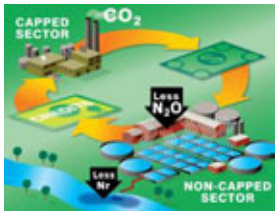
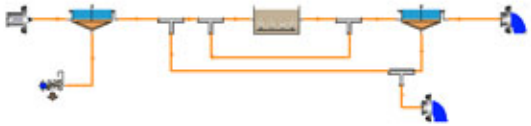
# RESOURCE RECOVERY FROM FECAL SLUDGE

## PILOT AND LAB-SCALE STUDIES AND BIOPROCESS MODELING

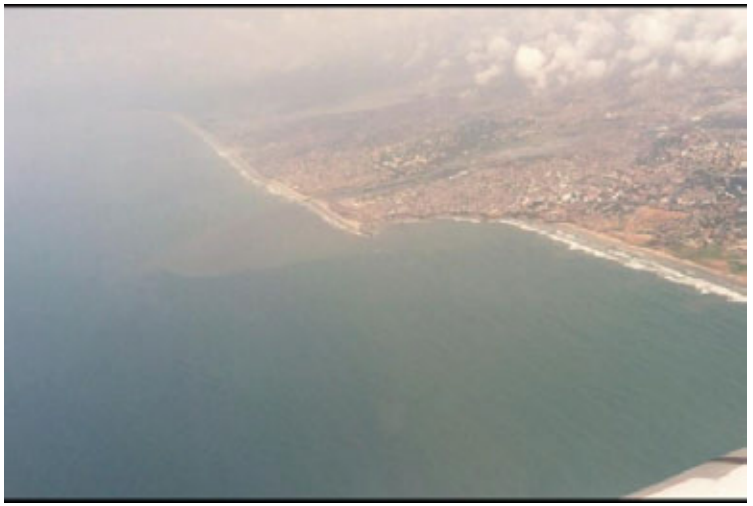
**Kartik Chandran**

Columbia University



**Mainstreaming Citywide Sanitation:**
**CSE, New Delhi, April 4th, 2016**

Lack of adequate sanitation is a global challenge

Often limited by access to reliable energy inputs and chemicals

**Is it possible to link sanitation with higher value chain biofuels and commodity chemicals?**

??

Our approach

Domestic and Food waste

Faecal sludge

Municipal solid waste

Animal by-product waste

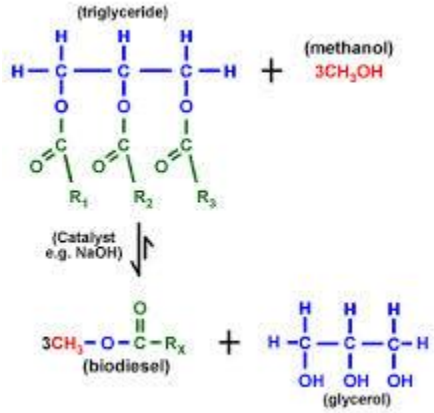
Channel through fermentation platform

Commodity chemicals, lip ids, biodiesel

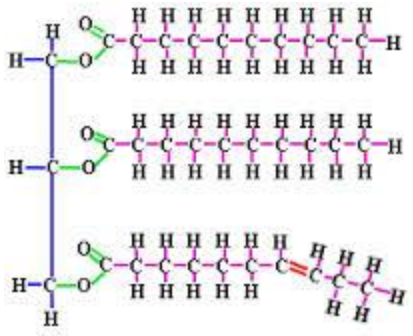
BILL & MELINDA GATES foundation

# Fecal sludge to biodiesel

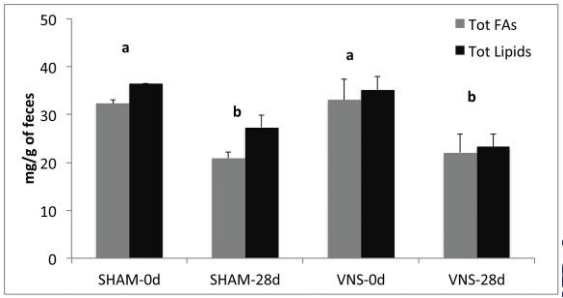
- Biodiesel

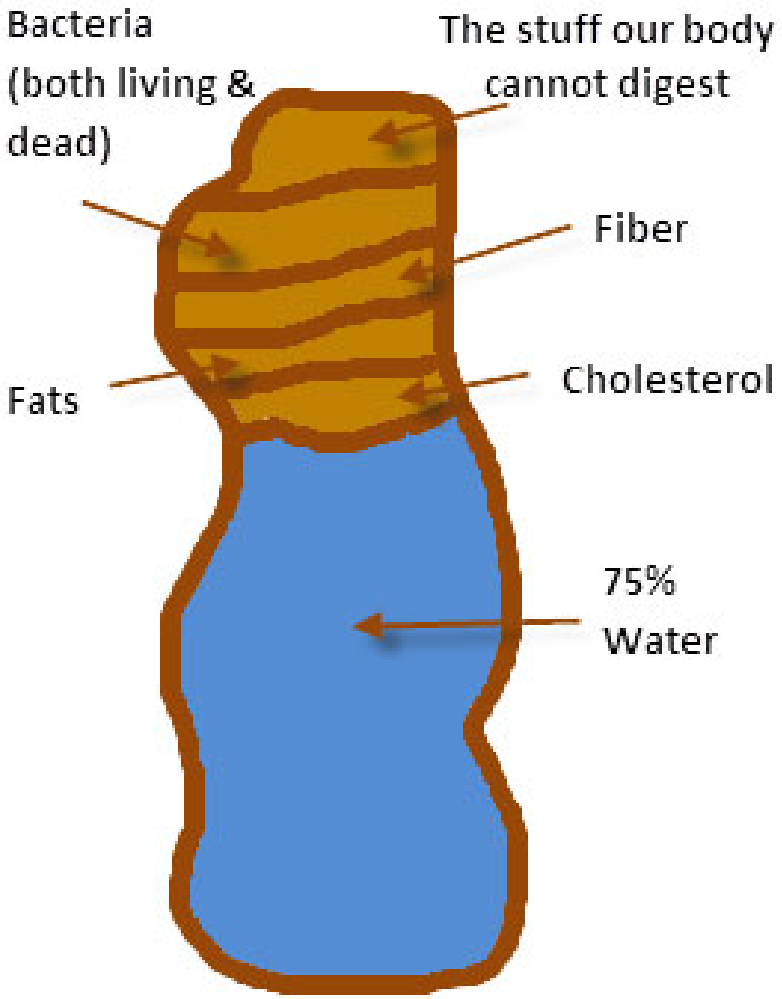



- Lipids

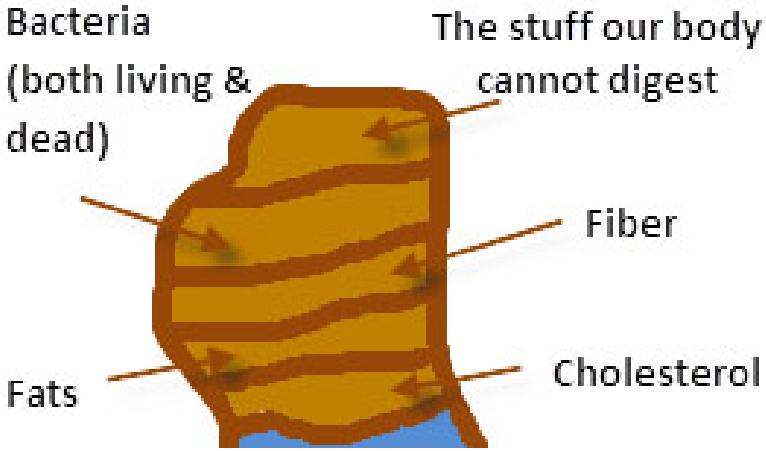


- Lipids in feces

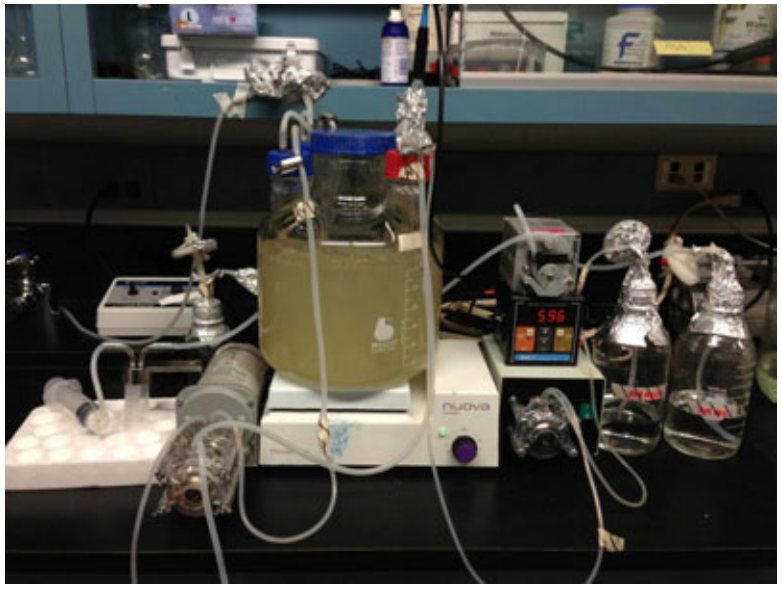


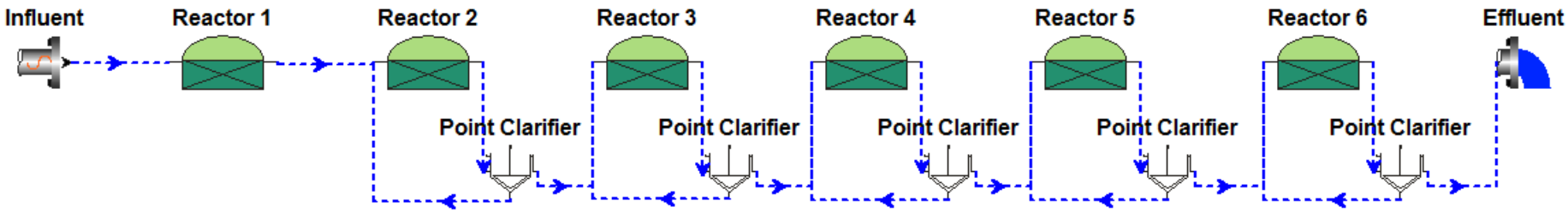





- **Biodiesel process agnostic to 'waste' stream?**

# Faecal Sludge to Biodiesel Project

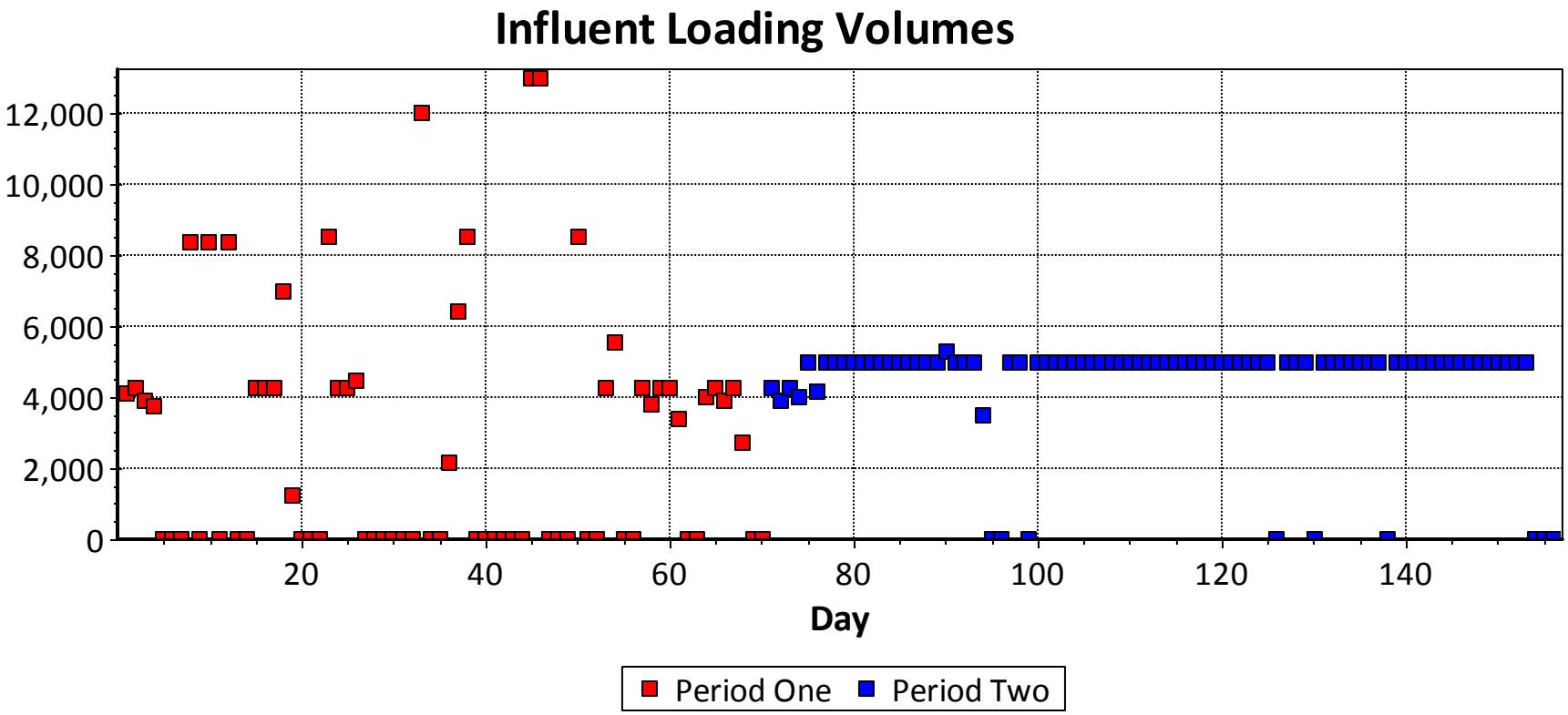




# Local project lab

# Practical Issues

## Realistic Loading Conditions




How does influent variability impact performance?

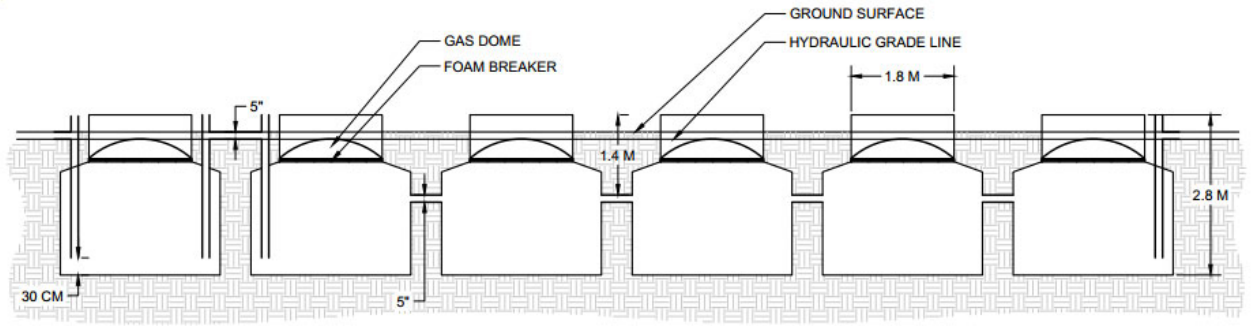
# FS Characterization – Extreme Variability

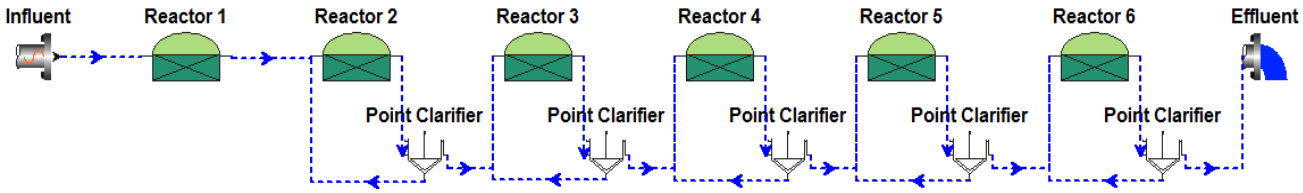
| | Average ± SD |
|---|---|
| Total COD (mg/L) | 22,951 ± 19,499 |
| Total VFA (mgCOD/L) | 1,417 ± 1,074 |
| pH | 8.01 ± 0.27 |
| Total Suspended Solids (mg/L) | 15,663 ± 16,867 |
| Volatile Suspended Solids (mg/L) | 12,617 ± 13,328 |

**Need to characterize beyond conventional parameters**

# Approach: Comprehensive Pilot Operations with Modelling Analysis

Pilot Scale Field Operations → Process Modeling



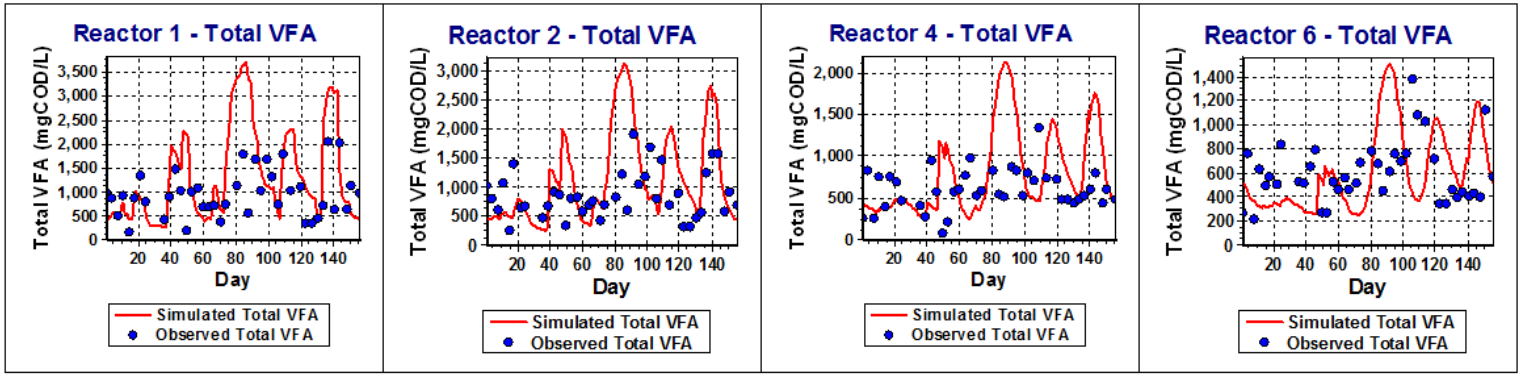
# Process Modelling Approach

**Purpose: Identify key characteristics of FS and FS fermentation and digestion (limit model adjustments)**

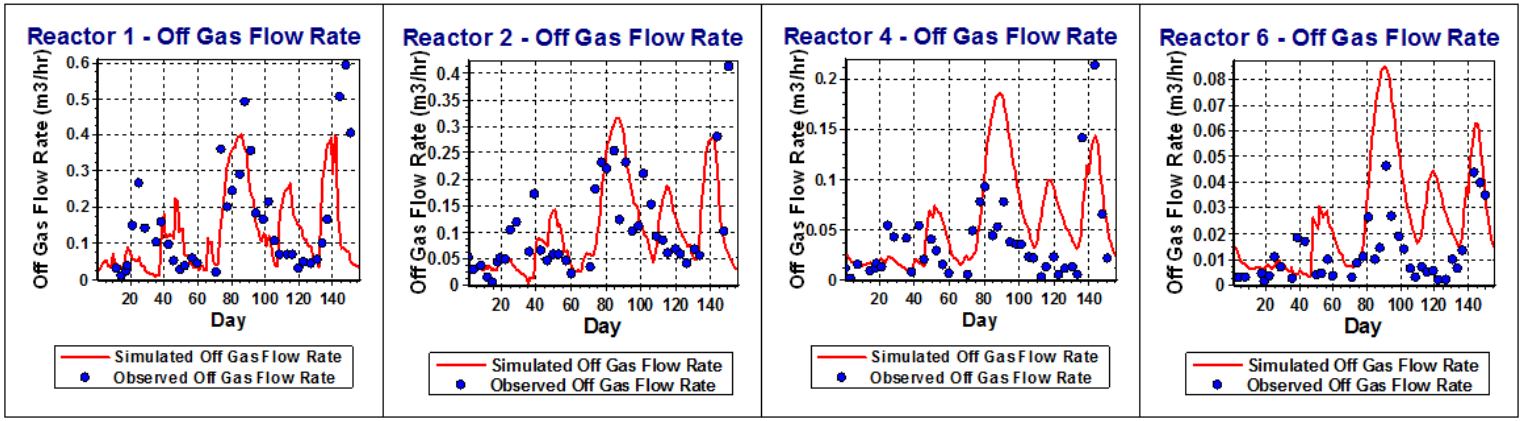
**Evaluated through calibration:**

- **COD fractionation (readily bio, unbiodegradable, etc.)**
- **Influent microbial concentrations**
- **Reaction rates (hydrolysis, acidogenesis, acetogenesis, and methanogenesis)**
- **Solids distribution (unmixed system)**

# Calibrated profiles for VFA and CH4




- VFA highest in influent
- Decreases throughout reactors



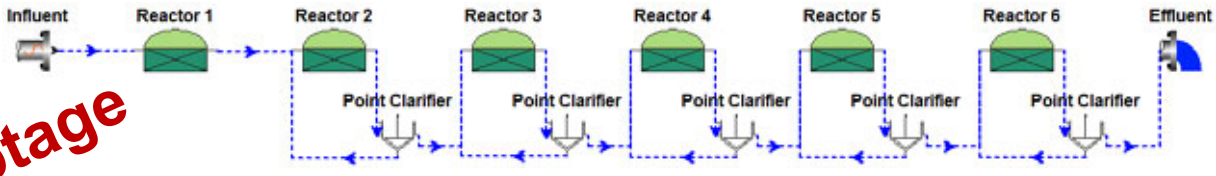

- Maximum gas production in Reactor 1
- Decreases throughout reactors

# FS Fermentation and Digestion Model

Pit Latrines

Public

Private Septage




$CH_4$

???

VFA

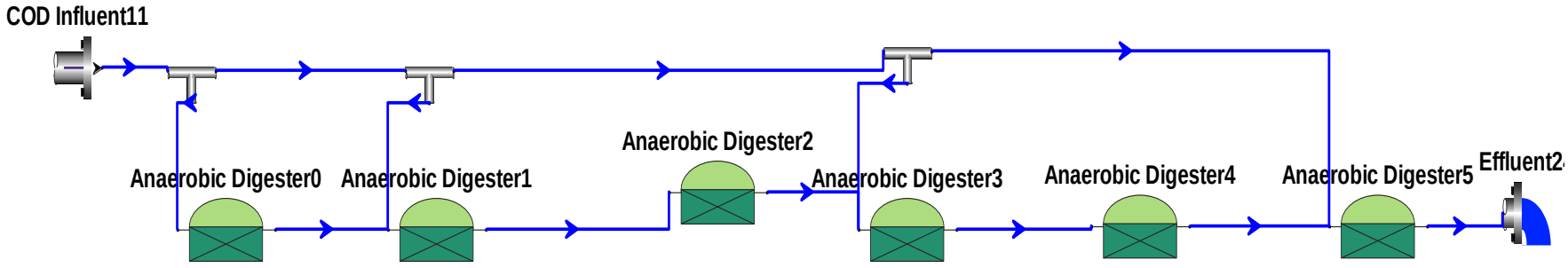
This model will be released and shared publicly

# Practical issues

## Overcoming mixing limitations and increasing process flexibility

- Pumps in R1, R2, R4 and R6

- Step-feeding



# Maximizing lipid synthesis

**Organic waste**

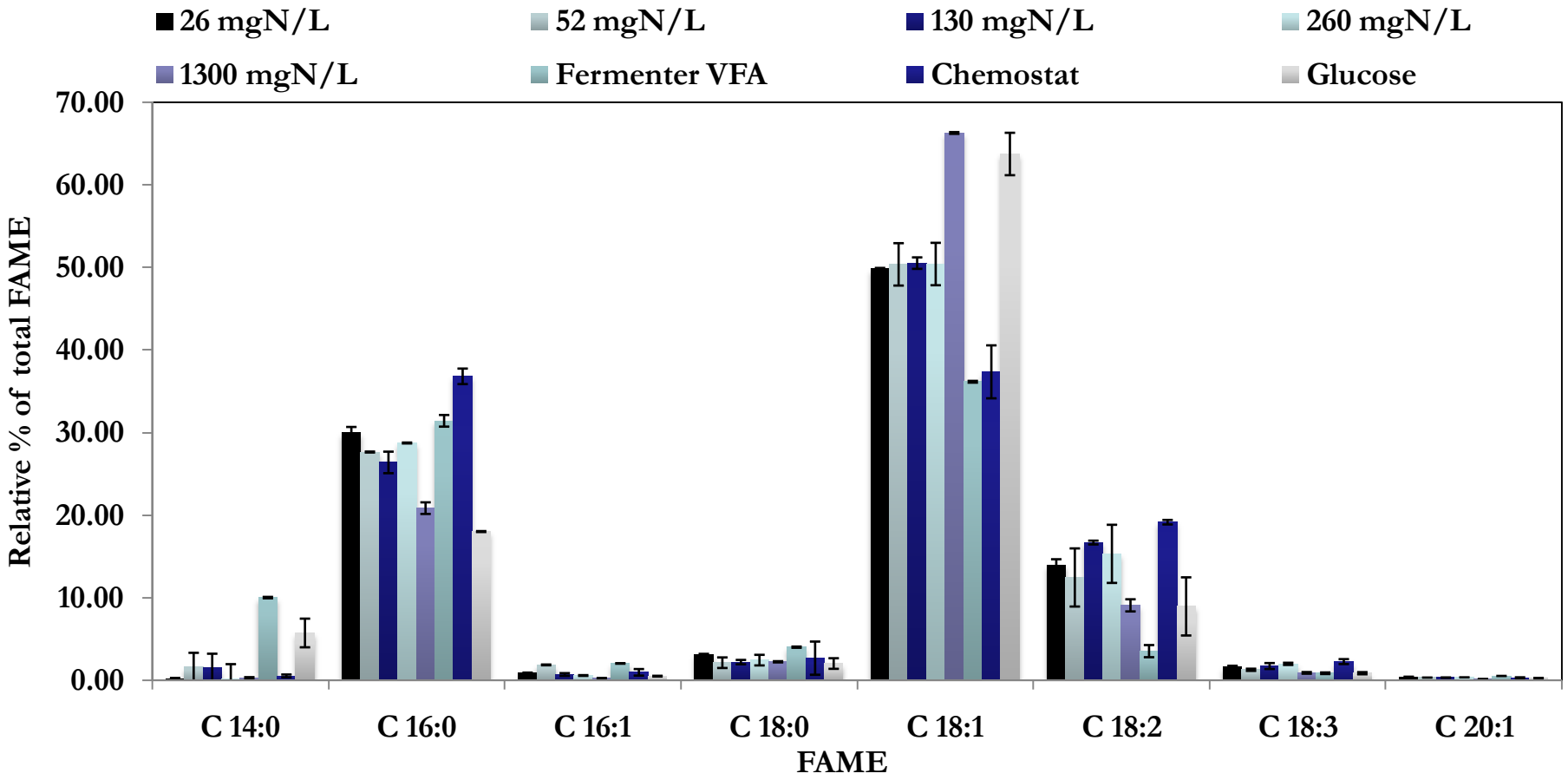
**Anaerobic fermentation to produce volatile fatty acids (VFA)**

**Convert VFA to lipids**

**Harvest and extract lipids**

**Convert lipids to biodiesel**

# FATTY ACID COMPOSITION




- Major fatty acids accumulated by *C. albidus* were predominantly palmitic acid (C16:0), oleic acid (C18:1), and linoleic acid (C18:2)

- Similar to soybean oil and jatropha oil, which are used as feedstock for biodiesel production in the US and the EU

# Other options for resource recovery

## Biogas to chemicals

ENVIRONMENTAL Science & Technology

Article

pubs.acs.org/est

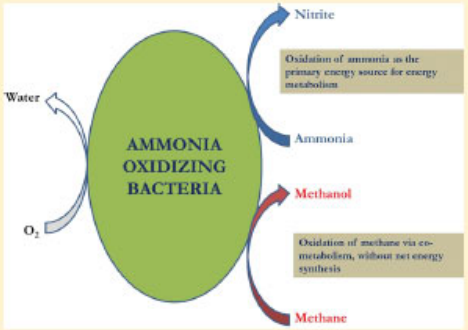
### High-Rate, High-Yield Production of Methanol by Ammonia-Oxidizing Bacteria

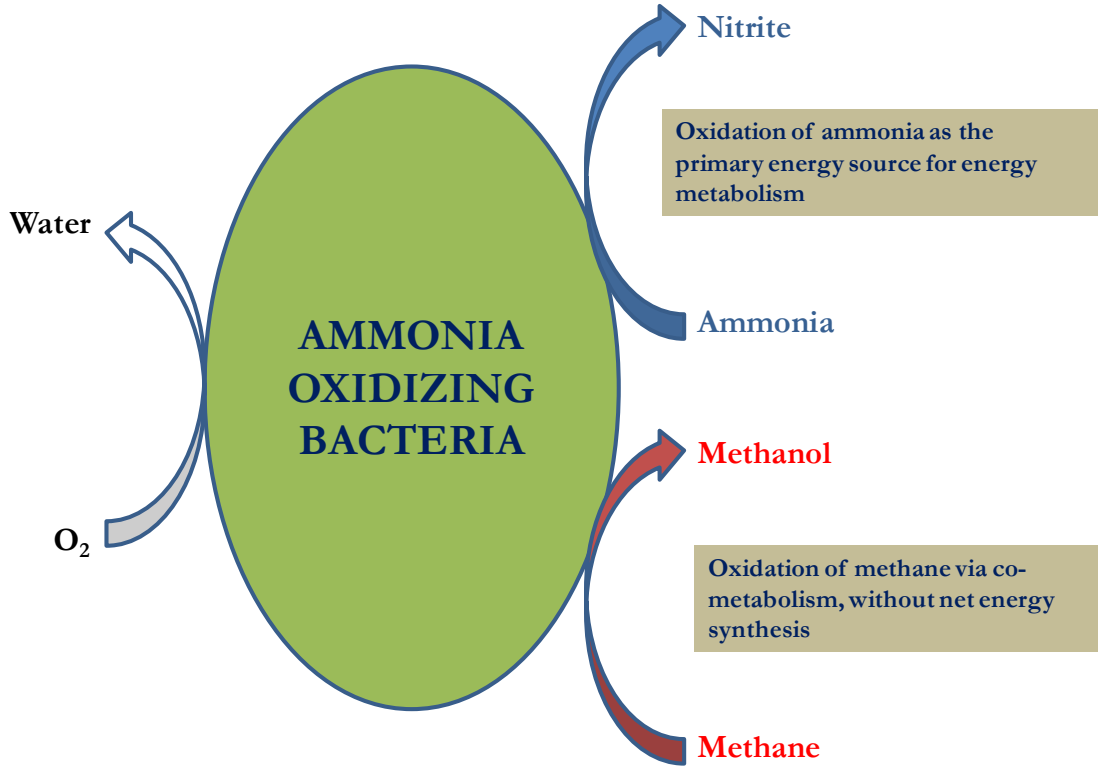
Edris Taher and Kartik Chandran*

Department of Earth and Environmental Engineering, Columbia University, 500 West 120th Street, New York, New York 10027, United States

Supporting Information

**ABSTRACT:** The overall goal of this study was to develop an appropriate biological process for achieving autotrophic conversion of methane ($CH_4$) to methanol ($CH_3OH$). In this study, we employed ammonia-oxidizing bacteria (AOB) to selectively and partially oxidize $CH_4$ to $CH_3OH$. In fed-batch reactors using mixed nitrifying enrichment cultures from a continuous bioreactor, up to 59.89 ± 1.12 mg COD/L of $CH_3OH$ was produced within an incubation time of 7 h, which is approximately ten times the yield obtained previously using pure cultures of *Nitrosomonas europaea*. The maximum specific rate of $CH_4$ to $CH_3OH$ conversion obtained during this study was 0.82 mg $CH_3OH$ COD/mg AOB biomass COD-d, which is 1.5 times the highest value reported with pure cultures. Notwithstanding these positive results, $CH_4$ oxidation to $CH_3OH$ by AOB was inhibited by $NH_3$ (the primary substrate for the oxidative enzyme, ammonia monooxygenase, AMO) as well as the product, $CH_3OH$, itself. Further, oxidation of $CH_4$ to $CH_3OH$ by AOB was also limited by reducing equivalents supply, which could be overcome by externally supplying hydroxylamine ($NH_2OH$) as an electron donor. Therefore, a potential optimum design for promoting $CH_4$ to $CH_3OH$ oxidation by AOB could involve supplying $NH_3$ (needed to maintain AMO activity) uncoupled from the supply of $NH_2OH$ and $CH_4$. Partial oxidation of $CH_4$-containing gases to $CH_3OH$ by AOB represents an attractive platform for the conversion of a *gaseous* mixture to an *aqueous* compound, which could be used as a commodity chemical. Alternately, the nitrate and $CH_3$ OH thus produced could be channeled to a downstream anoxic zone in a biological nitrogen removal process to effect nitrate reduction to $N_2$, using an *internally* produced organic electron donor.






- Concomitant oxidation of $CH_4$ and $CO_2$ fixation
  - Digester gas contains $CO_2$
  - Foulant for chemical catalyst; but a food source for AOB
  - Moisture- not really an issue
- Prospect of combining C &N cycles

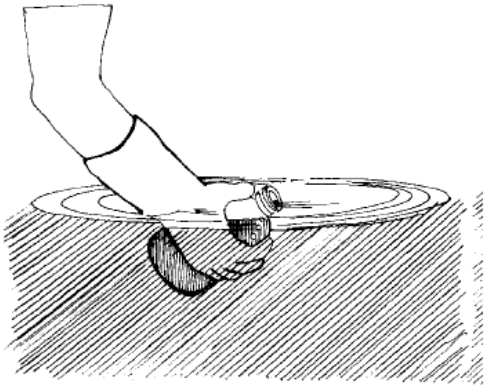
# Benchmarking Data Collection

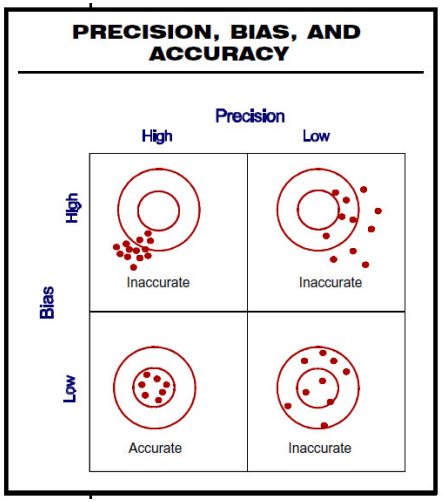
## Quality Assurance Project Plan and Data Quality Indicators

# Data Quality Indicators

| Quality Control Indicator | Sample Type | Frequency | Parameter | Acceptance criterion (%) |
|---|---|---|---|---|
| Precision | Check standard | 1 per 10 | RPD | ± 25 |
| | Field Duplicate | 1 per 10 | RPD | ± 25 |
| | Lab Duplicate | 1 per 10 | RPD | ± 25 |
| Accuracy | Known spike | 1 per 20 | % recovery | 75 – 125 |
| Completeness | All | Annual | % missing | To be determined |
| Performance audit | Known sample | ≥4/Year | RPD | ± 10 |

# Data Quality indicators






## ACCURACY

Attendance at QC training sessions is required for Volunteer Creek monitors. In the field, monitors use a Jones Wide-Range pH Kit, which covers a full range of expected pH values. During a recent training session, the monitors recorded the following results when testing a pH standard buffer solution of 7.0 units.

| | | | |
|---|---|---|---|
| 7.5 | 7.2 | 6.5 | 7.0 |
| 7.4 | 6.8 | 7.2 | 7.4 |
| 6.7 | 7.3 | 6.8 | 7.2 |

*Accuracy = average value - true value*

The average of these measurements is equal to 7.08 units. Since we know that the reference or "true" value is 7.0 units, the difference between the average pH value is off or biased by + 0.08 units. This level of accuracy is satisfactory for the data quality objectives of the project.

# Data Quality indicators

## STANDARD DEVIATION

The Volunteer Creek Monitoring Project wants to determine the precision of its temperature assessment procedure. They have taken 4 replicate samples:

Replicate 1 ($X_1$) = 21.1$^0$ C
Replicate 2 ($X_2$) = 21.1$^0$ C
Replicate 3 ($X_3$) = 20.5$^0$ C
Replicate 4 ($X_4$) = 20.0$^0$ C

To determine the **Standard Deviation (s)**, use the following formula:

$$s = \sqrt{\sum_{i=1}^{n} \frac{(X_1 - \overline{X})^2}{n-1}}$$

where $x_i$ = measured value of the replicate, $\bar{x}$ = mean of replicate measurements, n = number of replicates, $\sum$ = the sum of the calculations for each measurement value--in this case, $X_1$ through $X_4$

First, figure out the mean, or average of the sample measurements. Mean = ($X_1$ + $X_2$ + $X_3$ + $X_4$) ÷ 4. In this example, the mean is equal to 20.68$^0$ C.

Then, for each sample measurement ($X_1$ through $X_4$), calculate the next part of the formula. For $X_1$ and $X_2$, the calculation would look like this:

$$\frac{(21.1 - 20.68)^2}{4\text{-}1} = \frac{(-0.42)^2}{3} = \frac{0.1764}{3} = 0.0588$$

For $X_3$ the calculation would be 0.0108; and for $X_4$ it would be 0.1541

Finally, add together the calculations for each measurement and find the square root of the sum: 0.0588 + 0.0588 + 0.0108 + 0.1541 = 0.2825. The square root of 0.2825 is 0.5315.

So, the standard deviation for temperature is 0.532 (rounded off).

## RELATIVE STANDARD DEVIATION

If we use the same replicate measurements as above in the standard deviation example, we can determine the **Relative Standard Deviation (RSD)**, or coefficient of variation, using the following formula:

$$RSD = \frac{s}{\overline{X}} \times 100$$

where s = standard deviation and $\bar{x}$ = mean of replicate samples.

We know s = 0.5315 and that $\bar{x}$ = 20.68. So, the RSD = 2.57. This means that our measurements deviate by about 2.57%.

## RELATIVE PERCENT DIFFERENCE

If the Volunteer Creek project had only two replicates (21.1$^0$ C and 20.5$^0$ C) they would use **Relative Percent Difference (RPD)** to determine precision.

$$RPD = \frac{(X_1 - X_2) \times 100}{(X_1 + X_2) \div 2}$$

where $X_1$ = the larger of the two values and $X_2$ = the smaller of the two values. In this example, $X_1$= 21.1$^0$ and $X_2$ = 20.5$^0$.

$$RPD = \frac{(21.1\text{-}20.5) \times 100}{(21.1+20.5) \div 2} = \frac{60.00}{20.8} = 2.88$$

So, in this example, the RPD between our sample measurements is 2.88%.

# Data Quality indicators

## COMPLETENESS

The Volunteer Creek Monitoring project planned to collect 20 samples, but because of volunteer illness and a severe storm, only 17 samples were actually collected. Furthermore, of these, two samples were judged invalid because too much time elapsed between sample collection and lab analysis. Thus, of the 20 samples planned, only 15 were judged valid.

The following formula is used to determine **Percent Completeness (%C).**

$$\%C = \frac{v}{T} x 100$$

where v = the number of planned measurements judged valid and T = the total number of measurements.

In this example, v = 15 and T = 20. In this case, percent completeness would be 75 percent. Is this enough information to be useful?

## Method detection limit

$$S^2 = \frac{1}{n-1}\left[\sum_{i=1}^{n} X_i^2 - \frac{\left(\sum_{i=1}^{n} X_i\right)^2}{n}\right] \quad (1)$$

$$S = \sqrt{S^2} \quad (2)$$

$$MDL = t_{(n-1,\alpha=0.01)} * S \quad (3)$$

# Contact information

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# Acknowledgements

**The Bill & Melinda Gates foundation**

**Water Environment Research Foundation Paul Busch Award**

**National Science Foundation**

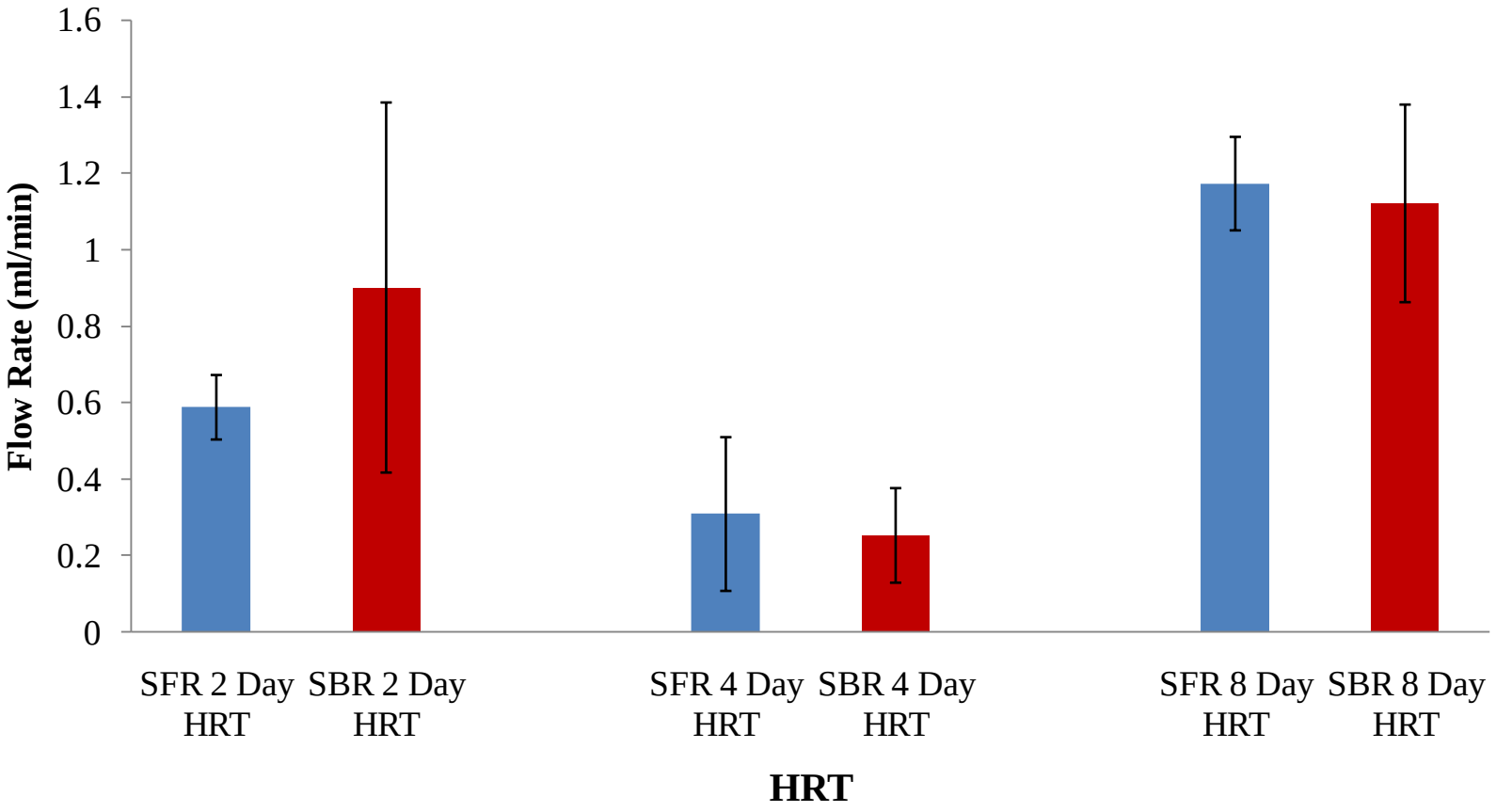
**FS2BD project team**

# Overcoming mixing limitations and increasing process flexibility

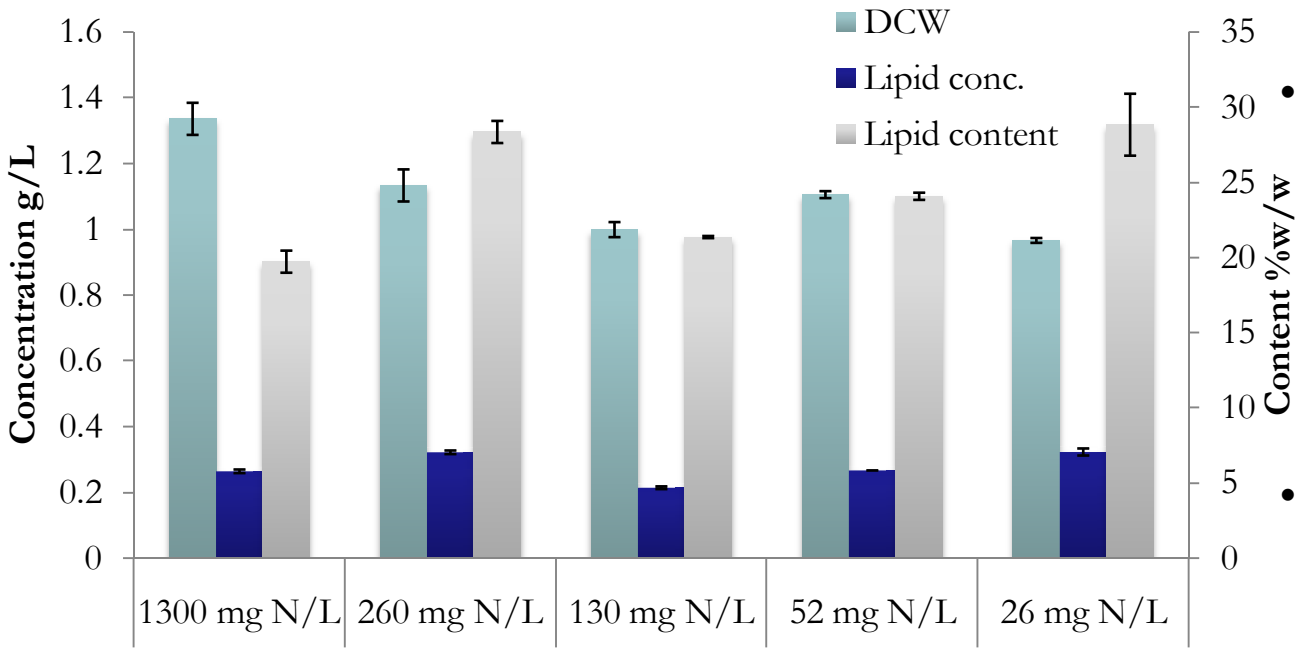
## Step-feed anaerobic digestion

- Reactor configuration
  - V=6L
  - Sequencing batch
  - Step-feed
- Reactor operation
  - HRT: 2-8 d
  - T=37°C
  - pH =7 ± 0.25

# Methane Flow Rate

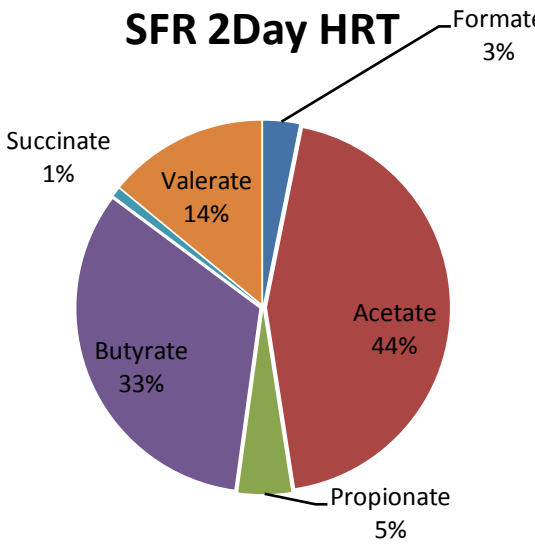


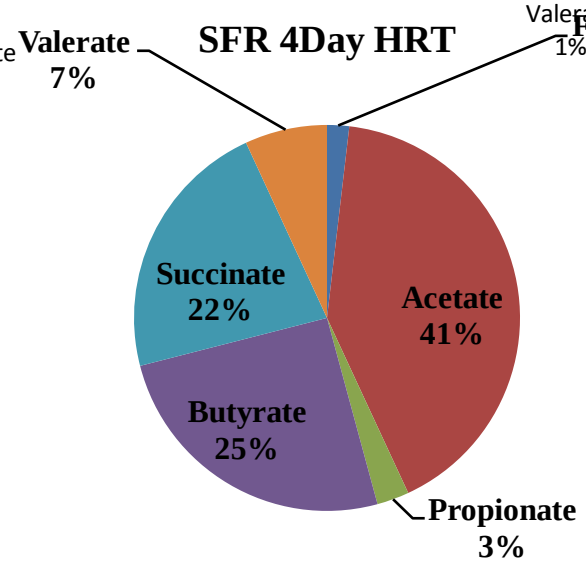
# EFFECT OF NITROGEN CONCENTRATION

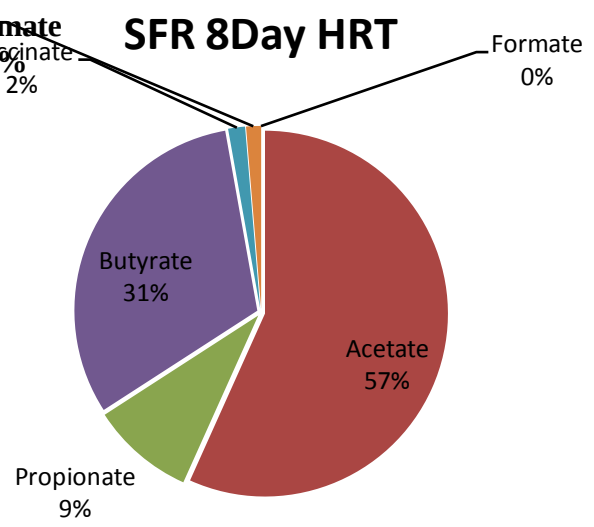


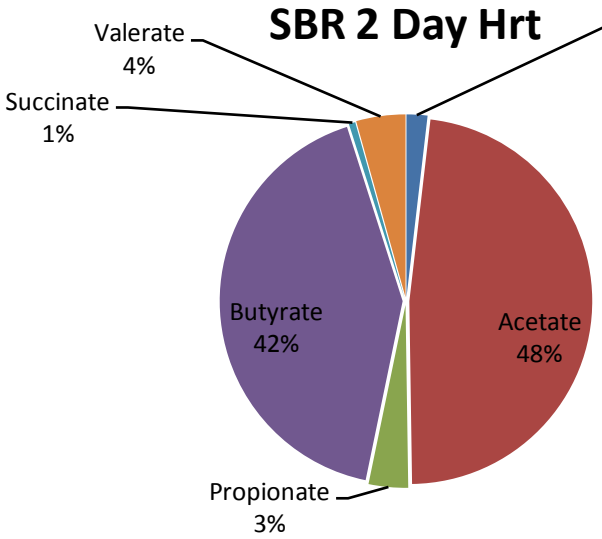

| $NH_3$-N (mg/L) | Biomass (g/L) | $\mu_m$ ($h^{-1}$) | Lipid content | $Y_{L/\Delta COD}$ (mg/g) |
|---|---|---|---|---|
| 1300 | 1.335 | 0.0412 | 19.70% | 44.10 |
| 260 | 1.133 | 0.0425 | 27.80% | 52.35 |
| 130 | 0.998 | 0.0397 | 21.41% | 32.12 |
| 52 | 1.105 | 0.0355 | 24.22% | 40.80 |
| 26 | 0.935 | 0.023 | 28.81% | 41.22 |

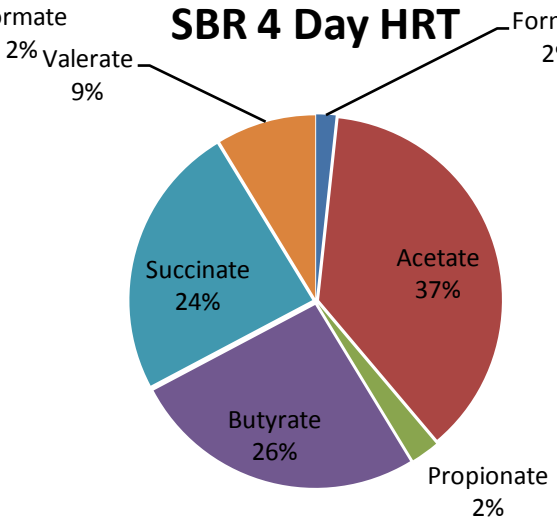
- **Nitrogen limitation was imposed by testing five different initial nitrogen concentration with initial VFA at 5000 mg/L.**
- **The lipid content increased to 28.81% under nitrogen limiting conditions from 19.7%, when excess nitrogen was available.**
- **Increase in C/N ratio under nitrogen limiting conditions ($NH_3$-N ≤ 260 mg/L) did not have an effect on the biomass yield or the intracellular lipid content of *C. albidus***

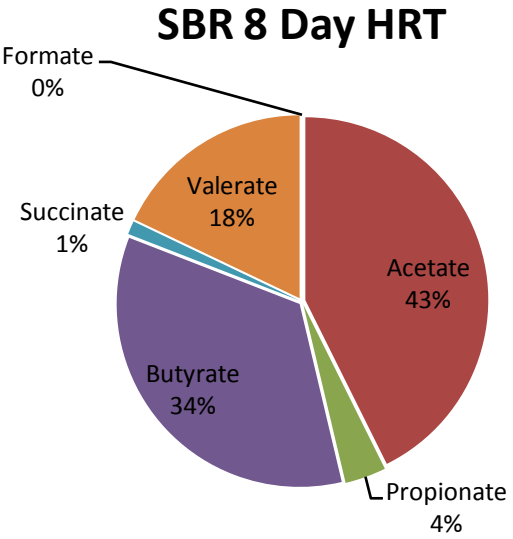

SFR 2Day HRT
Formate 3%
Succinate 1%
Valerate 14%
Acetate 44%
Butyrate 33%
Propionate 5%


SFR 4Day HRT
Valerate 7%
Succinate 22%
Acetate 41%
Butyrate 25%
Propionate 3%


SFR 8Day HRT
Valerate 1%
Formate 2%
Succinate 2%
Formate 0%
Butyrate 31%
Acetate 57%
Propionate 9%

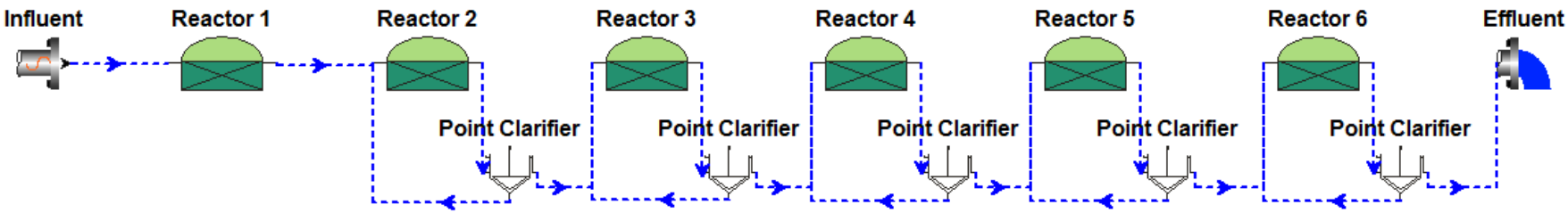

SBR 2 Day Hrt
Valerate 4%
Formate 2%
Succinate 1%
Butyrate 42%
Acetate 48%
Propionate 3%


SBR 4 Day HRT
Formate 2%
Valerate 9%
Succinate 24%
Acetate 37%
Butyrate 26%
Propionate 2%


SBR 8 Day HRT
Formate 0%
Valerate 18%
Succinate 1%
Acetate 43%
Butyrate 34%
Propionate 4%

# Final Model Configuration

| | Biowin Default | Final Model |
|---|---|---|
| Readily Biodegradable (g/g COD) | 0.27 | 0.09 |
| Soluble Unbiodegradable (g/g COD) | 0.08 | 0.09 |
| Particulate Unbiodegradable (g/g COD) | 0.08 | 0.47 |
| Ordinary Heterotrophic Organisms (g/g COD) | 0.01 | 0.05 |
| Acetoclastic Methanogens (g/g COD) | 0.00001 | 0.015 |
| Acetoclastic Methanogenesis Rate (1/day) | 0.3 | 0.1 |
| $CO_2$ Yield (moles $CO_2$/moles acetate) | 0.7 | 1.2 |



# Operation and Process Analysis of Faecal Sludge Anaerobic Fermentation and Digestion

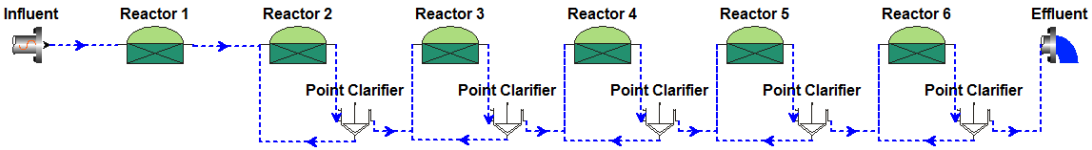

Justin Shih

Ato Fanyin-Martin, Edris Taher, Kartik Chandran

Columbia University

Chandran Lab

USA

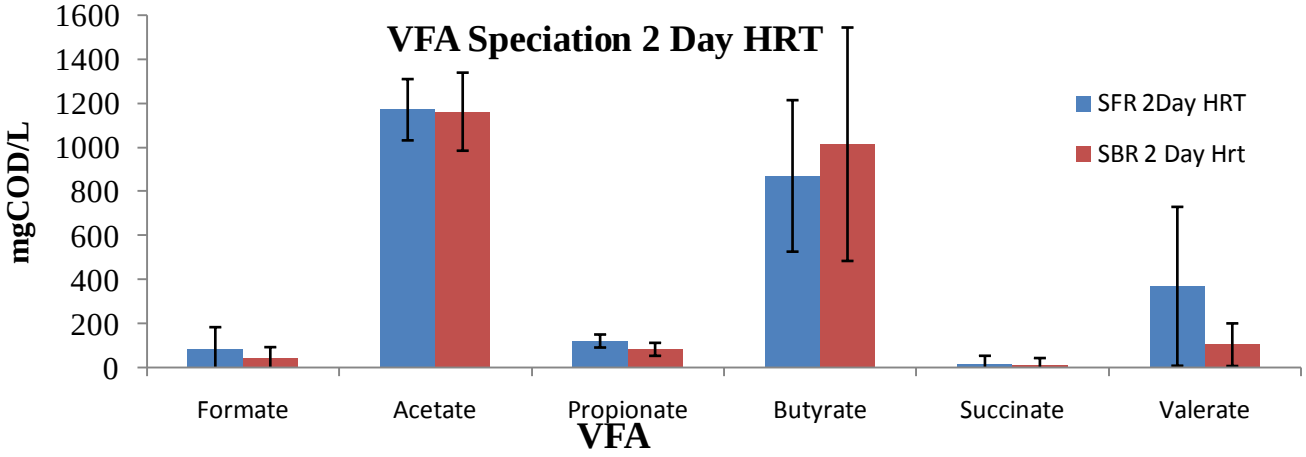

# Conclusions

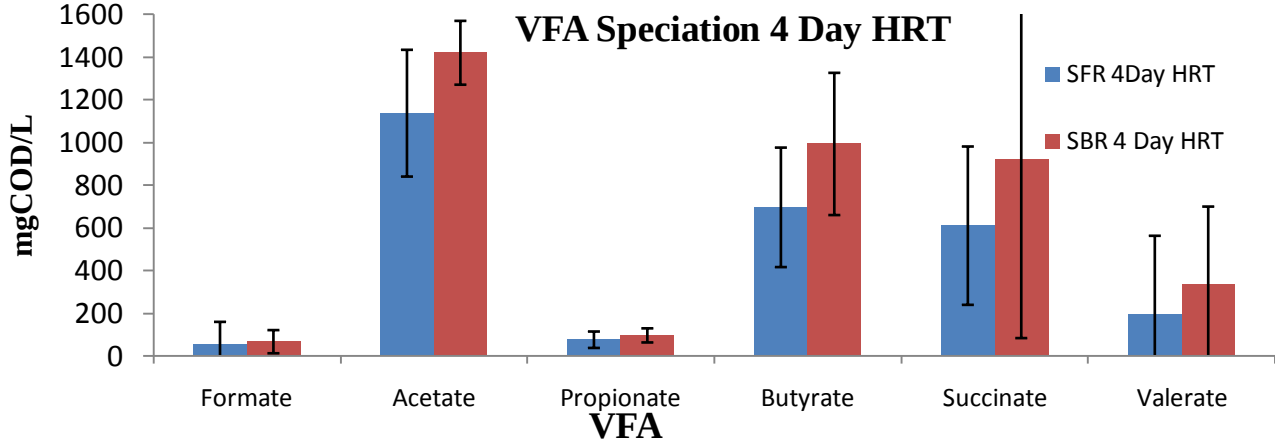
- **HRT affects VFA production and methane flowrate.**
- **VFA was highest at 8 day HRT**
- **They were however similar across all HRT's in the SBR but differed across HRTs in the SFR.**
- **Methane flowrate increased with an increase in HRT**
- **VFA Speciation follows a similar trend across HRT's with differences in yield.**
- **SFR and SBR are similar in a lot of respect across the HRT's**

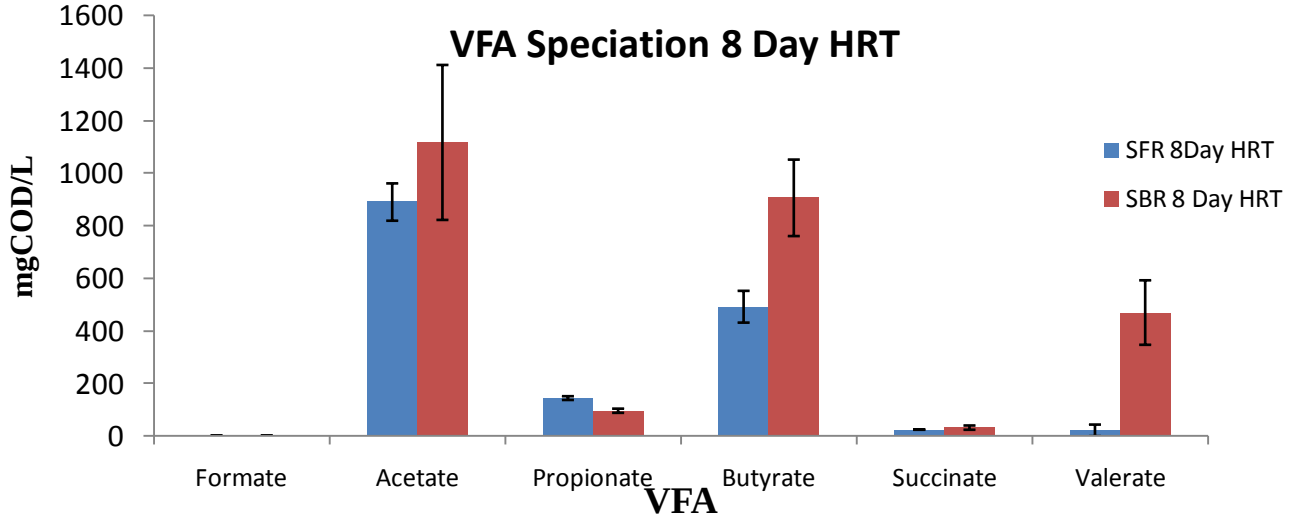
# Results and Discussion

- **Hypothesis**
  - **HRT**
  - **Operational Mode**

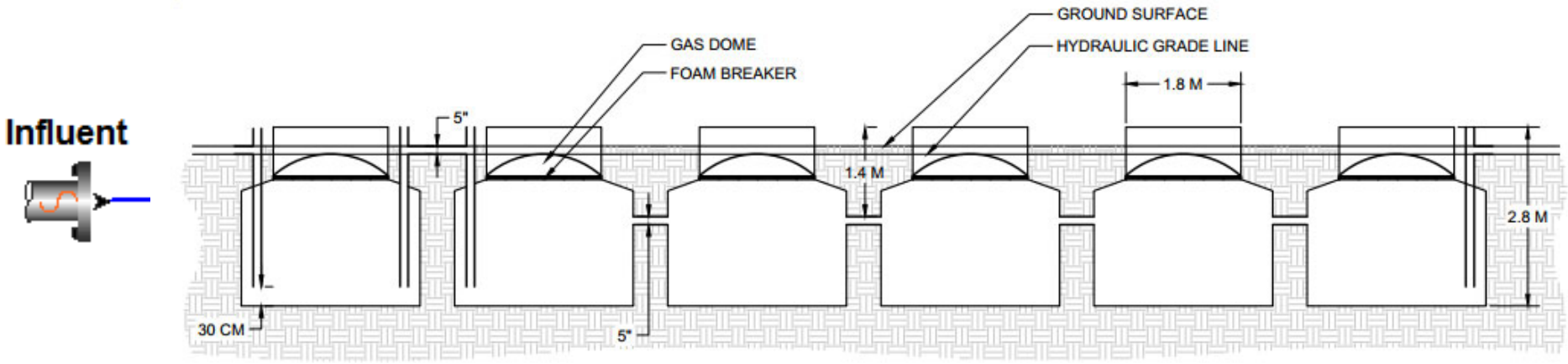
- **Results:**
  - **Liq phase**
    - **Hydrolysis, VFA speciation and VFA yield**
  - **Gas phase**
    - **Flow rate**

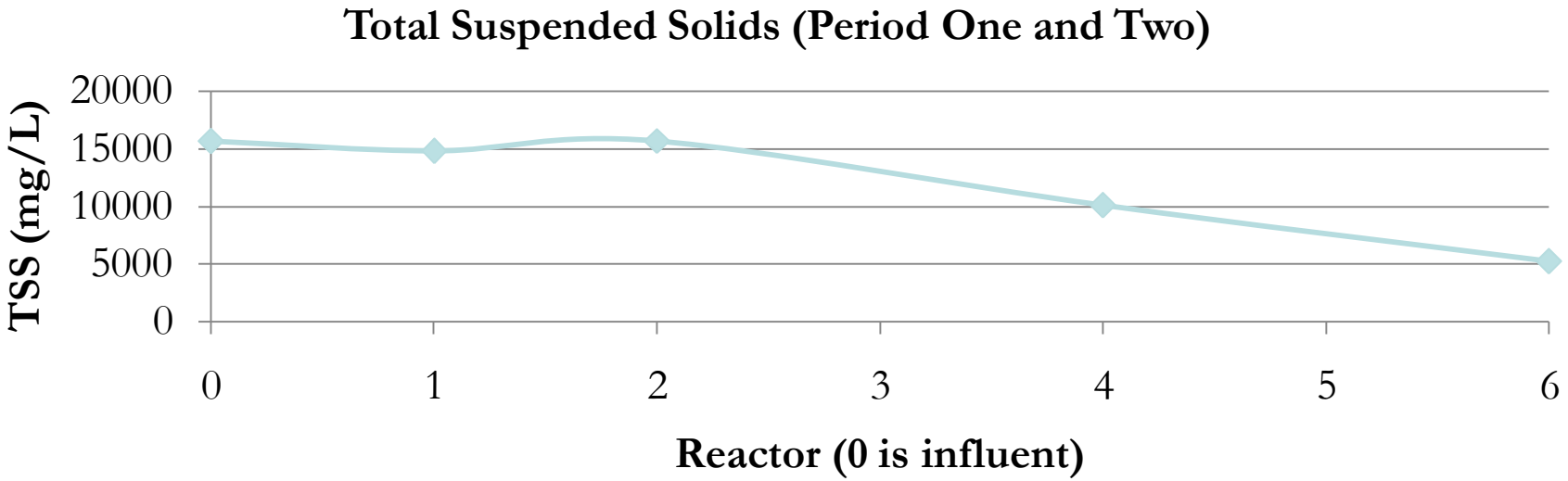

VFA Speciation 2 Day HRT
mgCOD/L
1600
1400
1200
1000
800
600
400
200
0
SFR 2Day HRT
SBR 2 Day Hrt
Formate
Acetate
Propionate
Butyrate
Succinate
Valerate
VFA


VFA Speciation 4 Day HRT
mgCOD/L
1600
1400
1200
1000
800
600
400
200
0
SFR 4Day HRT
SBR 4 Day HRT
Formate
Acetate
Propionate
Butyrate
Succinate
Valerate
VFA


VFA Speciation 8 Day HRT
mgCOD/L
1600
1400
1200
1000
800
600
400
200
0
SFR 8Day HRT
SBR 8 Day HRT
Formate
Acetate
Propionate
Butyrate
Succinate
Valerate
VFA

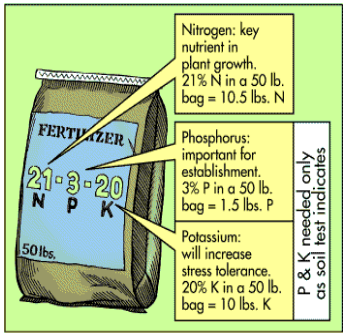
# Settling Distribution in Reactors




**Need to normalize for solids distribution to evaluate gas production efficiency.**

# Conversion of fecal sludge into chemicals

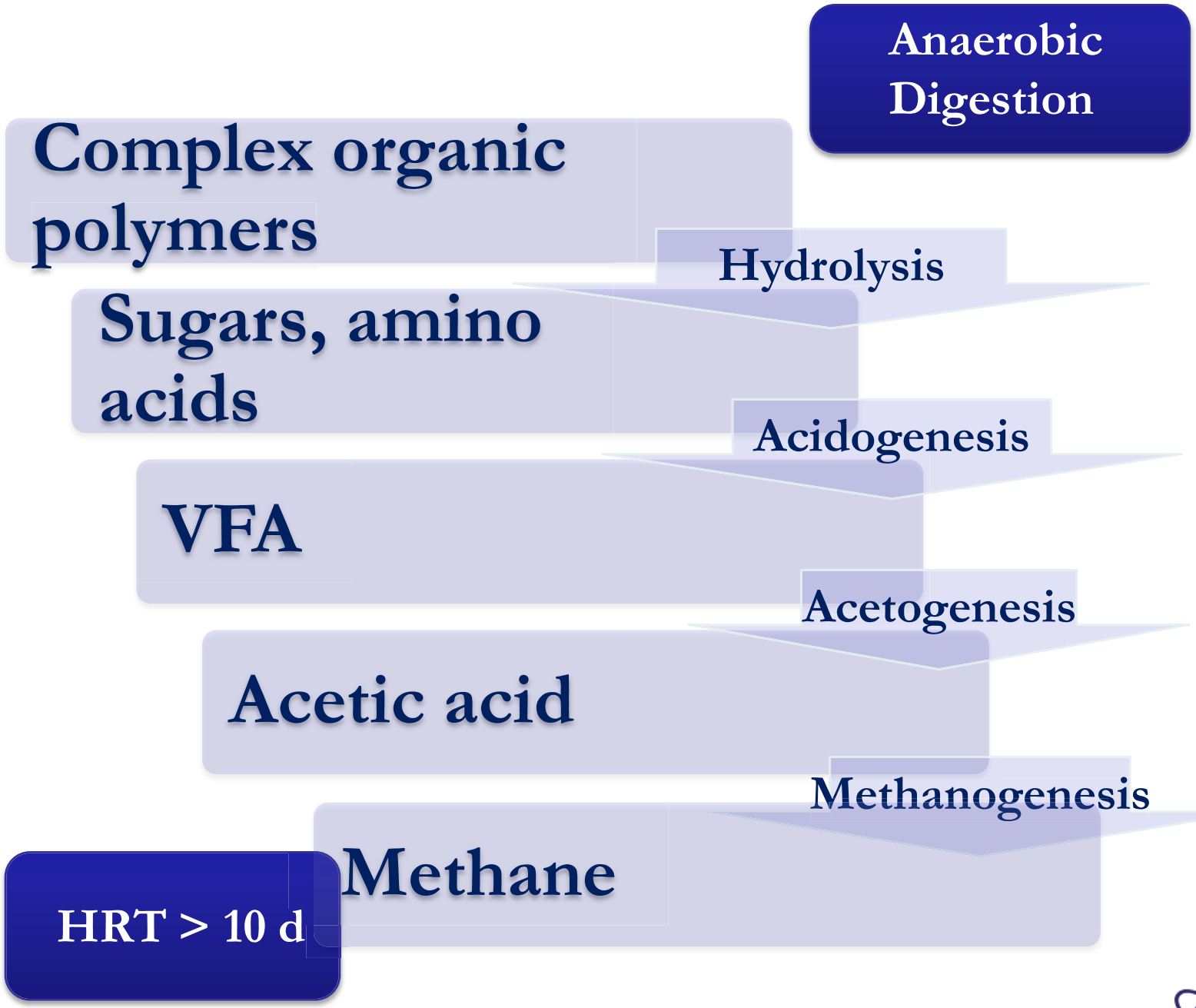

# Importance of Pilot Scale Research

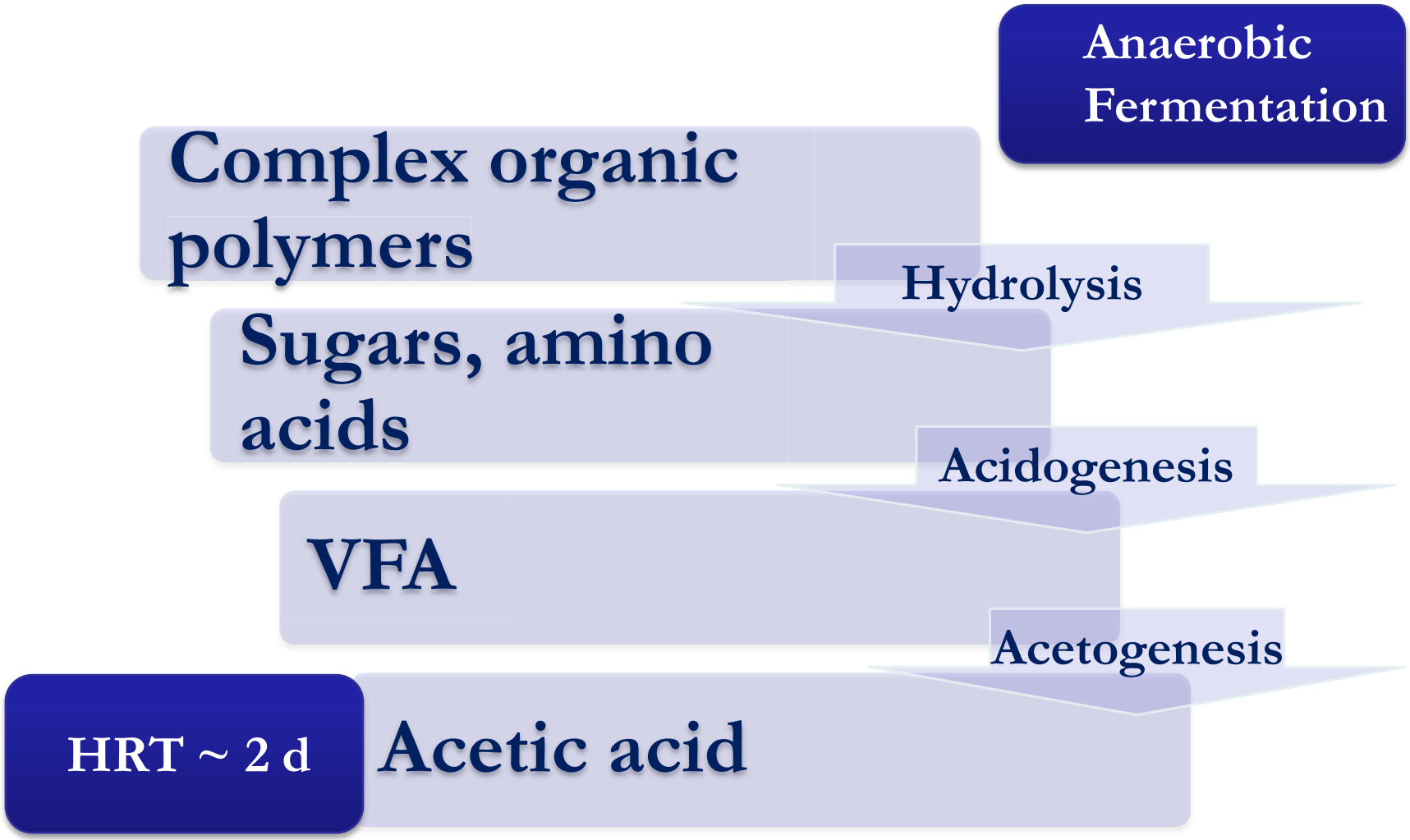
- Reflect true variability of FS from vacuum trucks
- More accurate field operations (unmixed reactors, flexible loading volumes)
- Baseline data for design guidelines
  - Minimize design retention time -> lower capital cost
  - Optimize for methane production
  - Optimize for additional resources (VFA)

# Approach: Comprehensive Pilot Operations with Modelling Analysis

Pilot Scale Field Operations → Process Modeling

- 5 months start up, 5 months full operation
- Two trains of six, $10m^3$ reactors
- 2-4 day HRT per reactor
- Measured parameters:
  - COD, VFA, TSS, VSS, pH, alkalinity, N H3-N
- Gas Analysis
  - $CH_4$, $CO_2$, $O_2$, $H_2S$


Anaerobic Digestion
Complex organic polymers
Hydrolysis
Sugars, amino acids
Acidogenesis
VFA
Acetogenesis
Acetic acid
Methanogenesis
Methane
HRT > 10 d




- Fermentation is more advantageous than just anaerobic digestion
- Fermentation can be incorporated into existing digestion processes

# Fermentation as a platform

- VFA for N and P removal
  - Using different types of biomass
  - Including food waste
- Chemicals
  - solvents, pharmaceuticals
- Biofuels
- Methanogenesis still can be conducted downstream
  - And probably needs to be conducted

# Dual-Phase Digestion and Fermentation of Sewage

PDS fermentation and storage at 26th Ward WPCP in New York City, 2002

- **Fermentation of PDS to produce fatty acids**
  - NYC spends about $15 million annually on synthetic chemicals
  - Also led to improved wastewater treatment efficiencies

# Overview of our process

**Organic waste**

Anaerobic fermentation to produce volatile fatty acids (VFA)

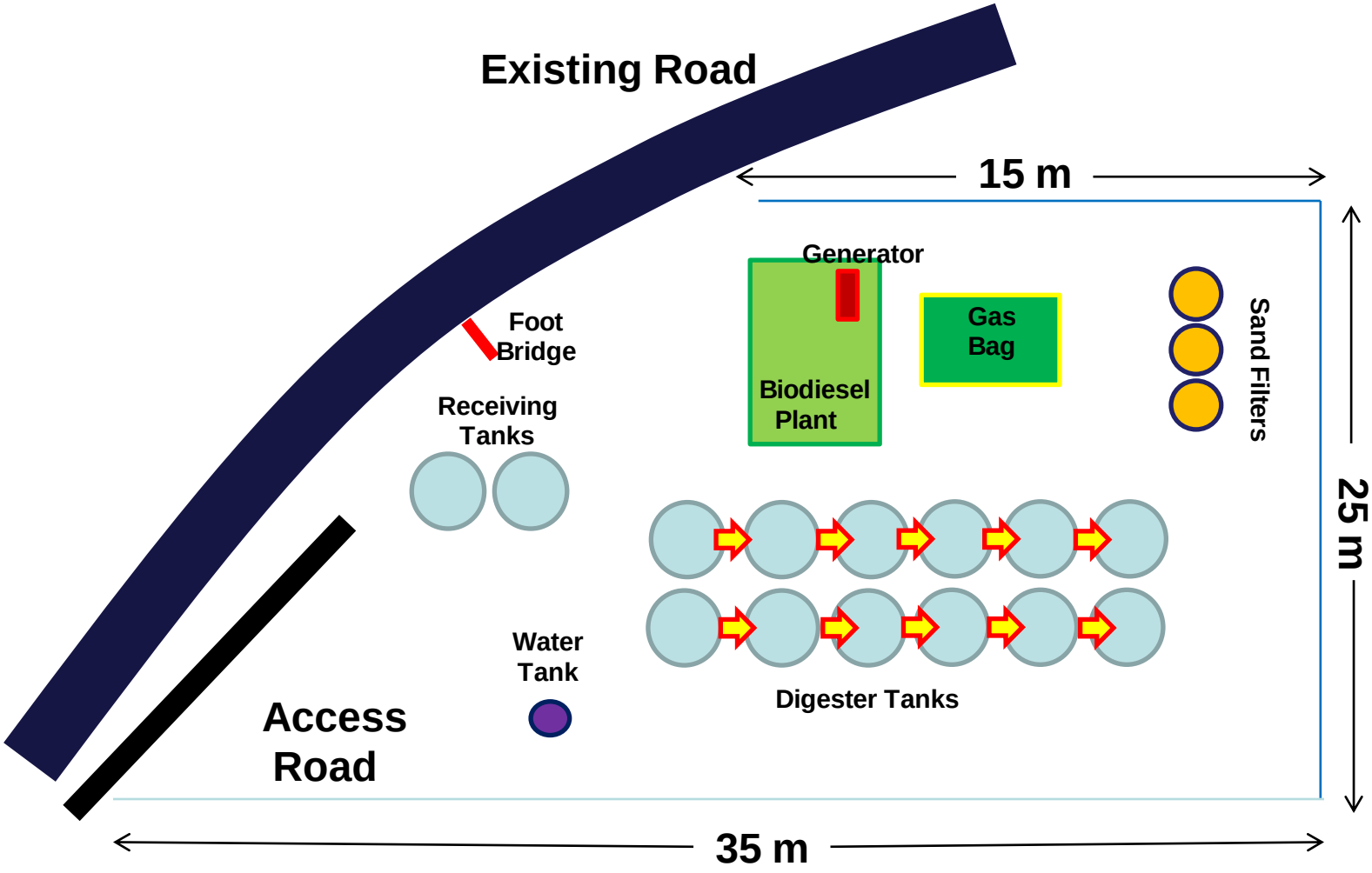
**Convert VFA to lipids**

**Harvest and extract lipids**

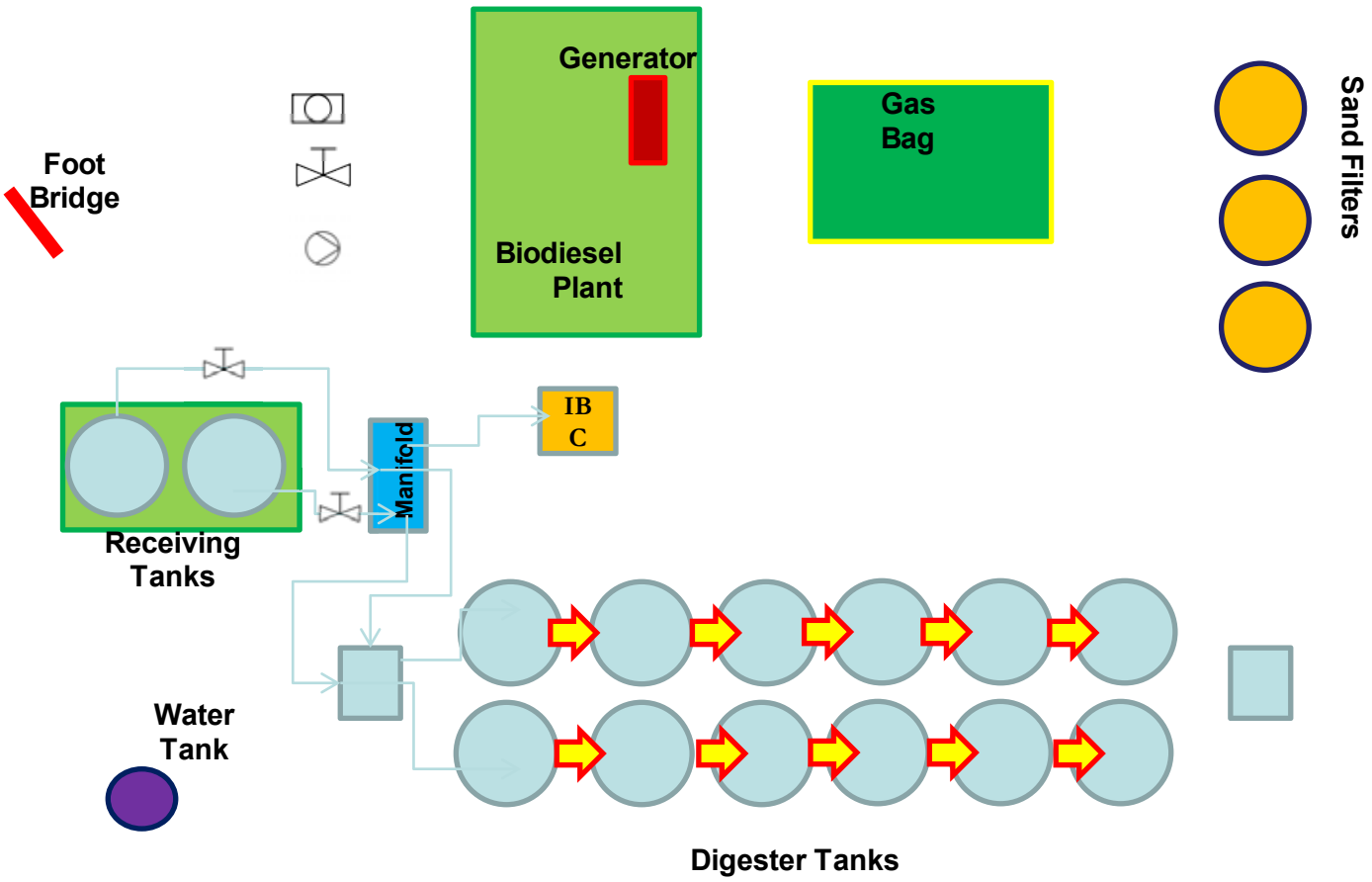
**Convert lipids to biodiesel**

# Dompoase Site Plan

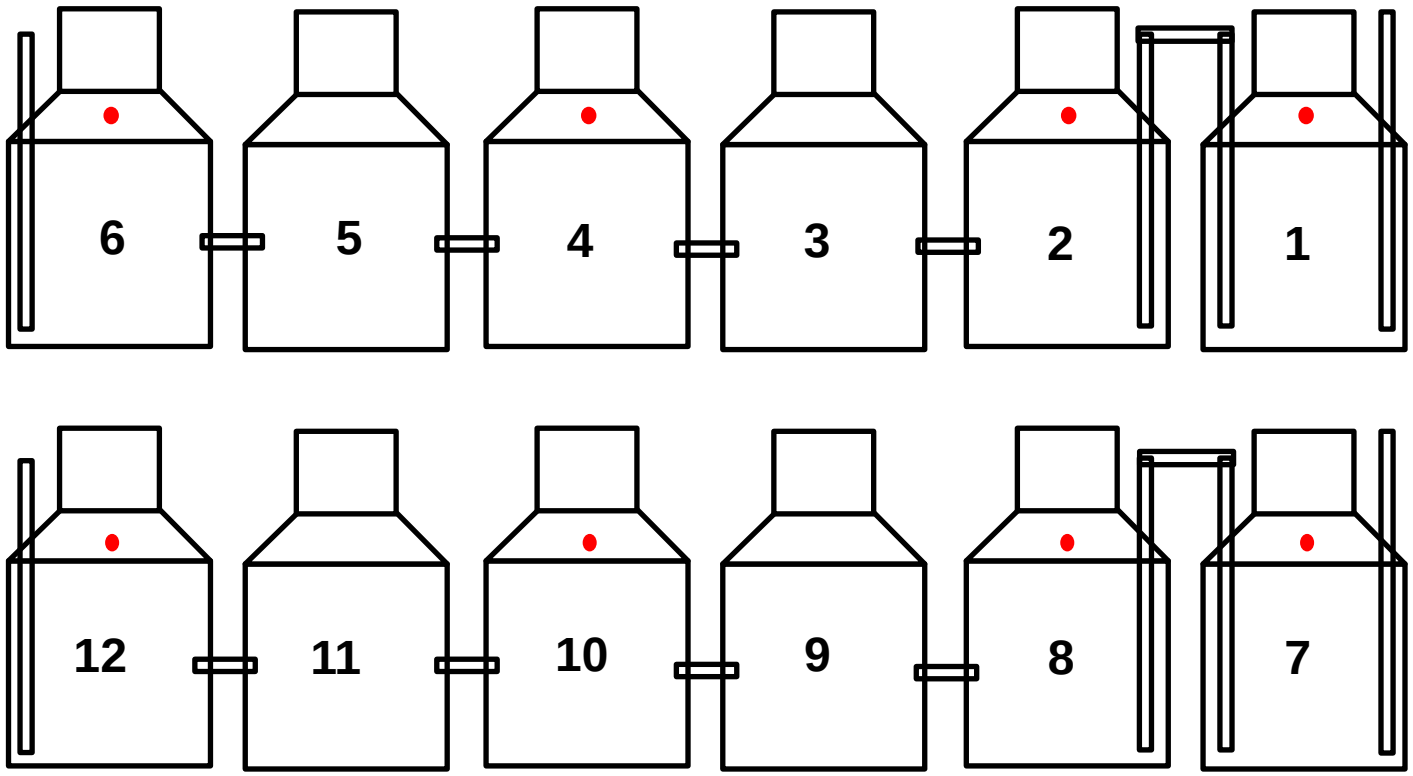
(surrounded by sludge)



# Dompoase Site Plan



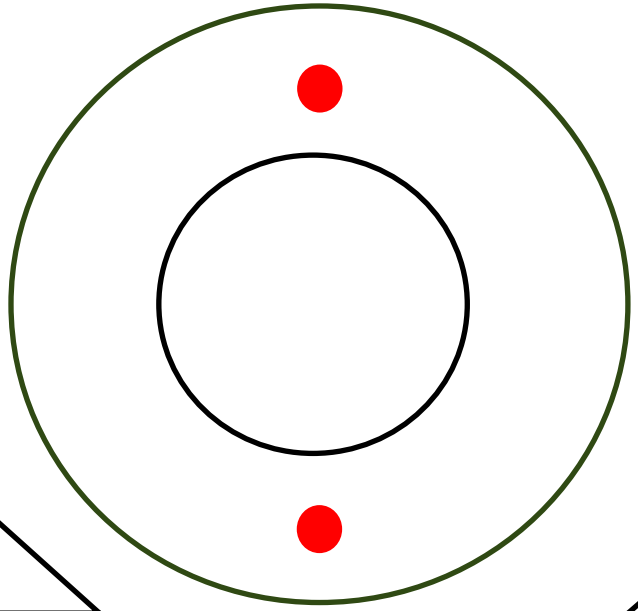
# Schematic – Side View



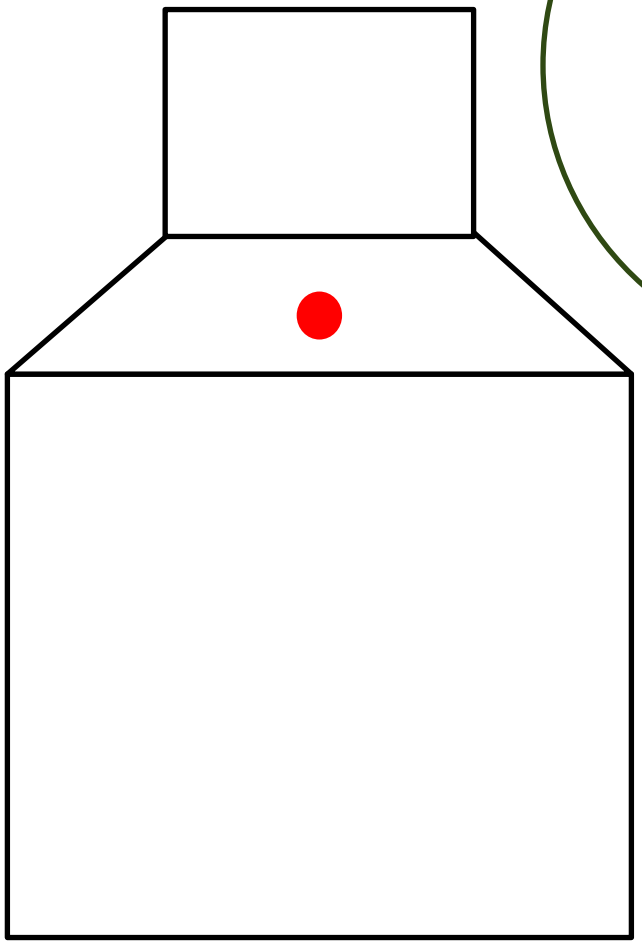

● = Approximate location of sample ports – 2 each per tank on opposite sides as shown in detail

# Digester Detail

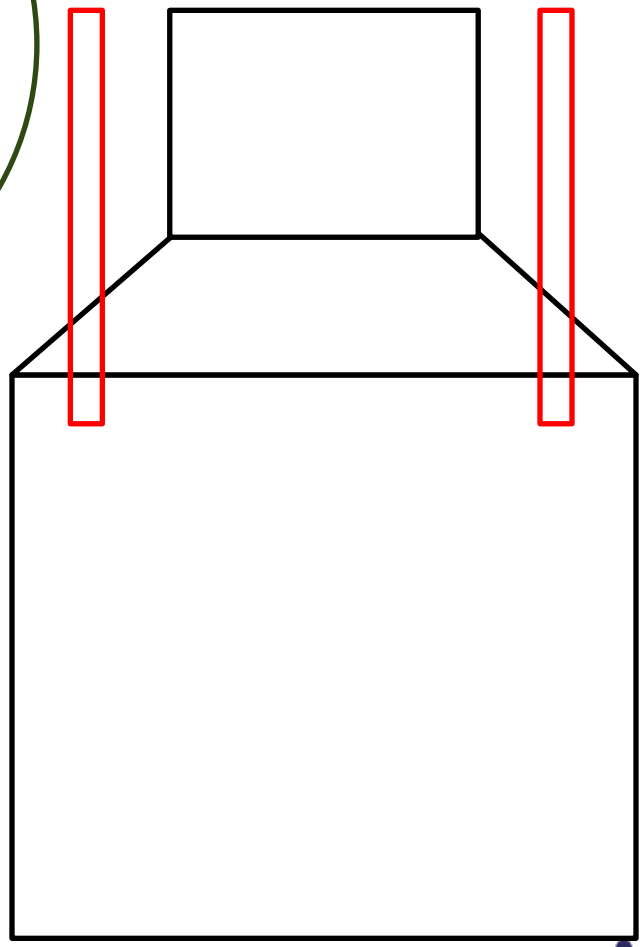
(not to scale)



Top View



Side View 1



Side View 2 – rotated 90 degree

# Plant schematic

# Plant schematic

**More photos for scale as well as to document that repeated visits to the site revealed no compaction being performed.**

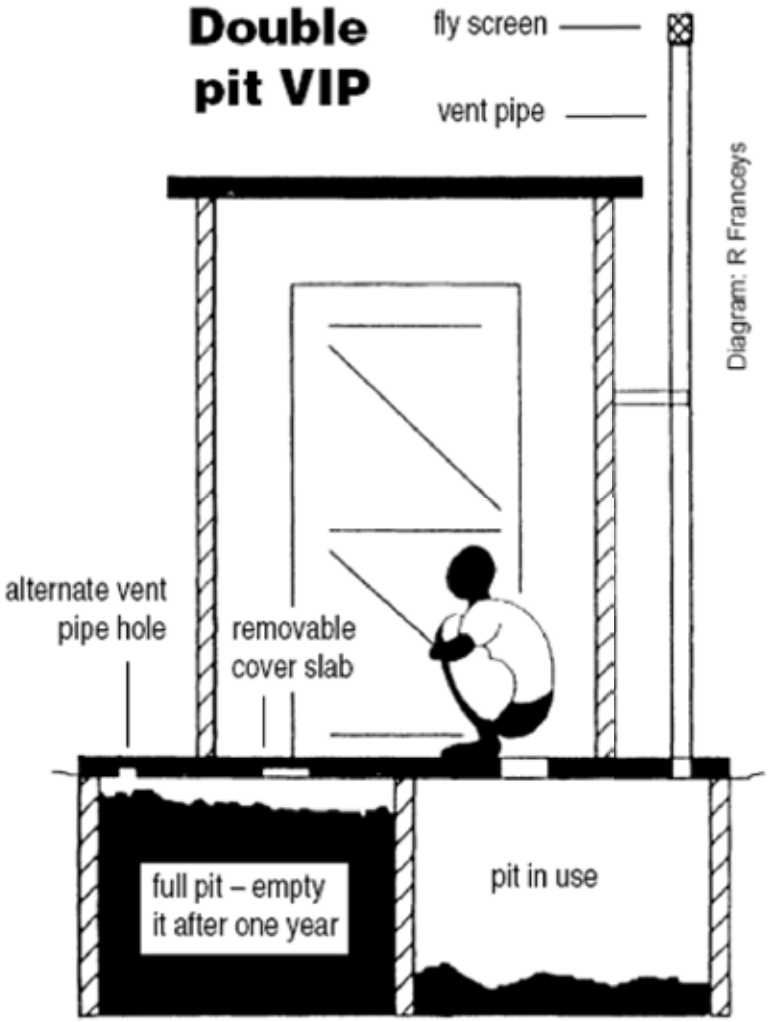
# Some practical issues- settling

**Note that the settling of the dirt fill has pulled the piping down and cracked the elbow. At the time of this photo, the settling was about six inches – it has continued to settle.**

# Source 1- Private septage

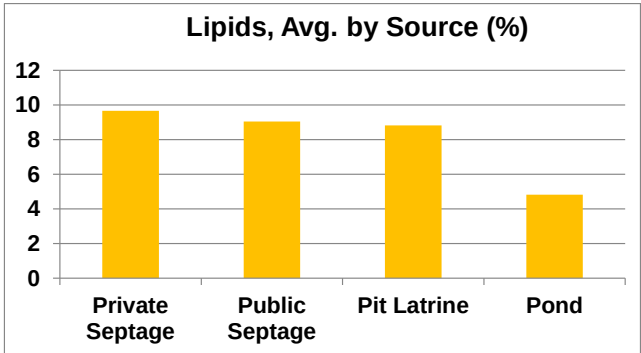
# Source 2- Public septage

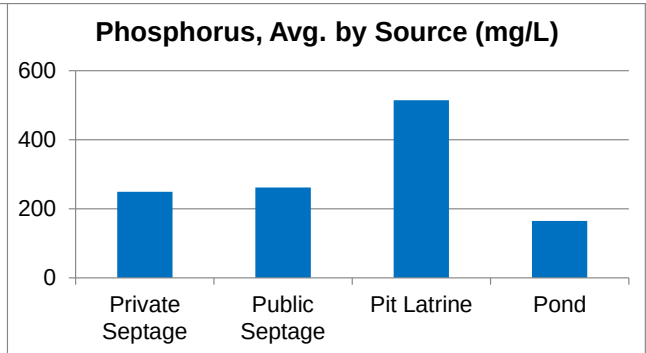
# Source 3- Pit Latrines

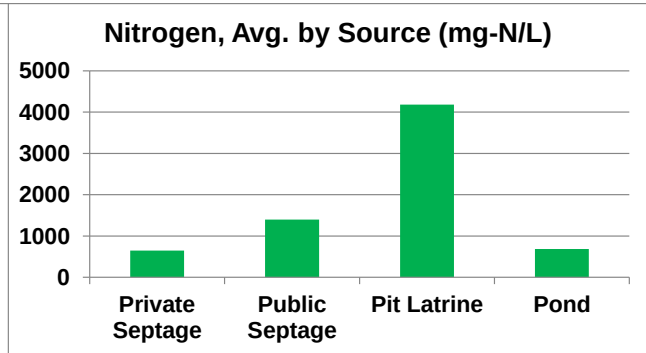


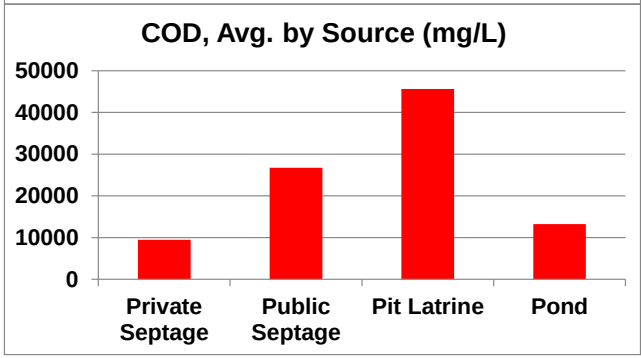
# Source 4- Ponds

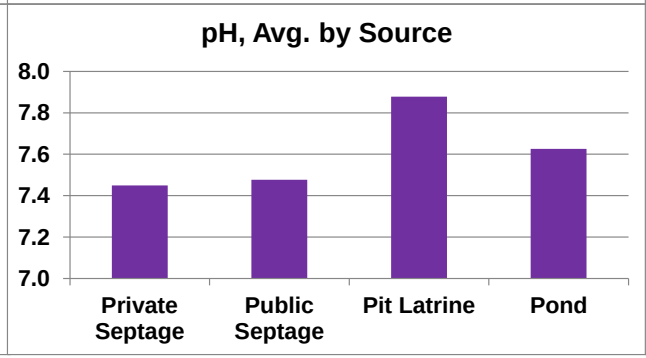
# Characteristics of fecal sludge

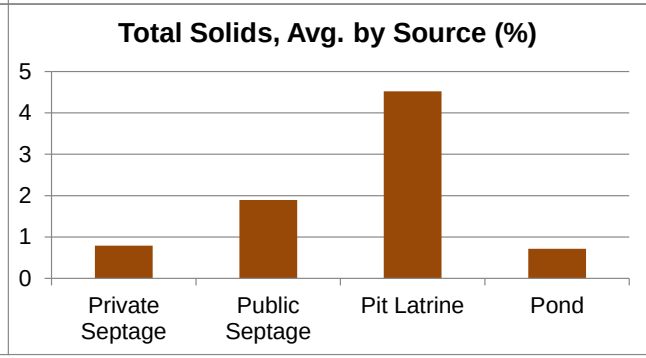


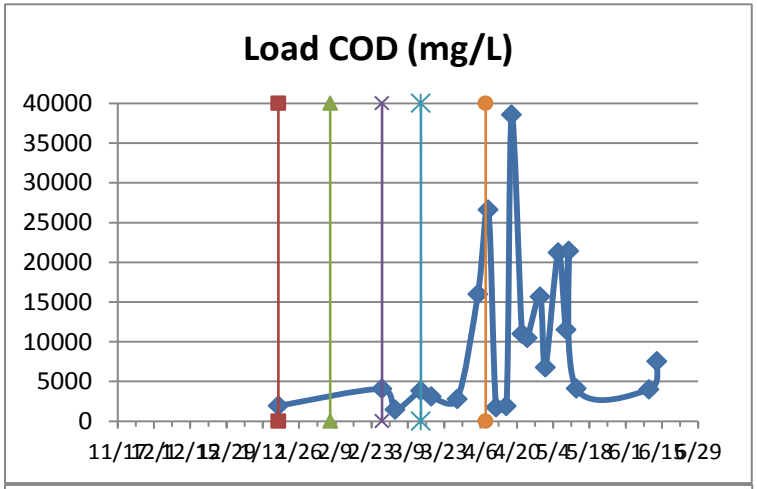



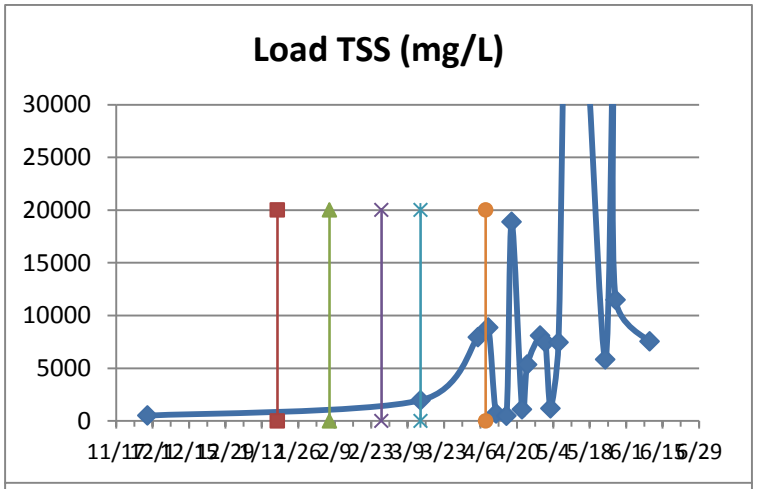



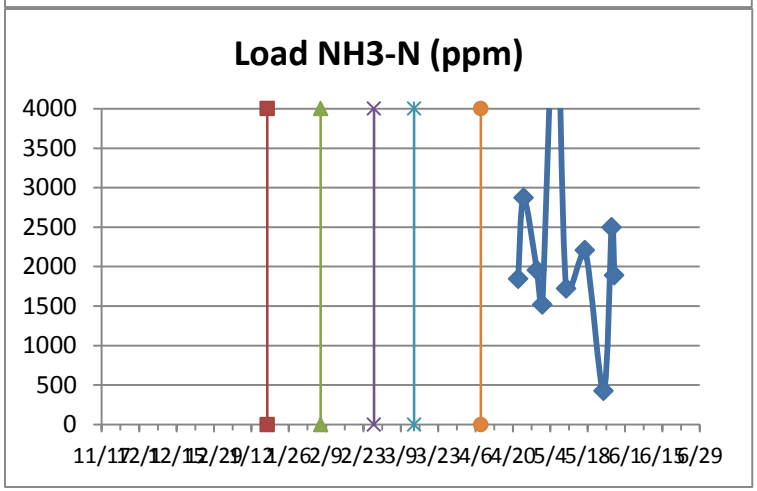



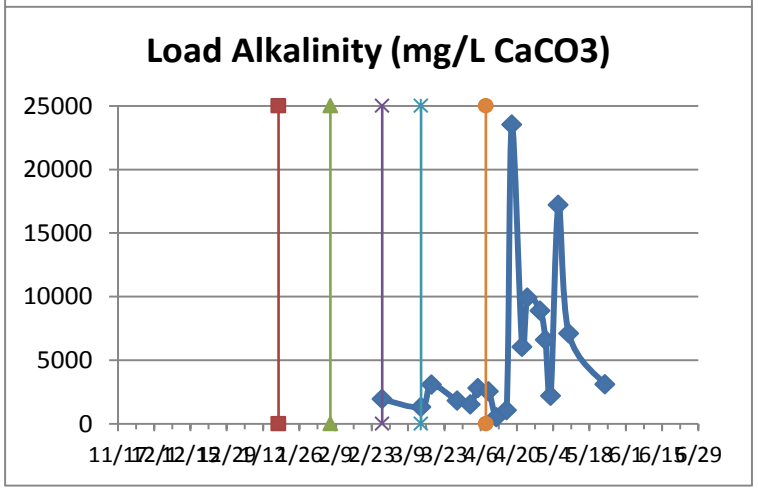



# Continuous monitoring of composite influent to reactors



# Step Feed Anaerobic Fermentation- A Novel Alternate for Faecal Sludge (FS) Processing

**Ato Fanyin – Martin**

**KNUST, Ghana**

**Earth and Environmental Engineering, Columbia University**

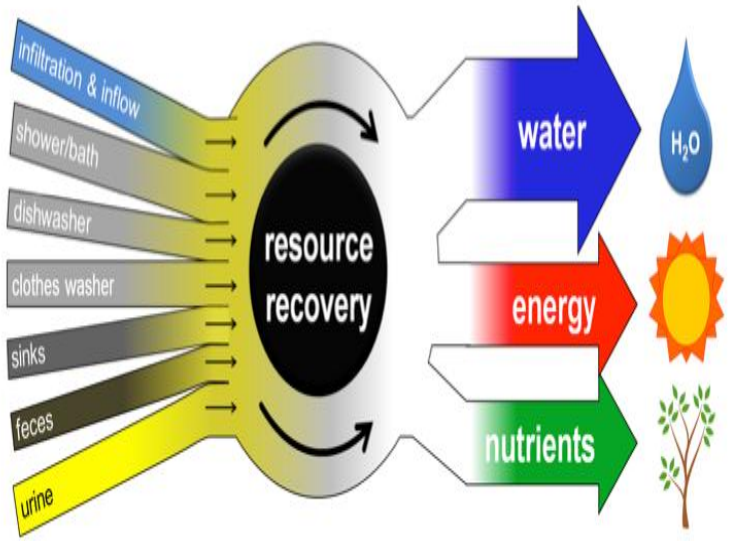
**Contact information: Prof. Kartik Chandran**

**E-mail: kc2288@columbia.edu**

# Where we Stand today

# What Do we Do?

Treatment




Resource recovery

# FS2BD Pilot Facility

**Processing of 5000L of FS a day with the aim of optimising VFAs and methane**

# Process Optimization

- **Pilot Plant Bottleneck**
  - **High VFA in the Influent**
  - **High biogas production in the front end digesters**
  - **Mixing**
- **Solution and Limitation**
  - **Step feeding**
  - **Lab limitation**
  - **Substitution of faecal sludge**
  - **Food waste**

# What is Faecal Sludge?

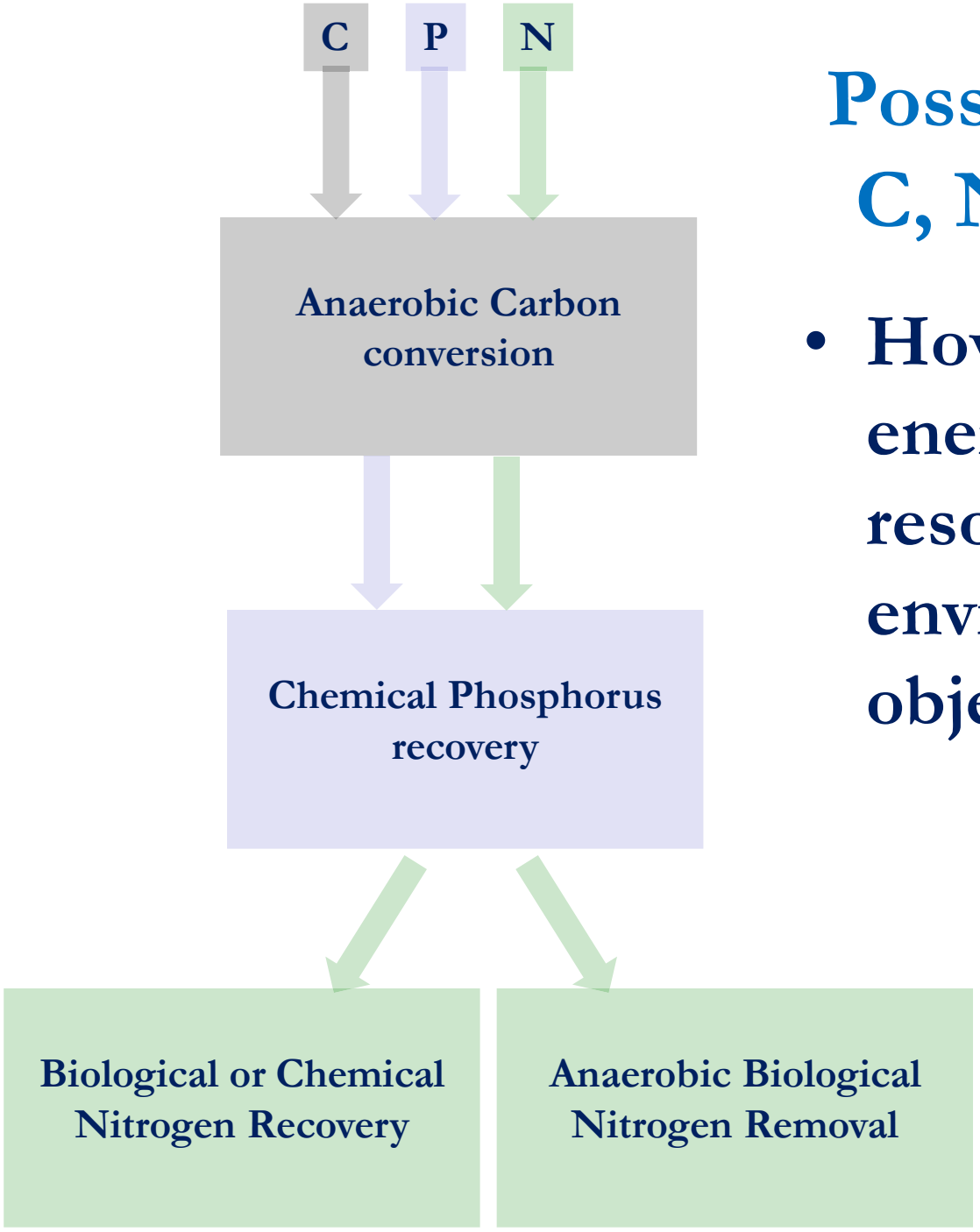
**Faecal sludge (FS) is sludge from on-site sanitation facilities (septic tanks, pit latrines, etc.) collected and transported by truck**

- **2.7 billion served by on-site sanitation**
- **Often discharged untreated to waterbodies**
- **Impacts to public health and water**

# What is Faecal Sludge?

**FS is principally different from sewage sludge, but still relies on sewage sludge research.**

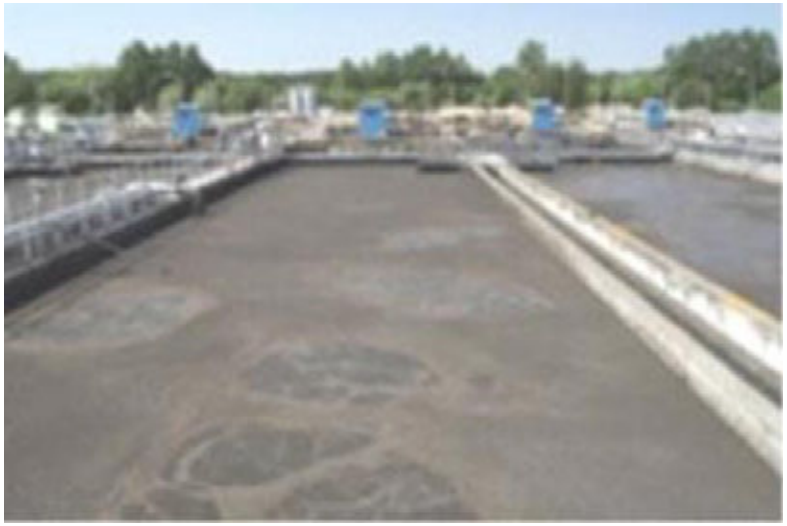
- **Mainly excreta, less kitchen waste contributions**
- **Extended storage time (weeks, months, years)**
- **Variable toilet systems (flush and non-flush)**

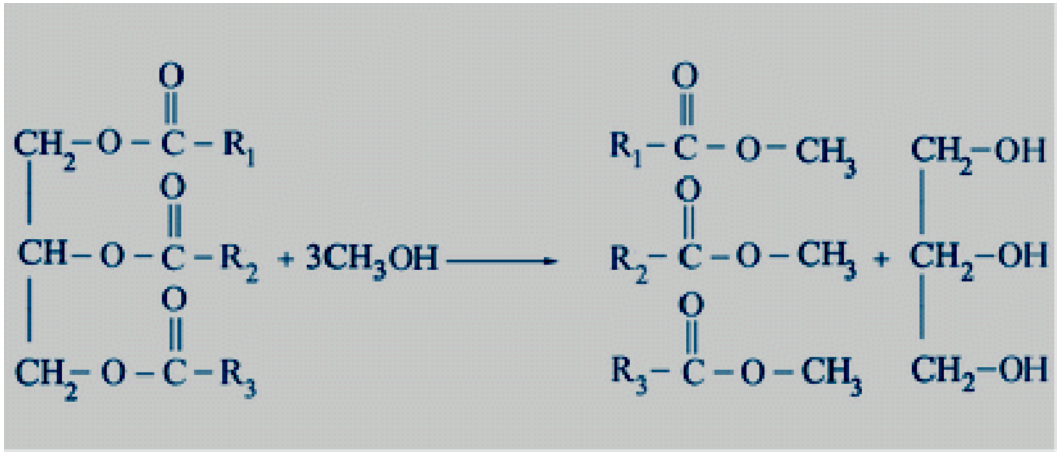



# Possible flowsheet for C, N and P recovery

- How to link recovery of energy or chemical resources with environmental process objectives

# Sewage sludge to biodiesel




- Using the fat content of biosolids
- Using MeOH for fuel production instead of N-removal

# Microbial conversion of VFA to lipids