Determination of Long Chain Fatty Acids in Anaerobic Digesters Treating Source Segregated Food Waste Using A Rapid Non-Derivatisation GC-FID Method

Ying Jiang, Yue Zhang, Charles Banks

1. Introduction

Long chain fatty acids (LCFAs) are the common products in food waste anaerobic treatment; mainly deriving from lipid hydrolysis stage of the anaerobic digestion. Even at low concentration , LCFAs can be inhibitory to anaerobic microorganisms due to their surface active property and tendency of adhering to the cell wall, thus impeding the passage of essential nutrient through the membrane. When operating anaerobic digesters treating high lipids content substrate such as food waste, to have a rapid method to monitor LCFAs concentrations in digestate is in urgent demand..

Traditional gas chromatography method for LCFAs requires the free fatty acid to be derivatised to a methyl ester (FAME) to enhances the volatility of free fatty acids. This process is time-consuming and involves toxic chemicals. Following the advance of the GC technology and emerging of the high performance capillary column, the methylation step can be omitted providing that a highly polar capillary column and optimised GC conditions are applied.

2. LCFAs Extraction Procedure

Anaerobic Digestate Sample (Around 1.5g) prepare in triplicate for QC.
0.05g NaCl, 2 drops of 50% H_2SO_4 , and 5ml of 50:50 Hexane- Methyl tertiary butyl ether (MTBE) mixtures were then added.

↓

Mixing in vortex mixer at 2400rpm

↓

20 minutes in ultrasonic bath to enhance extraction

↓

Organic layer is removed and centrifuged. Supernatants are now ready for GC analysis

↓

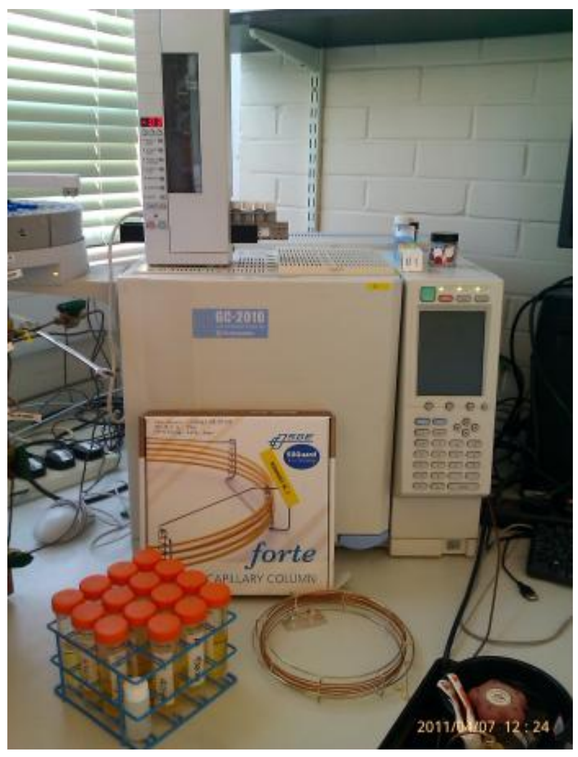

Fig. 2 Samples are ready for analysis, also shown are the GC (Shimazdu 2010) and column used in this study.

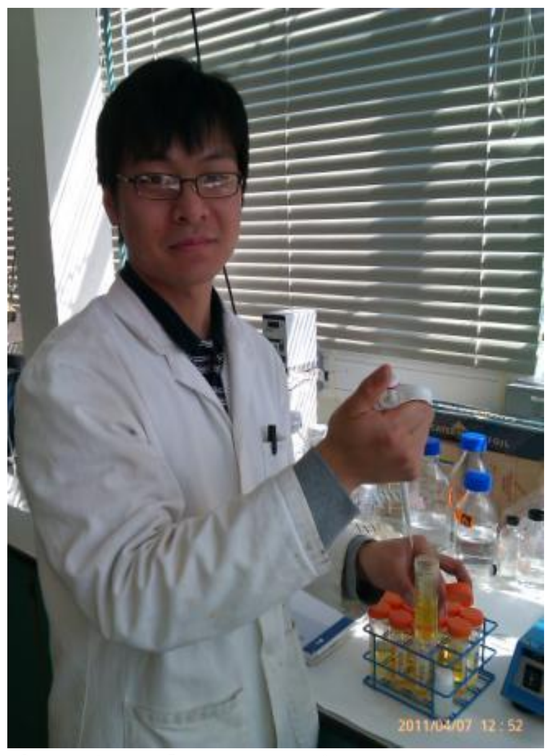

Fig.1 Sample Preparation

3. Instrument parameters

- FID Temperature: 280° C
- H_2: 40mL/min
- Air: 400mL/min
- Makeup flow: 30mL/min helium
- Column: 0.25mm × 30m, 0.25µm thickness, SGE Forte Capillary Column BP-21 (FFAP)
- Column flow: 2.0 ml/min, helium
- Oven temperature: initial 160° C; 10° C/min; 225° C, 20 min
- Injection volume: 1µL in 50:50 Hexane- MTBE mixtures

4. Method Validation

Palmitic, stearic, and oleic acid were used as LCFAs models injected into the GC system. Precision of the method was evaluated using two criteria: reproducibility and repeatability. precision results are reported as relative standard deviation (RSD, %), which should not exceed the 20% .

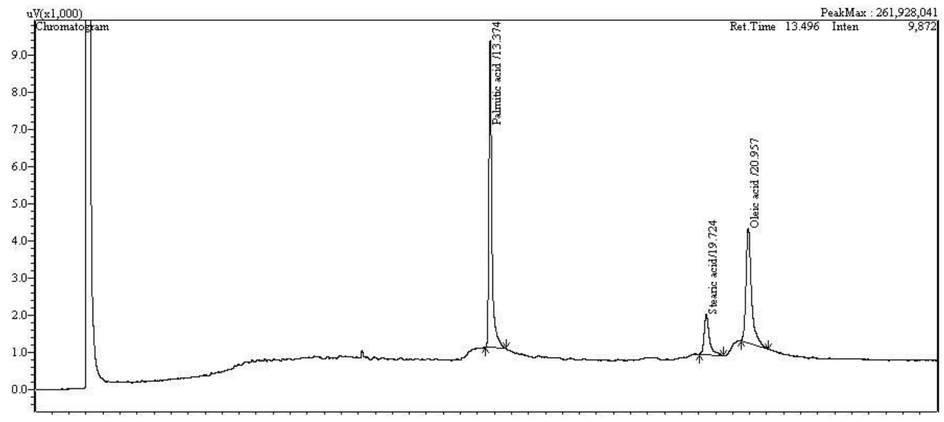

Fig.3 Chromatogram of 50mg/l standard fatty acids solution

In 6 repetitive runs at 3 concentration levels, the relative standard deviation (RDS) of peak responses for palmitic, stearic and oleic acid were all well below 20. The results of the study are shown in the bar graph in Fig. 4. GC flow control has provided very narrow retention time windows of ±0.016, 0.017, and 0.018 minutes for palmitic, stearic and oleic acid respectively over 1 month time throughout this study.
The generated calibration curves were linear over the concentration range studied with coefficients of correlation P≥0.99 for all the analysed LCFA.

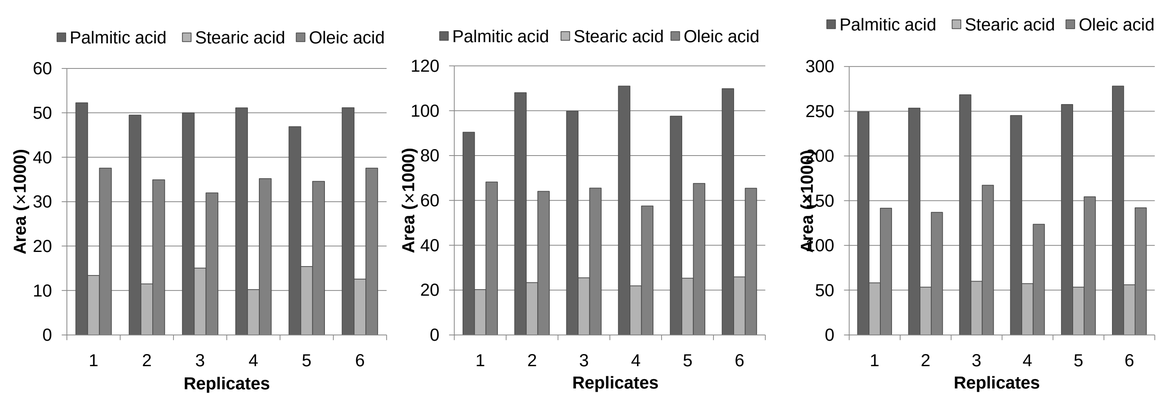

Fig.4 Precision of detector response following the sequential injections LCFAs standards (Concentration from left to right are 50mg/l, 250mg/l and 500mg/l)

In order to validate the extraction efficiency, 3 spiked samples were prepared, each were spiked with 0.1mg palmitic, stearic and oleic acid, the average spike recovery are 103.83%, 127.17% and 84.23%, respectively.

5. Application of the Method

The GC method described above has then been applied into monitoring LCFAs concentrations in a group of lab scale mesophilic (35° C) anaerobic digesters treating food waste adopting different trace element dosing scheme. LCFAs concentrations were significantly lower in digesters with addition of trace elements (Se, W, Mo, Co, Ni, B, Al, Cu, Fe and Zn) .

In the 24 hours kinetic study, digesters without trace element supplementations had noticeable accumulation of LCFAs after the feeding, indicated by the spiked up curve of LCFAs concentrations, whereas in digesters which have been regularly supplementing trace elements, the LCFAs concentrations remain stable at all time (as shown in Fig. 5, similar pattern were also observed with Stearic acid and Oleic acid).

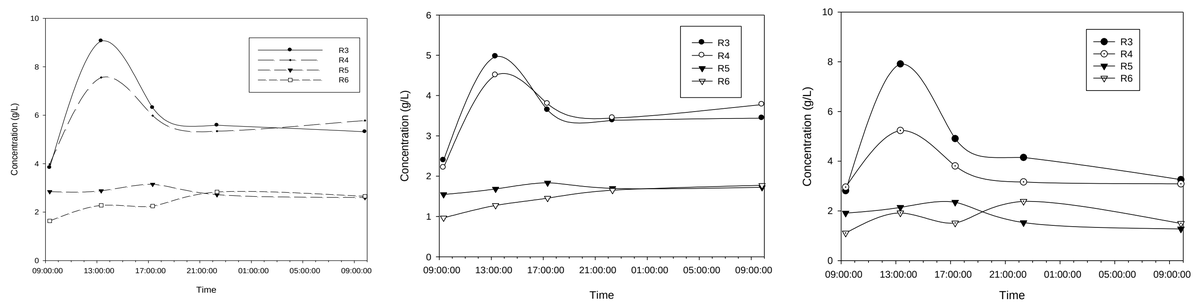

Fig. 5 LCFAs degradation kinetic within 24 hours (from left to right: Palmitic, Stearic and Oleic acid.)

UNIVERSITY OF Southampton
School of Civil Engineering and the Environment

School of Civil Engineering and the Environment,
University of Southampton, Southampton, UK
SO17 1BJ
Email: Y.Jiang@soton.ac.uk