Electronic Supplementary Material (ESI) for RSC Advances.

Rapid high conversion of high free fatty acid feedstock into biodiesel using continuous flow vortex fluidics

Joshua Britton and Colin L Raston

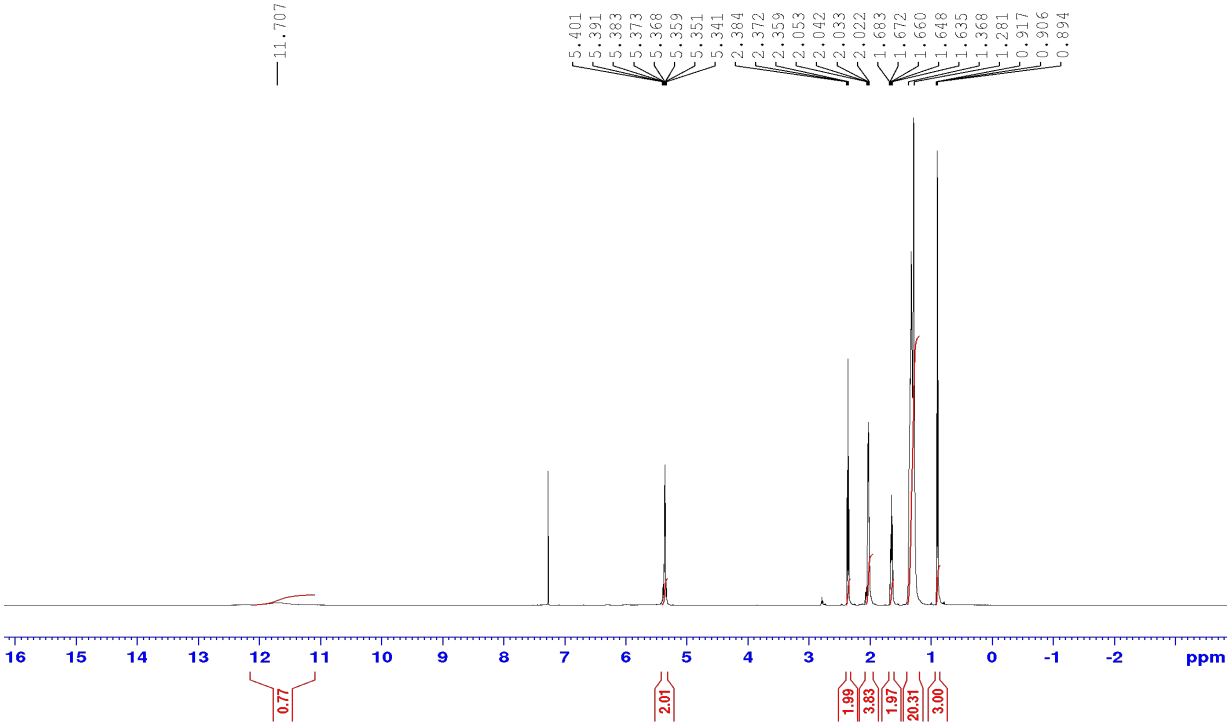
Chemical and Physical Sciences, Bedford Park, Flinders University, South Australia, SA 5042.

Table of Contents

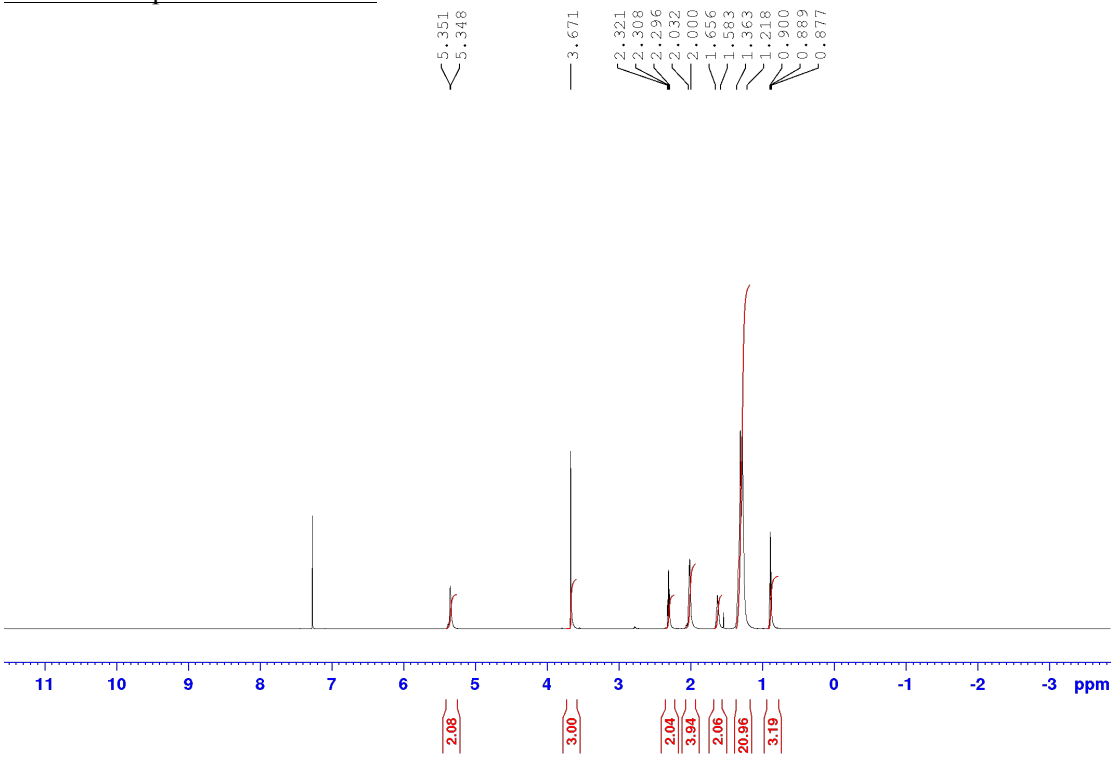
General spectroscopic details

^{1}H and ^{13}C NMR were obtained on a 600 MHz Bruker spectrometer and performed in $CDCL_3$.^{1}H NMR was referenced to 7.26 ppm whilst ^{13}C was referenced to 77.16 ppm. Typical quantitative conditions were used (Delayed pulse (D_1) – 10.00 and Number of Scans – 125) to ascertain the purity of the biodiesel. FT-IR were recorded using a Perkin Elmer FTIR spectrometer. GC-MS were recorded on a Varian CP-3800 gas chromatography unit coupled with a 2200 Saturn MS detection unit. Injection was at 100 °C with the temperature increased at a rate of 20 °C /min until 300 °C. A reverse phase column (30 M X 25 μM X 0.25 mM) was used, and mass spectrometry data was analysed with NIST 05 molecular recognition software.

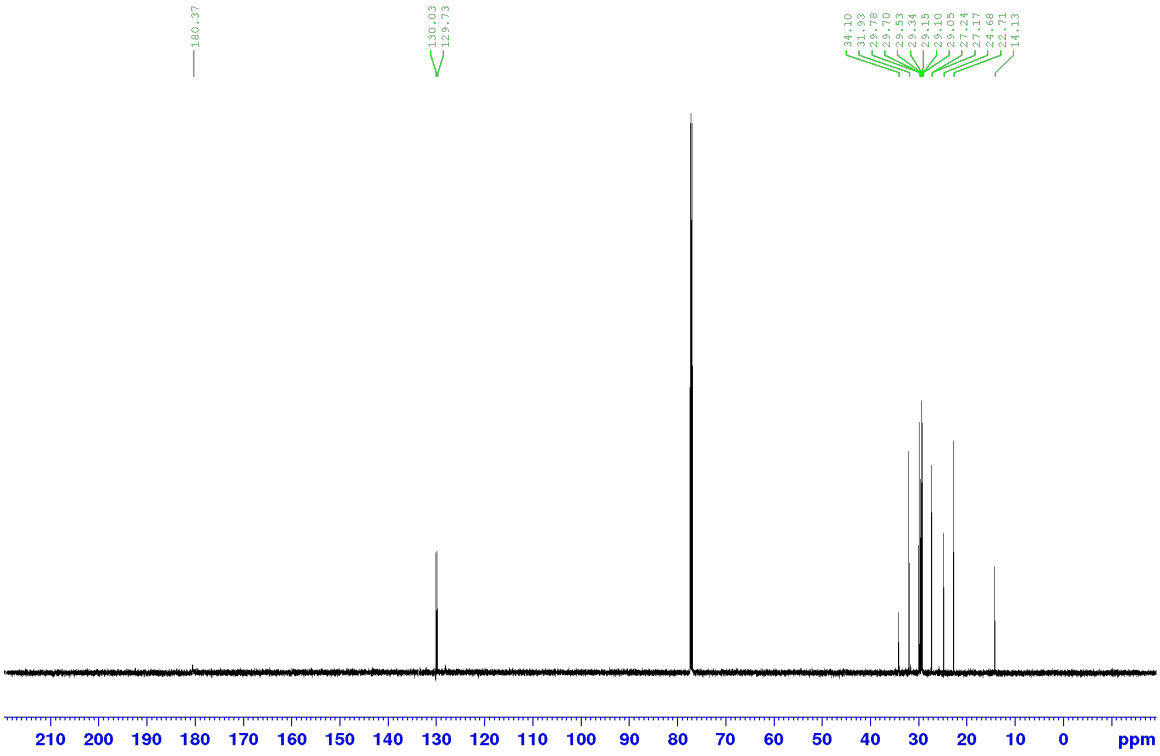
[1] H NMR of bio-oil before treatment



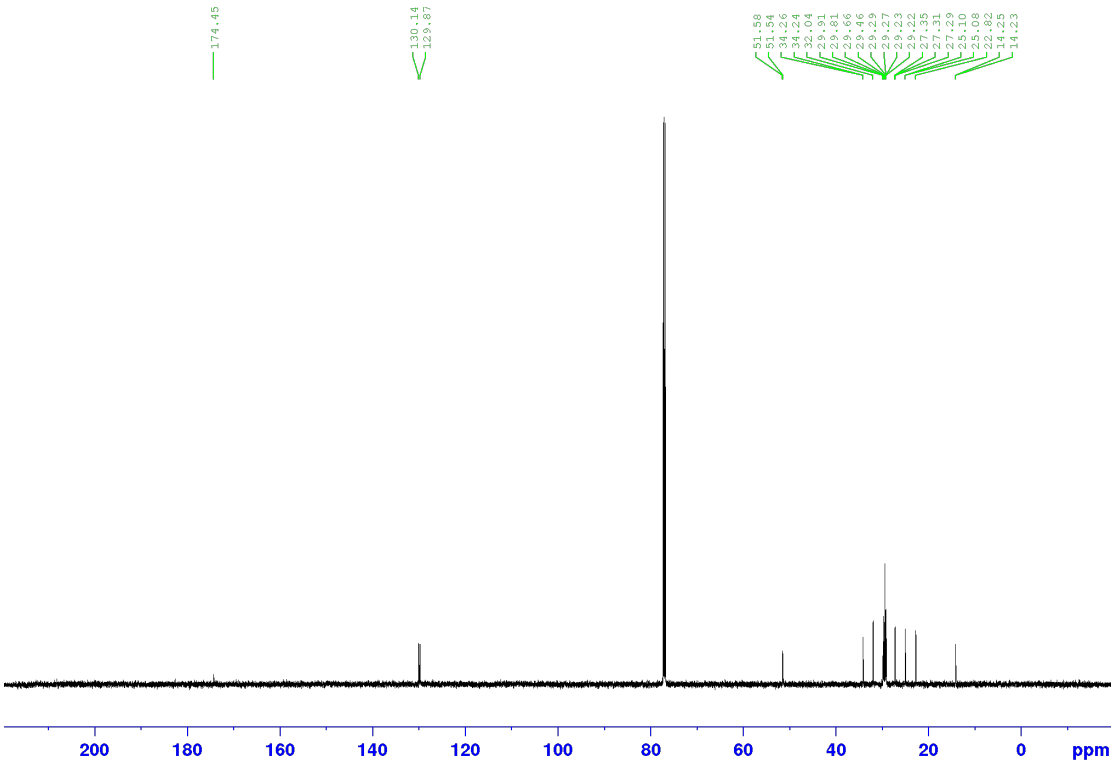
[1] H NMR of post-treated bio-oil



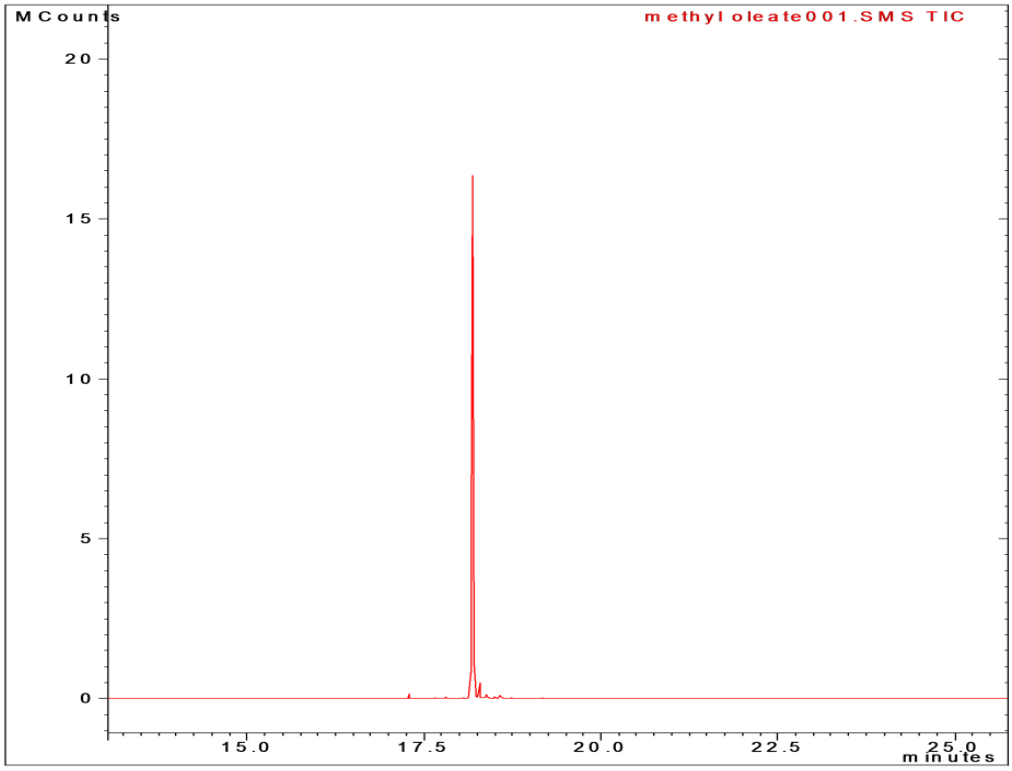
^{13}C NMR of bio-oil before treatment



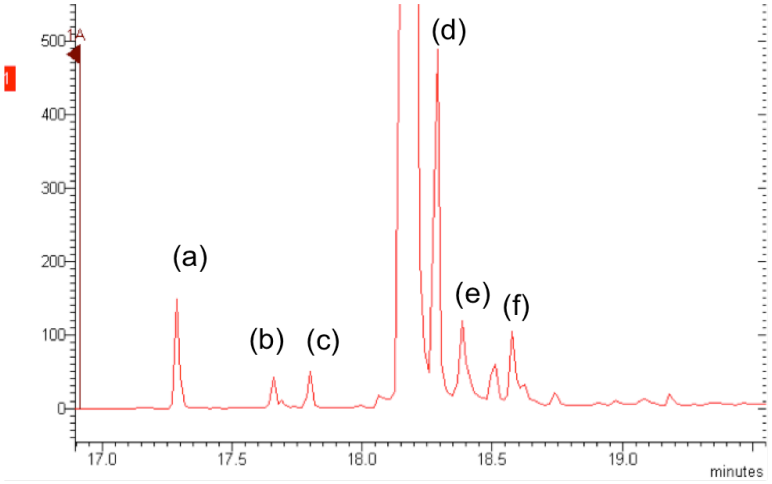
^{13}C NMR of post-treated bio-oil



GCSM of bio-oil after VFD processing



The above image displays the purity of the biodiesel synthesised using VFD flow chemistry. The main peak is the oleic acid methyl ester, with the smaller peaks being other methyl ester of lipids, as follows;



From peak left to peak right;

a) Methyl hexadecanoate – CAS :112-339-0
b) Methyl 14-methylhexadecanoate – CAS:2490:49:5
c) Methylheptadecanoate – CAS:1731-92-6
d) Methyl Stearate – CAS:112-61-8
e) 8,11-Octadecandienoic acid methyl ester – CAS :56599-58-7
f) Methyl Linoleate – CAS:112-63-0

Acid to FFA % weight calculation

$$\textbf{Methanol to FFA molar ratio}: \frac{\left(\frac{Methanol:oil_{Molar\ ratio}}{MW\ of\ oil}\right)}{\left(\frac{FFA\ \%}{MW\ FFA}\right)}$$

Methanol : bio-oil molar ratio =

60 mL of methanol - 47.508 g – 1.4828 moles

10 mL of bio-oil – 9.95 g – 0.0112375 moles

Ratio : 131.95:1

Thus $\textbf{Methanol to FFA molar ratio}: \frac{\left(\frac{131.95}{885.4}\right)}{\left(\frac{94.32}{282.46}\right)} = \mathbf{0.44628}$

$$\textbf{Acid to FFA \% weight}: \left(\frac{\textbf{Acid to oil \% weight}}{\textbf{FFA \%}}\right)$$

0.2 molar equivalents of Sulphuric Acid: 0.6992 g

Oil weight – 9.95 g

Therefore : (0.6992/9.95)* 100 = 7.027 %

Thus $\textbf{Acid to FFA \% weight}: \left(\frac{\mathbf{7.027}}{\mathbf{94.37\%}}\right) = \mathbf{0.074464}$