhance the yield and composition of methane.

Six Sigma DMAIC methodology behaves as a roadmap to understand various unexplored areas in this process that could help in organizing and updating the standard operating procedure for the experiments. The improvement was observed after introducing Six Sigma tools and concepts in the experiment. The yield was increased from 11 gal to 28 gal in 60hr, which is 154.5% increase in yield and an increase in the percentage composition of methane in the yield from 50% to 76%.

Keywords: Six Sigma, methane gas, distillery wastewater, anaerobic digestion

1. INTRODUCTION

Six Sigma consists of tools and techniques for strategic process improvement. It was first introduced in 1986 by Bill Smith and Mikel J. Harry, engineers from Motorola [1][2]. The Six Sigma methodology is used for finding defects and minimizing variation through a continuous strategy that reduces the defects to 3.4 defects per million opportunities in process and production designs. The Six Sigma strategy helps in continuous improvement towards minimization of error, delays, and defects in any organization process [3]. Expanding industrial growth has resulted in a huge amount of wastewater generation discharged into the environment. Polluted wastewater contains many organic, inorganic, and solid compounds which causes enormous effects on the environment [4]. Most of this wastewater can be treated by an anaerobic digestion process, where it produces biogas as a product that can be used in domestic purposes such as vehicle fuel and electricity generation [5]. Food industries, food waste from houses, slaughter houses, and breweries generate a huge amount of solid and liquid waste, which are large sources of carbon content. Wastewater from these sources is high in chemical oxygen demand (COD), and plays an important role in the production of biogas. Chemical oxygen is defined as the capacity of water to consume oxygen during degradation processes [6]. The anaerobic digestion is a process of generating biogas and is a preliminary purification step of wastewater using different kinds of microorganisms in the absence of oxygen [8]. Anaerobic digestion reduces the organic components level and chemical oxygen level in the wastewater by generating a source of sustainable energy [2][7].

This paper presents a case study of DIMAC (Define Measure Analyze Improve Control) methodology of Six Sigma in two-stage expanded granular bed reactor. The purpose of this research was to describe the application of Six Sigma methodology in streamlining the defective components, instruments, and processes used for anaerobic digestion systems in a two-stage expanded granular bed reactor and to examine the test conditions that contributed to the high yield of biogas production and the increase in methane composition of the biogas.

2. EXPERIMENTAL SETUP

Process wastewater treatment takes place in a high-rate anaerobic digestion process called ‘expanded granular sludge bed’ (EGSB). EGSB is divided into four units: process wastewater storage unit, pre-acidification (PA) unit, main reactor unit, and hot water system. Process stillage beer wastewater was brought from Square One Brewery & Distillery and the wastewater samples were characterized for total solids (TS), total volatile solids (TVS), total suspended solid (TSS), and total dissolved solids (TDS). The samples were analyzed at the Department of Chemistry and Material Research Center at Missouri University of Science and Technology as per standard methods provided by the U. S. Geological Survey and U. S. Department of Environmental Protection. The pH was measured using a Pasco airlink probe (model no. 671-136). Volatile acids, COD (HACH model no. DRB 200 was used for digestion of COD vials), phosphate, sulfate, total alkalinity, total ammonia, and total nitrogen were measured using a spectrometer from HACH (Model no. DR3900), and the reagents used were provided by HACH (TNT vails: 872, 823, 845, 865, 870, 833, and 828, respectively).

A process wastewater storage tank with a holding capacity of 212 L Figure 1 was used to prepare the required strengths of chemical oxygen demand (COD) from 90,000 g/L COD concentration provided from Square One. About 45 L of required COD strength was prepared and stored.

Figure 1. Wastewater Storage Tank

This wastewater was sent through a gravity flow to a pre-acidification tank, which is the second unit of the AD system. The level inside the PA tank was controlled by a floating valve arrangement. A pre-acidification stainless steel tank with a gross volume capacity of 125 L was fed with 65 L (Working Volume) of process wastewater for the pre-acidification process as shown in Figure 2.

Figure 2. Pre-Acidification Tank

Wastewater remained inside the reactor for 48 hr by maintaining temperature of 34ºC–35ºC though a direct heating element under constant agitation. The temperature of the wastewater was monitored (Pico TC-08) and controlled using a thermocouple connected to a heating element. During this period, the first three steps of the anaerobic digestion process takes place (i.e., hydrolysis). During this stage, a sodium hydroxide (NaOH) solution was added to maintain the pH of 4.5–5.0 of the wastewater by using a Milwaukee MC122 pH meter with an automatic peristaltic pump, which disintegrates the large polymers. Additional sodium bicarbonate (NaHCO3) was added to maintain the alkalinity of the wastewater, which accounted for a small change in pH of the wastewater. Acidogenesis then took place to convert the polymers into volatile fatty acids with the bacteria present in the wastewater and then it is further converted into acetic acid, carbon dioxide, and hydrogen during the acetogenesis stage. Carbon dioxide and hydrogen were stored in an air tank using Focal-Flux vacuum pumps (Model no. VAC-100). Acetic acid and unreacted volatile acids were pumped into the main reactor using a basic variable-frequency drive peristaltic pump (Model no. BT100S). Wastewater from the PA tank with different organic loading rates was pumped accordingly. A nutrient medium was added to the reactor through a septum port. A nutrient medium consisting of mineral base I, mineral base II, nutrient base, and buffer base are shown in Table 1.

Table 1. Nutrient Medium Composition

	Component	Amount of Component (mg/mL)
Mineral Base I	Cobalt (Co)	0.062
	Iron (Fe)	1.126
	Manganese (Mn)	0.0139
	Boron (B)	0.0044
	Zinc (Zn)	0.0119
	Molybdenum (Mo)	0.0020
	Nickel (Ni)	0.0062
	Selenium (Se)	0.0104
	Copper (Cu)	0.0026
Mineral Base II	Calcium (Ca)	5.4
	Magnesium (Mg)	2.36
Nutrient Base	Nitrogen (N)	13.9
	Phosphorus (P)	11.4
	Sulphur (S)	6.76
Buffer Base	Sodium Bicarbonate ($NaHCO_3$)	40

An expanded granular sludge bed reactor with a working volume of 45 L, Figure 3 was parted into four units: jacketed lower reactor, reactor bed, upper reactor, and recirculation buffer vessel.

Figure 3. Main Reactor

The lower part of the reactor is an aluminum body with a T-shaped liquid distribution system to distribute wastewater evenly inside the reactor. A port for gas was provided at the bottom with gas sparger for initial nitrogen injection and for hydrogen injection studies. Above the T-distributor there was a liquid distribution system, it is consisting of 171 holes of 2 mm diameter to support the biomass brought from Anheuser-Busch Brewery. Methanogenic bacteria were loaded into the lower reactor up to 60–70% of reactor volume. The reactor bed was constructed from acrylic material with a 7.5 in diameter and a 63 in height surrounded by 10 in jacket for hot water circulation. The upper reactor was specially designed for solid, liquid, and gas separation. The gas flowed out from the top of the reactor and was collected in a glass container. The solid biomass stayed inside the reactor, and the wastewater left the reactor and was collected in a buffer tank where part of it was recirculated and part of it was sent down the drain after analysis. The stainless steel hot water system with an 87 L capacity Figure 4 heated the water with a with heating element and recirculated the water using centrifugal pumps. The temperature was maintained using a thermocouple connected to a controller.

Figure 4. Hot Water System

3. METHODOLOGY

Six Sigma requires allocating high objectives, collecting data, and analyzing the results to reduce the defects in equipment and processes used in anaerobic digestion systems. Figure 5 indicates the five phases of six sigma methodology used in the process.

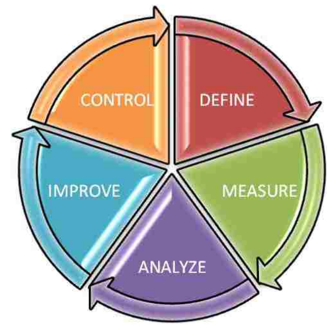

Figure 5. DMIAC Approach

DMIAC was used to existing process of AD system Figure 6 for maximizing the production of biogas and to increase the percentage composition of methane in the biogas.

Figure 6. Two-Stages Expanded Granular Sludge Bed Reactor System

3.1. DEFINE PHASE

The define phase defines the problem statement of the project and the goals to be achieved to satisfy the customer requirements. The aim of this project was to produce biogas in an anaerobic digester from distillery wastewater using methanogenic bacteria and to achieve the desired production of high purity methane. The problem states that biogas plants are known as “waste to energy” plants because they process organic waste from food industries, markets, and gastronomy to produce energy to be used as vehicular fuel or for domestic purposes. Producing methane and minimizing waste while keeping the operating costs at a minimum has always been a challenge. The purity and yield of methane can be increased by purging hydrogen gas into the reactor. Understanding the above issues and addressing them has been the core focus of this project. The main goal was to produce a high yield of biogas from distillery wastewater and to improve the composition of methane in the biogas further efficiently from 50% to 70%. This project was directed at the Department of Chemical and Biochemical Engineering, Missouri University of Science and Technology. Dr. Joseph Smith and Haider Al-Rubaye were the principal investigators for this project. The stakeholders of this project were Manohar, Haider, Akilesh, and Humayun. The project focused on the full potential of biomass technology within the United States. Our goal was to maximize the production of methane, which in turn reduces the carbon footprint and CO_2 emissions into the environment. This was in turn intended to reduce operating costs and improve sales of biogas.

3.2. MEASURE PHASE

In the measure phase, the flow diagram of the process was brought forward to understand the possible processes and factors that could affect the project goals. The process flow diagram (PFD), as shown in Figure 7, consists of various stages from feed storage to biogas production and includes a P-01 centrifugal pump for pumping wastewater from a 55 gal barrel to a V-01 wastewater storage tank. The wastewater flowed via gravity to a R-01 pre-acidification reactor. A floating valve arrangement controlled the level.

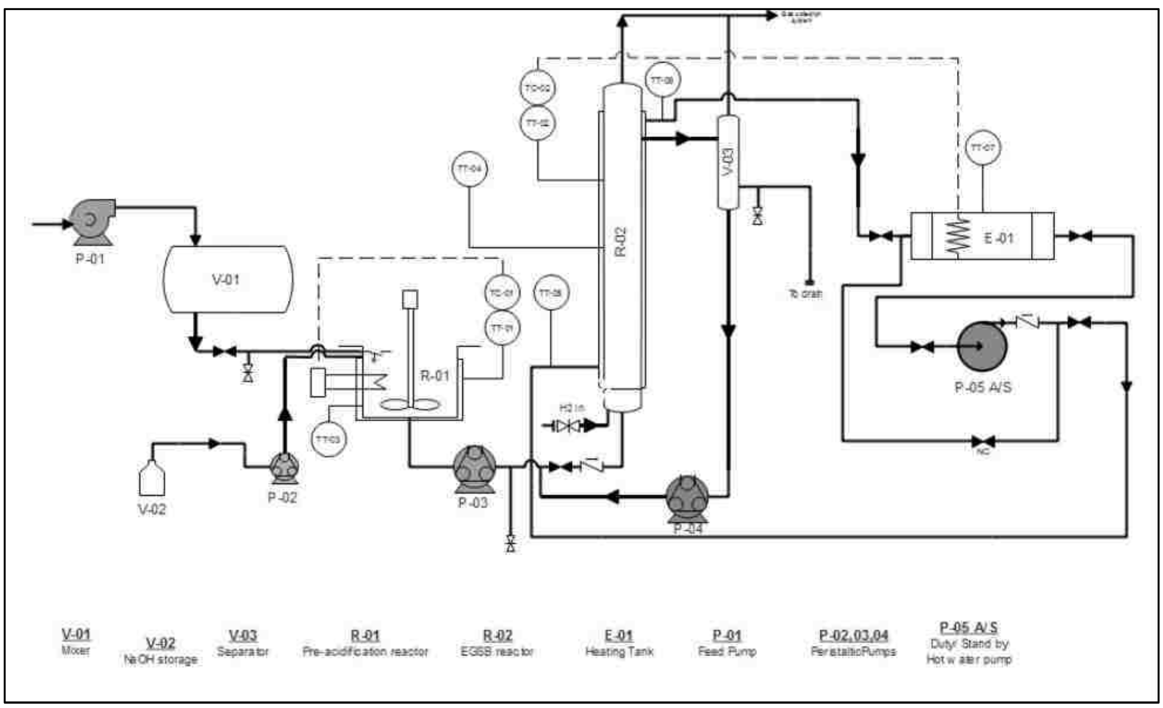

Figure 7. Process Flow Diagram

A V-02 sodium hydroxide container with P-02 peristaltic pump was used for maintaining the pH level inside the PA tank. A TC-01 temperature controller was used for maintaining the required temperature inside the PA tank using a heating element. A P-03 is a variable frequency drive peristaltic pump used to pump wastewater from PA tank to the R-02 main reactor. The main reactor temperature was maintained using a TC-02 temperature controller connected to a hot water system. A V-03 is a buffer recirculation tank where the wastewater from the main reactor was collected. Part of the wastewater was sent back to the main reactor using a P-04 variable frequency drive peristaltic pump, and the rest was drained. E-01 is the hot water system used for generating hot water at the required temperature using a heating element and the P-05 A/S are the centrifugal pumps for recirculating water through the main reactor jacket. T-03, TT-04, TT-05, TT-06, and TT-07 are thermocouples used to measure temperatures at different locations, which were monitored using a Pico data logger. From the PFD, a simple block diagram of the process was drawn to prioritize the most significant sub-processes, as shown in Figure 8.

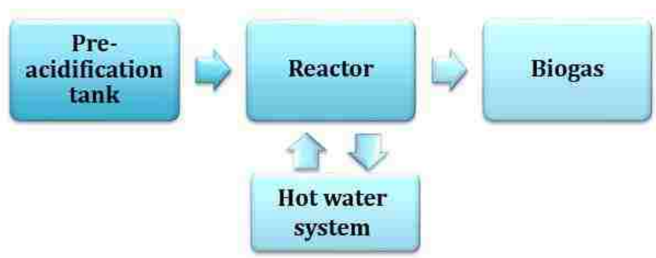

Figure 8. Simplified Block Diagram

Biogas produced from the main reactor was collected in a gas container. A water displacement method measured the biogas production rate, which is a manual method where the amount of gas generated inside the reactor will pressurize the water and displace the water to a water collection tank. One gal of displaced water is equal to 1 gal of generated gas Figure 9.

Figure 9. Water Displacement Method to Measure the Biogas Production Rate

The percentage composition of methane in the generated biogas was analyzed using an FTIR instrument Figure 10. The instrument used was NEXUS (470-FTIR) for the analysis with a 4 Cm^-1 Resolution and 16 scans. The gas cell (25 cm diameter 5 cm length) was used, from the Chemistry Department of Missouri University of Science and Technology.

Figure 10. Fourier-Transform Infrared Spectroscopy

The gage R&R (Repeatability and Reproducibility) study helped to investigate and identify if the measurement system used in the process was reliable or had a high variability. Also, the variability was caused due to different operators in operation. Methane production rate measurement system was required to calibrate and add a measuring scale to the container in the water displacement system. This was used to identify how much biogas was produced during the process. To calibrate and draw this scale, a 1 gal container was used to fill the tank and label the scale accurately. This study was conducted with three appraisers for two trials each, as shown in the Table 2.

The precision to tolerance capability ratio (CR) was 29.17%, and according to AIAG guidelines [9][11], the measurement system's variation should be less than 10% of the process variation to be acceptable. This high value was mainly due to the low range of the specification limits (USL, LSL) since a 1 gal measuring cylinder was used for this study instead of another container. It was also noticed that the mean value for Appraiser 1 was low while the mean range for Appraiser 3 was high, which could have led to this higher CR value [9][10][12]. The gage R&R study was performed for feed pump P-03 and recirculation P-04 pumps to identify the nominal flow rate of the pump Table 3.

Table 2. Gage R&R Study for Methane Production Rate Measurement Container

Sl.No.	Haider				Anand				Manohar			
	1	2	Mean	R1	1	2	Mean	R2	1	2	Mean	R3
1	0.93	0.91	0.92	0.02	1.08	1.09	1.085	0.01	0.95	0.93	0.94	0.02
2	1.05	1.09	1.07	0.04	1.08	1.06	1.07	0.02	1.01	0.99	1	0.02
3	0.92	0.9	0.91	0.02	1	0.98	0.99	0.02	1.08	1.1	1.09	0.02
4	1.07	1.08	1.075	0.01	1.02	1	1.01	0.02	1.09	0.91	1	0.18
5	0.9	0.91	0.905	0.01	1.07	1.08	1.075	0.01	0.91	0.92	0.915	0.01
6	0.93	0.95	0.94	0.02	1.02	1.05	1.035	0.03	0.99	0.97	0.98	0.02
7	1	1.06	1.03	0.06	0.95	0.95	0.95	0	0.92	1.02	0.97	0.1
8	0.98	0.96	0.97	0.02	0.98	0.95	0.965	0.03	0.98	0.91	0.945	0.07
9	0.99	0.99	0.99	0	0.92	0.91	0.915	0.01	0.92	0.99	0.955	0.07
10	0.9	0.91	0.905	0.01	1	1.01	1.005	0.01	1.01	1	1.005	0.01
Average	$\bar{x}1/R1$		0.9715	0.021	$\bar{x}2/R2$		1.01	0.016	$\bar{x}3/R3$		0.98	0.052

R	0.029667	Rx =	0.0085	$\sigma_{Measurement}$ =	0.018228
$\sigma_{Repeatability}$ =	0.017523	$\sigma_{Reproducibility}$ =	0.005021	σ_{Total} =	0.062493
				CR	0.291683

Table 3. Gage R&R Study Data for Pump P-03 and P-04

Average				
Sl. No.	**Volume (mL)**	**Time (min)**	**Volumetric Flow (mL/min)**	**RPM**
1	5	16.7486667	0.298565934	0.1
2	5	5.36333333	0.932261097	0.3
3	5	3.43	1.460077336	0.5
4	10	5.01666667	2.007449684	0.7
5	10	3.20333333	3.123815399	1
6	10	1.40333333	7.128158549	2
4	100	6.39333333	15.64160741	5
5	100	2.13	46.9490467	15
6	100	1.05333333	94.94436381	30
7	100	0.70553333	141.7540502	45
8	100	0.52773333	189.5322678	60
9	100	0.41106667	243.3589374	75
10	100	0.33886667	295.2580972	90
11	100	0.2833	352.9827038	105
12	100	0.25	400	120
13	100	0.2166	461.6805171	135
14	100	0.1833	545.553737	150

Two peristaltic pumps were purchased from Golander. This pump was calibrated by the vendor for standard conditions. Various trials were tested with different volumes of the water at different time ranges to identify the flow rate. The average of these volumes was taken, and a linear regression analysis was performed. This analysis resulted in an equation explaining the relationship between the dependent variable, (RPM) and the independent variable (volumetric flow), as shown in the Figure 11. The coefficient of the determination (R2) value was 0.9967, which indicates a good fitting. From this study, it was identified that 1 RPM ~= 3 mL/min.

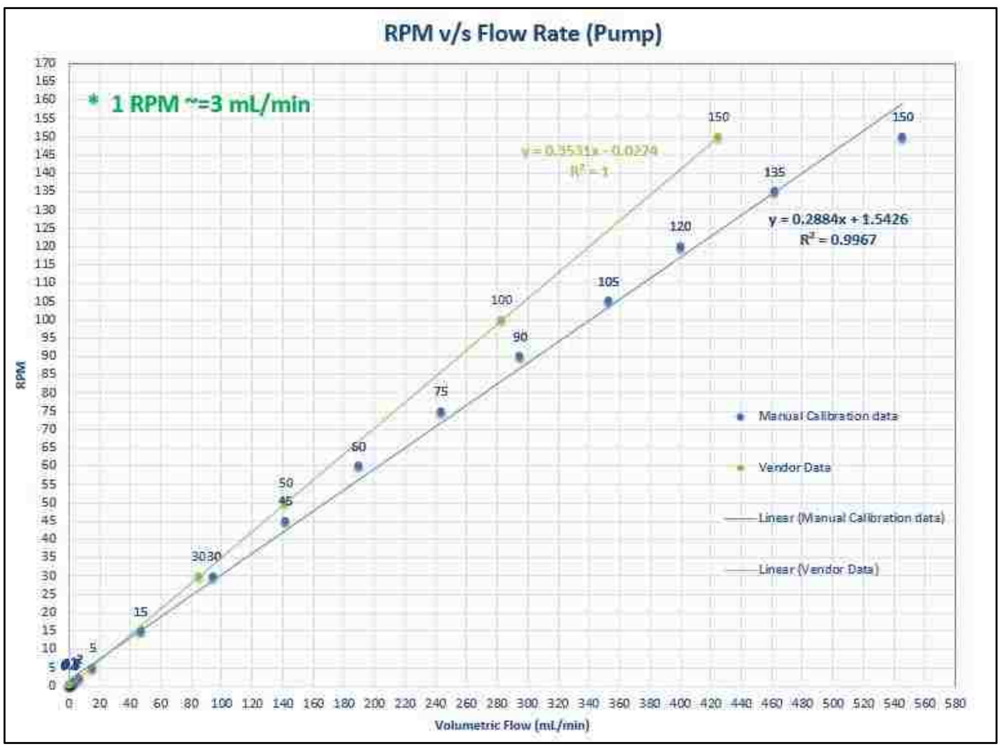

Figure 11. Plot for Volumetric Flow Rate Vs RPM Comparing Vendor and Experimental Data

3.3. ANALYZE PHASE

The analyze phase was performed by benchmarking and brainstorming rounds to find out the possible factors affecting biogas production and methane percentage improvisation. A fishbone diagram developed from brainstorming and referring to similar works is shown in Figure 12.

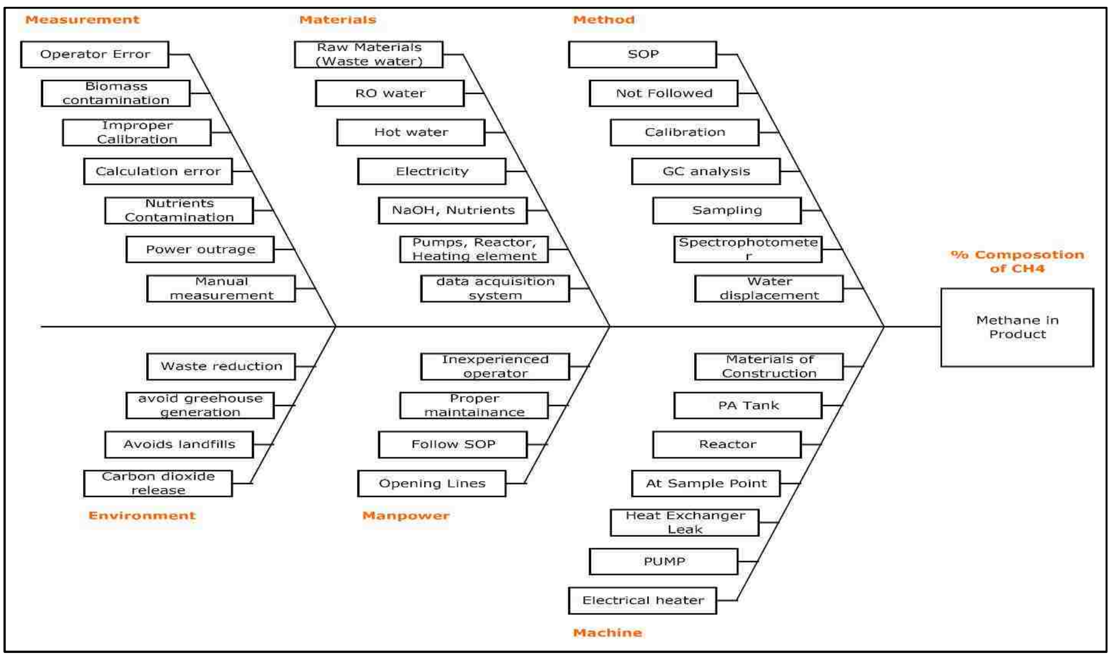

Figure 12. Cause and Effect (Fishbone) Diagram

This diagram helped to consider all the possible causes that have a direct or indirect effect on the methane yield. Applicable Ms from the 7 Ms were applied in developing this fishbone diagram. Following Figure 13 shows the various factors affecting the anaerobic digestion of the waste feed for biogas production were tabulated.

Figure 13. Factors Affecting the AD Process

These factors were identified by benchmarking the available literature on this topic. From the PFD and C&E diagram, it was concluded that the major factors that would significantly affect the process were temperature, pH, and organic loading rate (OLR).

Failure mode and effect analysis (FMEA) is a systematic and qualitative approach tool. FMEA was created using a spreadsheet by anticipating the possible process, instrument, equipment failures and overcoming measures for those failures. This study provided the identification of the failures before they occurred and possible solutions to avoid those failures. FMEA for AD process is shown in Table 4. This shows the main process functions are PA tank unit, reactor unit, and hot water system. Possible failure modes for each unit was detected and classified based on severity, occurrence rate, and ease of detection for the errors according to [13], which resulted in high-risk priority numbers (RPN) for each scenario, this was overcome by acting, upgrading the instrumentation, and automation, and replacing some equipment which resulted in low risk priority numbers

Table 4. Failure Mode and Effect Analysis of AD System

FAILURE MODE AND EFFECTS ANALYSIS

Item:	Methane Production	Responsibility:	Akhilesh, Humayun, Manohar	FMEA number:	MET143
Model:	Current	Prepared by:	Akhilesh, Humayun, Manohar	Page :	1 of 1
Core Team:	Akhilesh (Engineering), Humayun (Engineering), Manohar (Engineering)			FMEA Date (Orig):	12/6/2016 Rev: 1

Process Function	Potential Failure Mode	Potential Effect(s) of Failure	Sev	Class	Potential Cause(s)/ Mechanism(s) of Failure	Occur	Current Process Controls	Detec	RPN	Recommended Action(s)	Responsibility and Target Completion Date	Action Results: Actions Taken	Sev	Occ	Det	RPN
Pre-acidification tank	Heater failure	Temperature not maintained	9		Loss of product or low output	3	Operator training and instructions	3	81	Follow SOP			9	2	1	18
	Flow	Incomplete reaction or overflow	8		Spillage or loss of product	3	Operator training and instructions	3	72	Follow SOP			8	2	1	16
	pH controller	pH out of range	9		Loss of product or low output	5	pH display	3	135	pH controller installed			9	3	1	27
	Pump	Insufficient substrate supply	7		No supply to reactor	4	None	4	112	Controller installed			7	1	1	7
Reactor	Jacket	Temperature not maintained	9		Loss of product or low output	3	Operator training and instructions	6	162	Temprature controller installed			9	1	1	9
	Temperature probes	Temperature not detected	9		Loss of product or low output	3	Operator training and instructions	8	216	Data acquisition system installed			9	1	1	9
	Liquid - solid separator	Bloackage of effluent	9		Overflow or liquid flows through gas outlet	2	Operator training and instructions	2	36	Follow SOP			9	1	1	9
Hot water system	Heater failure	Temperature not maintained	9		Loss of product or low output	3	Operator training and instructions	4	108	Follow SOP			9	2	1	18
	Pump failure	Temperature not maintained	9		Loss of product or low output	3	Operator training and instructions	5	135	Controller installed			9	2	1	18
	Temperature probes	Temperature not detected	8		Loss of product or low output	3	Operator training and instructions	8	192	Temprature controller installed			8	2	1	16

The main ingredients for the process biomass and wastewater were characterized. Biomass is the porous material in which the microorganism will be impregnated Figure 14.

Figure 14. Biomass

Biomass plays a vital role in the production of methane. To maximize the production of biogas, biomass was analyzed, and a few of them are shown in Table 5 for VSS (volatile suspended solids), TSS (total suspended solids), and TDS (total dissolved solids). The feed for the AD process was distillery wastewater from a brewery. To get a high yield of biogas, the wastewater properties were analyzed, and it was determined that the COD (chemical oxygen demand) concentration in the water was a food source for microorganisms was maintained. The wastewater in the pre-acidification tank and effluent water were analyzed to see the proper usage of the COD level in the water, which is shown in Table 6.

Table 5. Characterization of Granular Biomass

CHARACTERISTICS OF BIOMASS GRANULAR PARTICLES	
VSS (mg/L)	161,471
TSS (mg/L)	422
TDS (mg/L)	5832
Particle size (mm)	2-8
pH	7-7.4

Table 6. Characterization for Wastewater at Different Stages of the Process

Waste Status	Total Nitrogen mgN/L	Total Alkalinity mgCaCO3/L	Phosphorous mgPO4/L	Sulfate mgSO4/L	Total Ammonia mgNH3/L	pH	COD mgCOD/L	VFA mgCH3COOH/L
Influent	115	_	164.5	134	26	3.5	29125	1747.66
Pre-acidification	112	491	178	434	40.8	4.8	14525.25	5041.66
Effluent	303	691	304.5	131	93.12	7.6	2224	463.5

3.4. IMPROVE PHASE

After identifying and analyzing the factors that are responsible for affecting the AD process, the next process was to recognize the feasible solutions for the failures. The solutions were implemented and checked for defects and for similar results to the designed experiments. As per the results, corrective and preventive measures were taken for significant improvement in the process.

The experiment was designed as a three factorial completely random experiment with the factors being pH, temperature, and organic loading rate. The pH had 12 levels, starting from 3.5 with an increment of 0.2 for every level. The temperature had five levels starting from 30ºC and ended at 40ºC in 2.5ºC increments. The OLR had three levels, namely 2, 4 and 6 COD g/L/day. The response was the amount of biogas produced in a given time period measured in gal/hr.

After creating the data table in JMP, the data was fit into the above model. it was found from the analysis of the variance table that the alpha value was less than 0.05, which means states that with 95% confidence that the response is not all the same and the response is affected by at least one of the factors or by an interaction of two or more factors, as shown in Figure 15.

The results were further analyzed to determine which factor is affects the response and if there is interaction between the factors that affect the response. To find the alpha value of the first factor was examined which is pH, and it was noted that it was less than 0.05, as shown in Figure 16, which means that 95% confidence that pH was influencing the output. Similarly, both the temperature Figure 17 and the OLR Figure 18 also have alpha values less than 0.05, so both factors also affect the output.

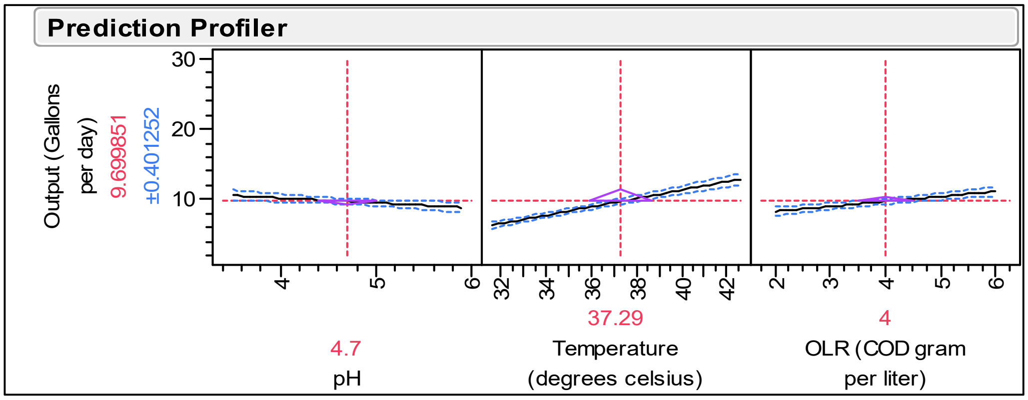

Figure 15. Prediction Profiler

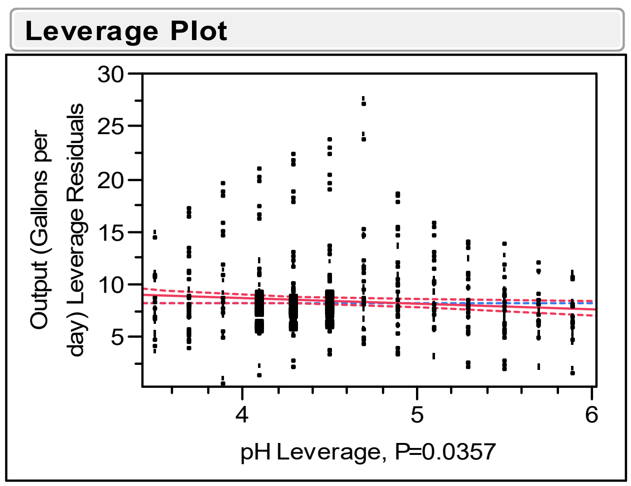

Figure 16. Leverage Plot for pH vs Biogas Production

Further it is also noted that alpha values of all the possible interactions between the factors Figure 19 to Figure 22. The only significant interaction was between the OLR and temperature, as the alpha value was less than 0.05. All the other interactions are noted to be insignificant.

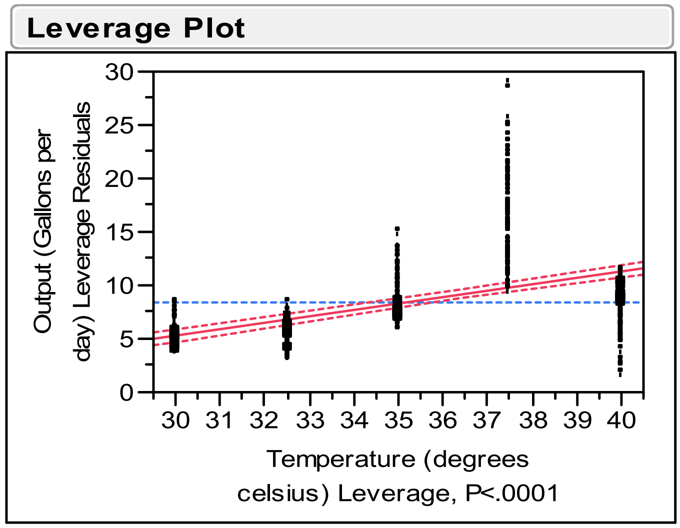

Figure 17. Leverage Plot for Temperature vs Biogas Production

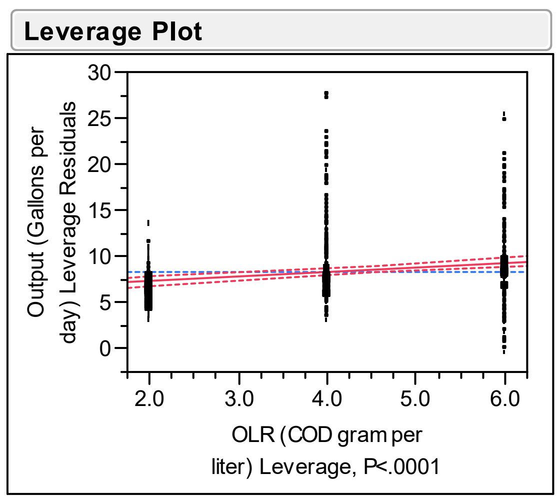

Figure 18. Leverage Plot for OLRs vs Biogas Production

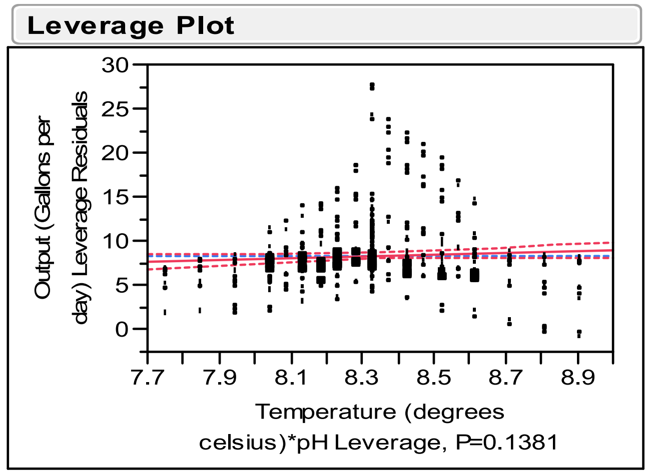

Figure 19. Leverage Plot for Temperature & pH vs Biogas Production

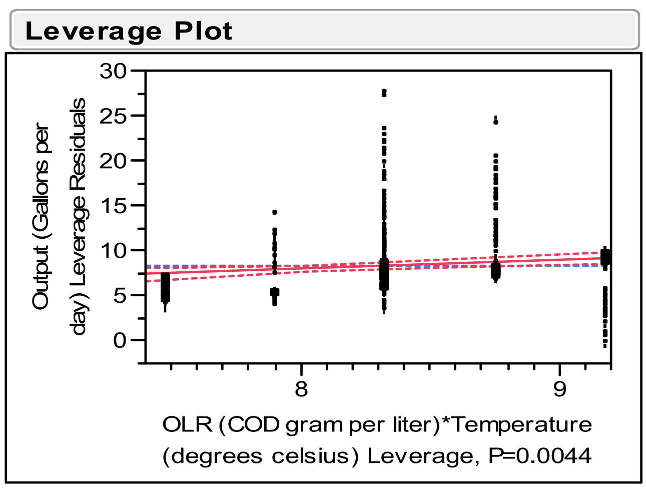

Figure 20. Leverage Plot for Temperature & OLRs vs Biogas Production

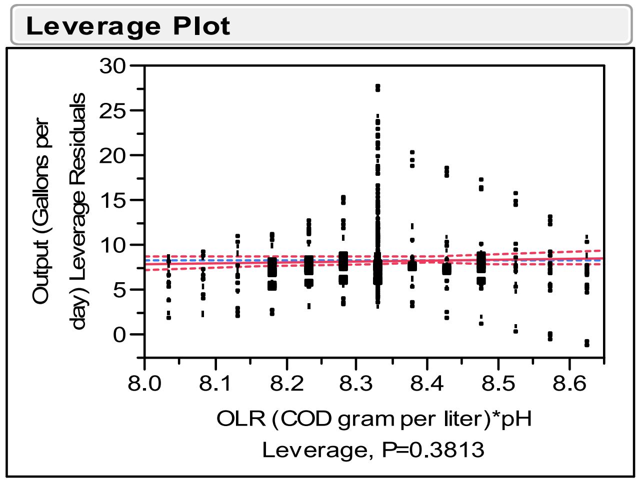

Figure 21. Leverage Plot for pH & OLRs vs Biogas Production

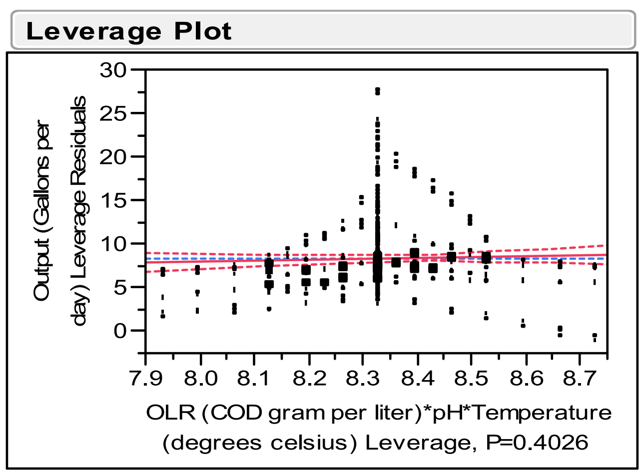

Figure 22. Leverage Plot for Temperature & OLRs & pH vs Biogas Production

3.5. CONTROL PHASE

To achieve the goal of the project and to stabilize the process, the optimum vales of temperature, pH, and organic loading rate were determined. The main factor in determining and influencing the production rate of biogas and the important factor for the increase in the methane composition percentage were found to be the addition of hydrogen. Hydrogen was added to the main reactor at different flowrates and different concentrations of the feed were analyzed Figure 23 to Figure 26. The results found that an increase in the flowrate of the wastewater or an increase in the organic loading rate consume more hydrogen into the system. Figure 23, Figure 24, Figure 25, and Figure 26 show the methane percentage change in the system for different volumetric flow rates at 5%, 10%, 20%, and 30% wastewater concentration, respectively.

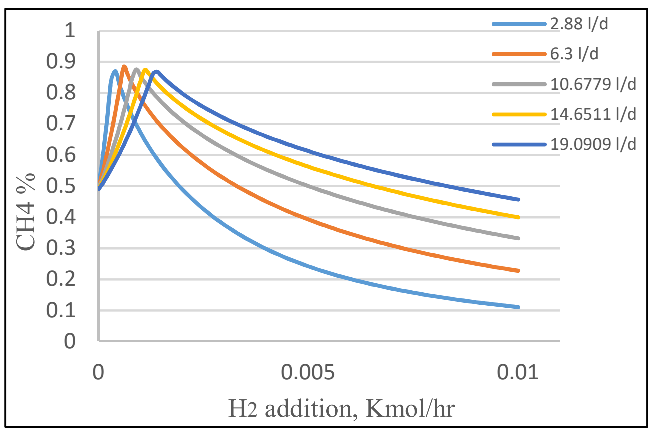

Figure 23. CH_4 Composition with H_2 Introduction to the System at Different Flowrate for 5% Conc.

4. CONCLUSION

Six Sigma implementation for this project has been considered successful as the critical process parameters, and the factors affecting the process were found and implemented. Expanded granular sludge bed reactor, pre-acidification reactor, and hot water system instrumentation has been upgraded as per the study.

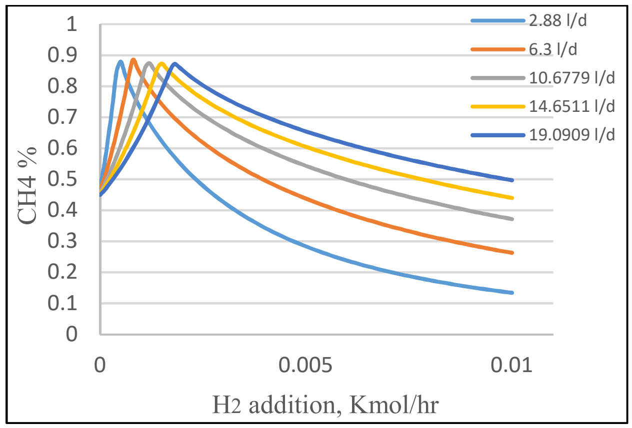

Figure 24. CH4 Composition with H2 Introduction to the System at Different Flowrate for 10% Conc.

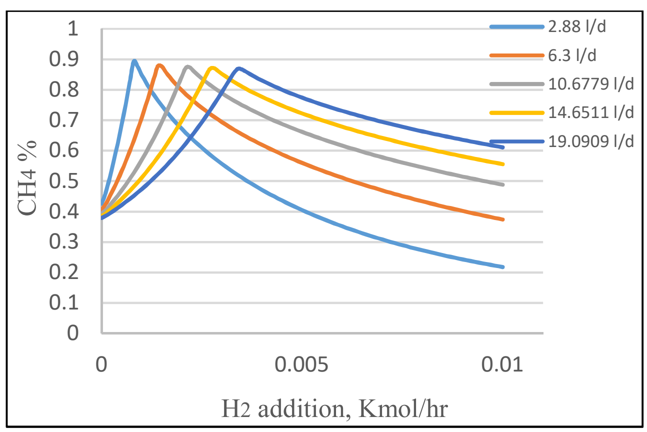

Figure 25. CH4 Composition with H2 Introduction to the System at Different Flowrate for 20% Conc.

Optimal values of pH, T and OLR were obtained for maximum production of biogas. The addition of hydrogen led to an increase in the methane production. Different trials were conducted to control the H_2 flow rate to obtain optimum methane yield at ideal H_2 concentration.

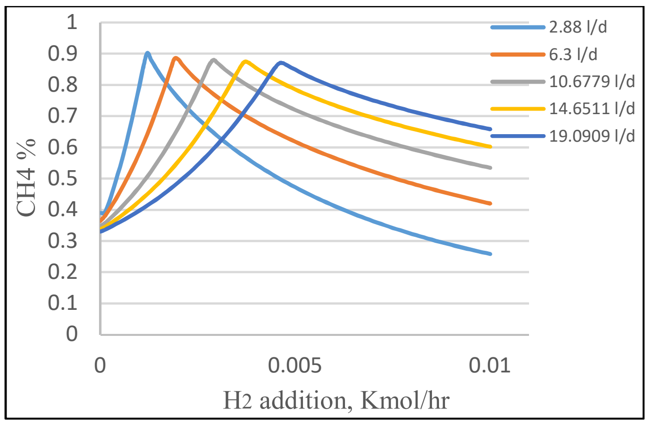

Figure 26. CH4 Composition with H2 Introduction to the System at Different Flowrate for 30% Conc.

The improvement got to observed after introducing Six Sigma tools and concepts in the experiment, the yield was increased from 11 gal to 28 gal in 60 hr which is an impressive 154.5% increase in yield. As shown in the Figure 27, the production rate of biogas increased before and after implementing Six Sigma.

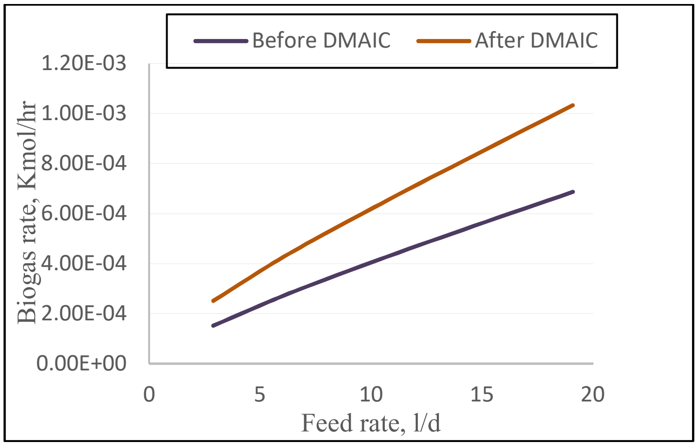

Figure 27. Effect H2 Addition on CH4%

The increase in the percentage composition of methane in the yield was from 50% to 87%. As shown in the Figure 28 between methane composition vs. feed rate, the red line is the percentage composition of methane without the introduction of hydrogen in the experiment, and the blue line shows the methane composition after the introduction of hydrogen.

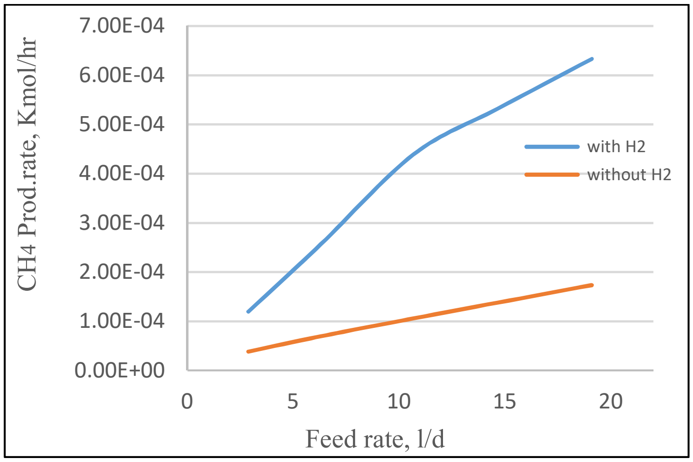

Figure 28. Biogas Production Before and After DMAIC

REFERENCES

[1] "The Inventors of Six Sigma". Archived from original on 2005-11-06. Retrieved 2006-01-29.

[2] Jump up Tennant, Geoff, "SIX SIGMA: SPC and TQM in Manufacturing and Services," Gower Publishing, Ltd. p. 6. 2001.

[3] Gutiérrez H.y De la Vara R, "Control Estadístico de Calidad y Seis Sigma," Mc Graw Hill Interamericana Editores, S.A. de C.V. México, D.F. 2004.

[4] M. Ghorbanian, R. M. Lupitskyy, J. V Satyavolu, and R. E. Berson, "Impact of Hydraulic Retention Time at Constant Organic Loading Rate in a Two-Stage Expanded Granular Sludge Bed Reactor," Environ. Eng. Sci., vol. 31, no. 6, pp. 317–323, 2014.

[5] K. Moriarty, "Feasibility Study of Anaerobic Digestion of Food Waste in St. Bernard, Louisiana," Natl. Renew. Energy Lab., no. January 2013.

[6] S. Montalvo, L. Guerrero, R. Borja, I. Cortes, E. Sanchez, and M. F. Colmenarejo, "Treatment of wastewater from red and tropical fruit wine production by zeolite anaerobic fluidized bed reactor," J. Environ. Sci. Heal. Part B -- Pestic. Food Contam. Agric. Wastes, vol. 43, no. 5, pp. 437–442, 2008.

[7] N. J. Themelis, “Anaerobic Digestion of Biodegradable Organics in Municipal Solid Wastes,” Found. Sch. Eng. Appl. Sci. Columbia Univ., no. May, pp. 1–56, 2002.

[8] H. Al-Rubaye, S. Karambelkar, M. M. Shivashankaraiah, and J. D. Smith, “Process Simulation of Two-Stage Anaerobic Digestion for Methane Production,” Biofuels, vol. 7269, no. April, pp. 1–11, 2017.

[9] L.A. Brown, B.R. Daugherty and V.W. Lowe, Measurement Systems Analysis, third edition, Auto Industry Action Group, 2003.

[10] Acheson J. Duncan, Quality Control and Industrial Statistics, fifth edition, Richard D. Irwin Inc., 1986.

[11] Donald S. Ermer and P.E. Prond, “A Geometrical Analysis of Measurement System Variations,” ASQC Annual Quality Congress Transactions, 1993.

[12] Donald S. Ermer, “Pythagorean Theorem to the Rescue or Reliable Data Is an Important Commodity,” The Standard, ASQ Quality Measurement Division, winter 2000/Spring 2001.

[13] Nancy R. Tague’s The Quality Toolbox, Second Edition, ASQ Quality Press, 2004, pages 236–240.

SECTION

2. CONCLUSION

Brewery wastewater containing high COD concentration was treated with removal efficiency of 90%. By recirculating the effluent into the system, the biogas production was increased to 51.41% and the methane composition was enhanced to 73.24% at 40% recirculation rate under 6 OLR g COD/L/day. Six Sigma methodology was successfully implemented for anaerobic digestion process, resulted in significant improvement in biogas production to 28 gal/day and introduction hydrogen led to increase n methane composition to 87%.

VITA

Manohar Manchenahalli Shivashankaraiah was born in Karnataka (India). Manohar earned his bachelor's degree in Chemical Engineering from Siddaganga Institute of Technology, Tumkur. During this time, he found an opportunity to work under the professor Binayak M. Hedge on catalyst generation and testing. He received his Bachelor of Engineering in June 2013.

Manohar joined the Novo Nordisk Engineering as a process engineer and served for two and half years.

Manohar joined the Missouri University of Science and Technology in January of 2016 for his master's degree program. There, he was given an opportunity to work as a graduate research assistant under Dr. Joseph Smith on three projects. Manohar Manchenahalli Shivashankaraiah graduated with his Master of Science in Chemical Engineering in May of 2018.