High Solid Anaerobic Digestion and Management of Distillers Grains from Cassava Ethanol

Examination Committee
Prof. C Visvanathan (Chairman)
Dr. Thammarat Koottatep
Dr. Abdul Salam

By Mrs. Phonthida Sensai
EEM, Doctoral Candidate
Content

• Problem of distillers grains from cassava ethanol production

• Rational of research

• Methodology applied in this study

• Results and Discussions

• Conclusions and Recommendations

• Question from external examiner
Introduction: Problem of Distillers Grains from Cassava Ethanol Production

Find an option of distillers grains treatment

- It can be applied with large amount of distillers grains
- It applied highest benefit
- No impact to the environment
Rational of Research

- Characteristics of feed -- Co-digestion requirement of feed
- Optimum S/I ratio -- avoid the problem of overload VFA production causing imbalance in the system
- OLR for HSAD optimization
- The proper treatment option for distillers grains from cassava ethanol production -- consider GHG emission and energy benefit

Limitation of HSAD

Ammonia toxicity
VFA accumulation
Objectives of Study

- To determine the characteristics of distillers grains after stillage separation for their biochemical methane potential and limitations for anaerobic process

- To optimize methane yield of distillers grains in high solid anaerobic digestion using different loading rates

- To investigate and compare various waste management options for minimizing GHG emissions.
Methodology

Phase 1

- Stillage characterisation
- BMP test (Investigate potential CH$_4$ production)
- BMP test with C/N ration variation (Optimize C/N ratio)

Phase 2

- Optimised condition for start up phase of HSAD
- High solid anaerobic digestion of distillers grains

Phase 3

- Compare various treatment options for distillers gain (CO$_2$ emission and energy benefits)
### Phase I: Distillers Grains Characterization

- **Distillers grains characteristics**
- **BMP test - Methane production potential of distillers gains**

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Volume of CH(_4) (mL)</th>
<th>CH(_4) Content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed stock</td>
<td>Inoculums 34.12 mL + DW up to 72 mL</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Inoculums 34.12 mL + Cellulose 1 g + DW up to 72 mL</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Inoculums 34.12 mL + Whole stillage 14.97 mL + DW up to 72 mL</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Inoculums 34.12 mL + Thin stillage 23.93 mL + DW up to 72 mL</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Inoculums 34.12 mL + Dry DG 4.92 g + DW up to 72 mL</td>
<td></td>
</tr>
</tbody>
</table>
BMP Test with C/N Ratio Variation

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Feed stock</th>
<th>Volume of CH₄ (mL)</th>
<th>CH₄ Content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Inoculums</td>
<td>34.12 mL</td>
<td></td>
<td></td>
</tr>
<tr>
<td>+ DW up to</td>
<td>72 mL</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cellulose</td>
<td>1 g</td>
<td>0.06 gNH₄HCO₃</td>
<td></td>
</tr>
<tr>
<td>+ DW up to</td>
<td>72 mL</td>
<td>0.04 gNH₄HCO₃</td>
<td></td>
</tr>
<tr>
<td>+ DW up to</td>
<td>72 mL</td>
<td>0.02 gNH₄HCO₃</td>
<td></td>
</tr>
<tr>
<td>+ DW up to</td>
<td>72 mL</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Inoculums: 34.12 mL + Cellulose 1 g + DW up to 72 mL

Cellulose: 1 g + DW up to 72 mL

C/N = 20/1

C/N = 25/1

C/N = 30/1
Phase II : HSAD Optimization

Different C/N ratio

<table>
<thead>
<tr>
<th>Condition</th>
<th>C/N ratio</th>
<th>OLR (kgVS/m³.day)</th>
<th>HRT (day)</th>
<th>Waste load (kg/day)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>40/1</td>
<td>3.5</td>
<td>39</td>
<td>0.65</td>
</tr>
<tr>
<td>2</td>
<td>30/1</td>
<td>3.5</td>
<td>40</td>
<td>0.7</td>
</tr>
</tbody>
</table>

OLR variation with mixture of distillers grains and swine manure (C/N ratio 30/1)

<table>
<thead>
<tr>
<th>OLR (kgVS/m³.day)</th>
<th>HRT (day)</th>
<th>Waste load (kg/day)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.5</td>
<td>40</td>
<td>0.7</td>
</tr>
<tr>
<td>5</td>
<td>28</td>
<td>1.0</td>
</tr>
<tr>
<td>6.5</td>
<td>21</td>
<td>1.3</td>
</tr>
<tr>
<td>8</td>
<td>17</td>
<td>1.6</td>
</tr>
<tr>
<td>10</td>
<td>14</td>
<td>2.0</td>
</tr>
<tr>
<td>12</td>
<td>12</td>
<td>2.4</td>
</tr>
<tr>
<td>14</td>
<td>10</td>
<td>2.8</td>
</tr>
</tbody>
</table>
Experimental Setup

- Reactor 60 L with 40 L working volume
- Paddle 5 min on 20 min off
- Temp 55°C
Methodology for GHG Emission and Energy Estimation for Stillage Treatment and Management

1. Develop a GHG emission and energy calculation model

2. Sensitivity analysis with 3 variables: distance, ethanol production capacity and percentage of solid separation

3. Using model to estimate GHG emission, energy input, energy output and surplus energy of each treatment option for distillers grains

4. Comparison of stillage treatment and management from cassava ethanol production between using UASB and solid-liquid separation (kgCO$_2$-eq/MJ.kg of stillage)

5. Potential changes and errors and their impacts on conclusions to be drawn from the model

6. Suitable option for distillers grains treatment and management

7. Developed strategy to manage stillage which emit lowest GHG emission (kgCO$_2$-eq/MJ.tonne of stillage)
Distillers Grains Characteristics

<table>
<thead>
<tr>
<th>Parameter (unit)</th>
<th>Distillers grains</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>3.8-4.05</td>
</tr>
<tr>
<td>TS (%)</td>
<td>24.6-27.61</td>
</tr>
<tr>
<td>VS (% TS)</td>
<td>75.8-80.19</td>
</tr>
<tr>
<td>TOC (%)</td>
<td>37.5-41.1</td>
</tr>
<tr>
<td>TKN (%)</td>
<td>0.9-1.2</td>
</tr>
<tr>
<td>C/N ratio</td>
<td>37/1-41/1</td>
</tr>
</tbody>
</table>

• High solid content

• Temperature of distillers grains after the distillation process is high (70-80°C)

• Low pH (3-4) and also high C/N ratio (37/1-42/1)
BMP Test Results

- Methane yield -- thin stillage > whole stillage > distillers grains

- Challenge in applying distillers grains in anaerobic digestion --- low pH and high C/N ratio

- Adjust C/N ratio can enhance methane yield

- Methane yield of C/N ratio – 30/1 > 25/1 > 20/1
Effect of Co-Digestion

- pH was stable around 7.81 to 8.08, inhibitor level i.e. C/N ratio 40/1 and C/N ratio 30/1

- Substrates with C/N ratio of 30/1, contributed for higher ammonia concentration than C/N ratio 40/1 which resulted in the little pH variation
Effect of Co-Digestion

- VFA/Alk of both conditions was in appropriate range i.e., 0.07-0.29, idle for anaerobic process

- C/N ratio 40/1 -- Low volatile fatty acid production and it decreased from 2500 to 500 mg/L

- C/N ratio 30/1, VFA production was 3,900-7,900 mg/L.

- Condition 1 (C/N 40/1)–methane yield reduced to 0.17 m³ from 0.27

- Condition 2 (C/N 30/1)–methane yield remained constant between 0.30-0.33 m³CH₄/kgVS.
S/I ratio Optimization

- To avoid acidification during start up phase
- S/I ratio 7:1 had more frequently pH adjustment than other S/I ratio
- S/I ratio 7:1 had lowest methane yield

To start up dry anaerobic process

S/I ratio < 1:1 is recommend
Effect of OLR: Process Stability

- The VFA/Alk ratio during this period was 0.12-0.31 which was well within the optimum range (< 0.4)

- VFA/Alk ratio keep on increasing when OLR was increased

- Ammonia concentration was in the range of 321-681 mg/L, it was due to the corrected C/N ratio (30/1) of feed

- Gaseous ammonia was within the inhibition level
• Methane yield at OLR 3.5, 5, 6.5 and 8 kgVS/m$^3$.day was 0.33-0.34 m$^3$CH$_4$/kgVS.day

• Maximum VS removal efficiency was 53%.

• VS removal dropped to 42% and methane yield dropped to 0.26 from 0.33 m$^3$CH$_4$/kgVS.day during increasing OLR from 8 to 10 kgVS/m$^3$.day

• Conversion efficiency of the reactor at OLR 3.5-8 kgVS/m$^3$.day was in the range of 62-66%.

• After increased OLR to 10 kgVS/m$^3$.day, conversion efficiency dropped slightly to 58%, because high VFA/Alk ratio.
### VS Balance

<table>
<thead>
<tr>
<th>OLR (kgVS/m³.day)</th>
<th>VS in (kg)</th>
<th>VS biogas (kg)</th>
<th>VS remained (kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.5</td>
<td>0.14</td>
<td>0.092</td>
<td>0.048</td>
</tr>
<tr>
<td>5</td>
<td>0.2</td>
<td>0.132</td>
<td>0.068</td>
</tr>
<tr>
<td>6.5</td>
<td>0.26</td>
<td>0.168</td>
<td>0.092</td>
</tr>
<tr>
<td>8</td>
<td>0.32</td>
<td>0.199</td>
<td>0.121</td>
</tr>
<tr>
<td>10</td>
<td>0.4</td>
<td>0.232</td>
<td>0.168</td>
</tr>
<tr>
<td>12</td>
<td>0.48</td>
<td>0.3</td>
<td>0.18</td>
</tr>
<tr>
<td>14</td>
<td>0.56</td>
<td>0.3</td>
<td>0.26</td>
</tr>
</tbody>
</table>

- The conversion efficiency was 62-66% which was highest during OLR 3.5-8 kgVS/m³.
- VS remaining at OLR 3.5-8 kgVS/m³.day was 34-38%. After the reactor was operated at 10 kgVS/m³.day, the VS residue increased to 42% due to the rate of changing the OLR was altered.
- Although, OLR 12 kgVS/m³.day showed the best performance compare the conversion rate and % VS remaining, the results shows the unstable performance of biogas production.
• Overall energy input decreased with an increase in OLR

• During operation at OLR 3.5 to 8 kgVS/m³.day was in the range of 10.79-11.33 MJ/kgVS

• A lot of fluctuation in terms of methane was observed due system operation at OLR 12 kgVS/m³.day
Comparison of GHG Emission

- HSAD could potentially reduce GHG emission as compared to landfill, landfill with energy recovery RDF and animal feed 94, 93, 87, and 4%.

- Composting released GHG emission less than HSAD 21%. However, composting consumed energy option whereas HSAD was an option which recovered energy.
Comparison of Energy

The comparison of energy input of each scenario showed that HSAD consumes highest energy for HSAD operation.

Landfill, composting and animal feed did not provide any energy output.

Energy output from RDF, HASD and landfill with energy recovery - 952, 1,360 and 1,529 MJ/tonne of distillers grains respectively.
GHG Emissions of Assessed Treatment Options

- Landfill, composting and animal feed consumed energy option from transportation with no energy output from these processes.

- RDF and HSAD were found to provide surplus energy. GHG emission from landfill with energy recovery RDF and HSAD were 4.50, 9.28 and 1.03 kgCO$_2$-eq/MJ recovered energy/tonne of distillers grains.

- HSAD was a suitable option for distillers grains due to its potential for energy recovery and low GHG emissions.
The Proper Option for Stillage Treatment and Management.

GHG emission of solid-liquid separation option was more beneficial than applied stillage directly with UASB and higher surplus energy as well.

GHG emissions clearly show that stillage treatment using solid-liquid separation emit less GHG emission than using UASB when percentage of solid separation < 30 %. Therefore, solid-liquid separation option should be promoted.
Conclusions


- The significant characteristics for whole stillage, thin stillage and distillers grains were low pH (3.80-4.16) and high C/N ratio (40/1-44/1).

- Distillers grains can be potentially used at the start up phase but after long run reactor performance drop because of high C/N ratio of feed.

- The optimum S/I ratio of dry anaerobic digestion for distillers grains was < 1 on gVS basis.
Conclusions


- Distillers grains was adjusted C/N ratio from 40/1 to 30/1 by swine manure. The results C/N ratio 30/1 enhance methane yield 43%.

- HSAD of distillers grains with co-digestion was carried out at OLR 3.5,5,8,10,12 and 14 kgVS/m³.day. During the entire operation, the system showed stability as indicated by pH, VFA/Alk and ammonia.

- But at OLR 14 kgVS/m³.day, system performance dropped due to an increasing in VFA/Alk ratio, which was close to the inhibition level.
Conclusions


- After OLR 10 kgVS/m³.day, system performance dropped due to an increasing in VFA/Alk ratio, which was close to the inhibition level.

- At OLR 12 kgVS/m³.day, maximum energy output was considered to be the best case for this research. But due to the accelerated change during OLR 10-14 kgVS/m³.day, system stability was observed to be low.

- At high OLR operation – require more acclimatization period than reactor operation of Low OLR.
Conclusions of phase III (Distillers grains management)

- HSAD had lowest GHG emission with energy recovery. Therefore, it can be concluded that HSAD could be an attractive treatment and management.

- The percentage of solid separation < 30% shows the lower GHG emission (kgCO$_2$-eq/MJ. tonne of distillers grains) than stillage treatment by using UASB. Therefore, the percentage of solid separation < 30% was considered as the suitable option for stillage treatment.
Recommendations and Future Work

- The careful studies need to be made at higher loading rates of HSAD to bring down the overall footprint of the reactor.

- Disinfection of the digestate from HSAD has received less attention due to non-availability of regulation policies. Thus estimating the reuse potential of sludge treated using HSAD.

- As changing the increasing rate of OLR posed as a stumbling block in the research, the optimum rate of increasing OLR should be investigated to overcome the problem of unstable performance of system during reactor operation at higher OLR.

- Potential research can be directed towards energy recovery from sludge via hydrogen ion. With co-benefits being reduced GHG emission and generation of electricity.

- In this research, the scope of study for distillers grains management focused only GHG emission and energy issues. Economics should also be considered to find the possible investment and return period.
4. Page 7: Table 2.1: the data on corn is not reasonable. It is common to have 10 L/L stillage. When you give a wide range of volume, the COD and BOD cannot be an exact value. Check and find a better reference.

Answer: I have found a better reference and put it in Table 2.1. According to Khanal 2008, stillage yield from corn ethanol industry was 10-16 L/L EtOH.

9. Section 2.7: When it is possible to sell the distillers grains as e.g. animal feed or RDF, why is landfilled? You should describe the answer in your text.

Answer: Landfill was the baseline condition for distillers grains treatment which was the worst case scenario, in the case that the cassava ethanol production did not utilize distillers grains for any purpose.

10. Theoretical yield of biogas from the stillage?

Answer: The theoretical yield of biogas from stillage was 0.35 m³ CH₄ / kg of COD (Hutnan et al., 2003).
Questions from External Examiner

13. Section 3.1.1: Anything about the specification of the decanter? (the one that you used to get thin stillage)

Answer: Whole stillage, thin stillage and distillers grains were collected directly from cassava ethanol plant, from which whole stillage was separated by decanter centrifuge. Therefore, the detail for the decanter was not added in section 3.1.1

15. Section 4.2: What is “specific methane production”? Do you mean methane yield or specific productivity?

Answer: In section 4.2, the results from BMP test was “methane yield” (NmLCH$_4$/gVS). Implying methane yield of a specific feedstock at a given condition for full utilization of the organic matter.

16. Section 4.11.3: it is difficult to understand why the GHG of compost is so low and RDF is so high. Considering the data for the landfill, the GHG for compost and RDF should be around 1000. You should consider where the methane (and similarly RDF) is used and calculate saving the GHG by those applications

Answer: the GHG emission from composting was 630 kgCO$_2$-eq/tonne of distillers grains. GHG emission from RDF has been modified to 608 kgCO$_2$-eq/tonne of distillers grains
Thank you