Torrefaction of Biosolids: Mass Spectrometry Elucidation of Product Gases

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Abstract

Sewage sludge is produced from wastewater at a rate in excess of 6.5 million dry metric tons per year. Currently in the US, much of the biosolids generated from the sludge are taken to landfills for disposal and are not being utilized. These biosolids contain combustible organic matter that has the potential to be used as a “green” fuel. Utilizing the torrefaction process, a mild form of pyrolysis, biosolids can be converted into a coal-like substance that can potentially be blended and co-fired with fossil fuel derived coal for the production of electrical power.

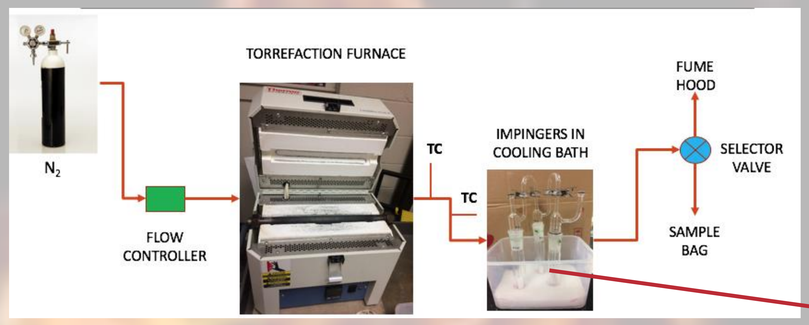

Figure 1 showing a block diagram of the biocoal production procedure and collection of gases downstream.

In addition to the “biocoal” produced via torrefaction, the product gases generated from the partially devolatized biomass can be recovered and used to heat the torrefaction reactor, provided it contains sufficient heating value, which would save on the costs of operation. The goal of this study was to utilize mass spectrometry to elucidate the chemical compounds in the product gas to determine the heating value, and whether or not it will suffice as a fuel gas to heat the torrefaction reactor.

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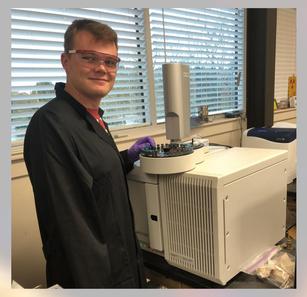
Method for Product Preparation

- Wet biosolids were dried in oven for 24 hrs at 110 °C
- Dried biosolids were ground to between No. 60 and 200 mesh sizes
- Ground biosolids were torrefied for 30 min at 250 – 350 °C under nitrogen flow (100 mL/min) in a tube furnace
- Condensable gases were collected in an impinger series cooled to -10 °C
- These liquid samples were then loaded into autosampler vials to be analyzed via GC-MS

Analytical Method

- Injector Conditions 280 °C split mode with a 60:1 split ratio
- Column Conditions
  - Restek RtX- 1ms, 10 m length x 0.10 mm inner diameter, 0.1 µm film
  - Column Program: 40 °C for 0.5 min, program to 150 °C at 5 °C/min with no hold, then 300 °C at 20 °C/min for a total run time of 35 min
  - Helium flowrate 0.6 mL/min
- Detector Conditions
  - Electron Impact 50 - 450 m/z



Data / Results

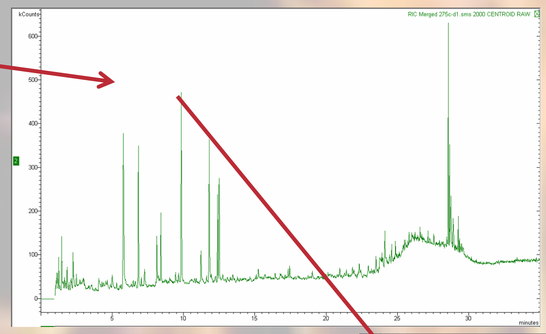
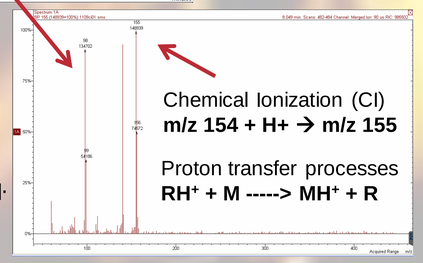
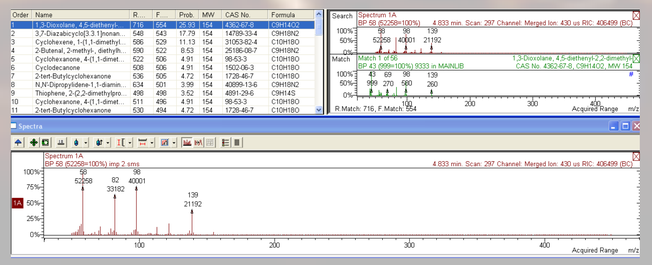


Figure 2 showing a total ion chromatogram of VOC gases from the impinger using Electron Impact (EI) GC/MS.

Positive chemical ionization
Mass spectrometry: Methane

$CH_4 + e$ -----> $CH_4^{+\cdot} + 2e$ ------>
$CH_3^+ + H\cdot$
$CH_4^{+\cdot} + CH_4$ -----> $CH_5^+ + CH_3\cdot$
$CH_4^{+\cdot} + CH_4$ -----> $C_2H_5^+ + H_2 + H\cdot$





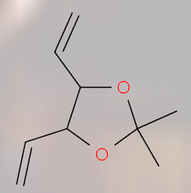


Figure 3 showing EI spectra of unknown in red and NIST Mass Spectral Library Match in green to 1,3-Dioxolane with the structure to the right.

Data / Results cont’d

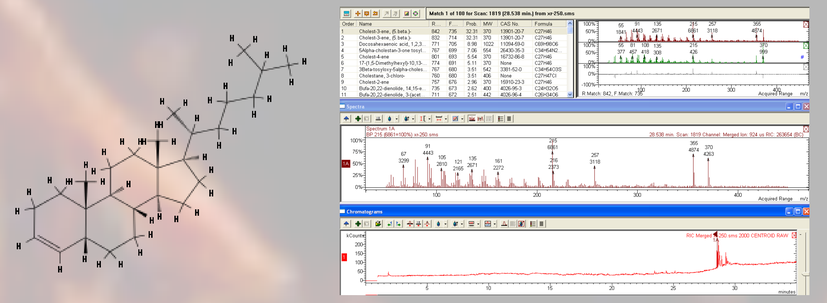


Figure 4 showing EI spectra of unknown in red and NIST Mass Spectral Library Match in green to cholest-3-ene with the structure to the left.

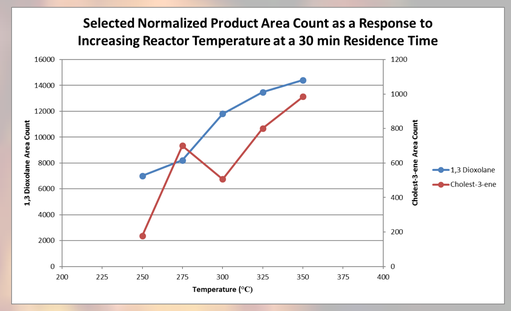


Figure 5 showing the response of both compounds plotted against different temperature conditions in the torrefaction reaction. Note in the cholest-3-ene plot there is a potential transition of melting to volatilization point between 275 and 325C.

Conclusions

This method has been demonstrated to elucidate the unknown compounds in the volatile gas produced from the torrefaction process. With the proper identification of the compounds, one can calculate the heating value of the product gas and possibly use it as a fuel source to heat the torrefaction reactor. This will allow the torrefaction reactor to operate autothermally and lower the fuel cost to produce a clean biocoal fuel.

Future Works

- Identify VOC’s and determine the heating value of product gas
- Continue to study the effects of torrefaction reactor conditions (temperature, residence time, carrier gas) on the VOC’s produced.