"Efficient Conversion of Triacylglycerols and Fatty Acids to Biodiesel in a Microwave Reactor Using Metal Triflates"

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Supplemental Information:

1. Procedure for determining reaction yield

1.1 Example of quantification of product intransesterification reaction

First, dichloromethane (DCM) was added to reach a final volume of 1 mL. Calculations were based on recovery of three equivalents of product (fatty acid methyl ester) per equivalent of starting material (triacylglycerol). Next, a 10-fold dilution of the resuspended reaction was made. Then, a subsequent dilution with a 502 μM solution of methyl stearate was made with to bring the analyte concentration within the range of the standard curve. Finally, the sample was analyzed by GC-MS and the TIC area ratio of methyl oleate to methyl sterate were compared to the midpoint of standard curve (theoretical yield) to calculate % yield.

Step 1. (35 μmol of glycerol triolein / 1 mL DCM) x 3 = 105 μmol methyl oleate / 1 mL DCM.

Step 2. 10x Dilution (100 μL product solution : 900 μL DCM) Concentration = 10.5 μmol / 1 mL DCM

Step 3. (10.5 μmol/ mL) / (0.509 μmol/ mL) = 20.6 x dilution needed (in 502 μMol methyl stearate)

Step 4. 1000 μL of a 502 μM methyl stearate solution / (20.6 – 1) = 51 μL of product from step 2 needed

Step 5. Divide GC-MS data for reaction (TIC area ratio methyl oleate / methyl state) by 0.84 (average midpoint of methyl oleate curve below) to obtain % yield.

1.2Example of quantification of product intransesterification reaction

First, dichloromethane (DCM) was added to reach a final volume of 1 mL. Calculations of esterifications were based on recovery of one equivalent of product (fatty acid methyl ester) per equivalent of starting material (free fatty acid). Next, a 10-fold dilution of the resuspended reaction was made. Then, a subsequent dilution with a 502 μM solution of methyl stearate was made with to bring the analyte concentration within the range of the standard curve. Finally, the sample was analyzed by GC-MS and the TIC area ratio of methyl oleate to methyl sterate were compared to the midpoint of standard curve (theoretical yield) to calculate % yield.

Step 1. (100 μmol of oleic acid / 1 mL DCM) = 100 μmol methyl oleate / 1 mL DCM.

Step 2. 10x Dilution (100 μL product solution : 900 μL DCM) Concentration = 10.0 μmol / 1 mL DCM

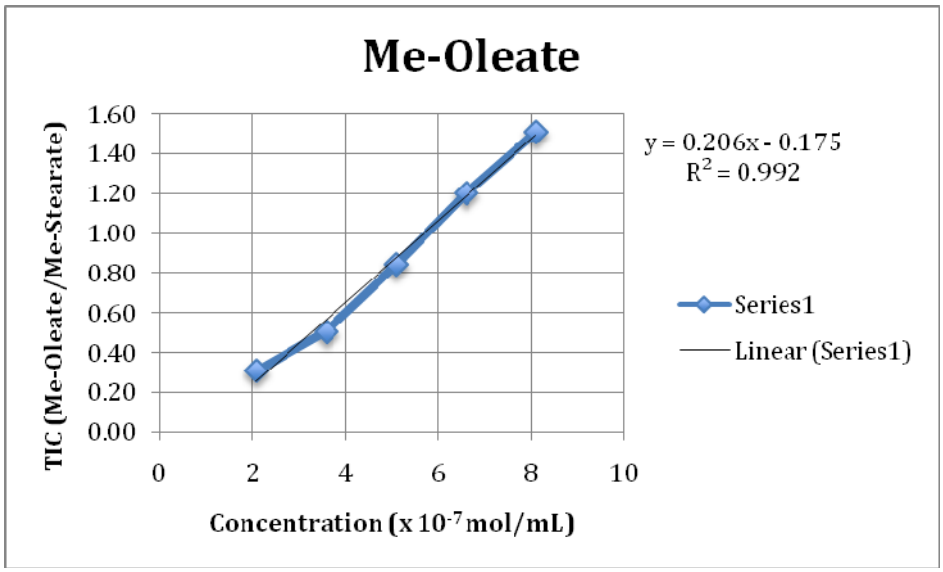
Step 3. (10.0 μmol / mL) / (0.509 μmol / mL) = 19.6 x dilution needed (in 502 μMol methyl stearate)

Step 4. 1000 μL (of 502 μMol methyl stearate) / (19.6 – 1) = 53.7 μL of product from step 2 needed.

Step 5. Divide GC-MS data for reaction (TIC area ratio methyl oleate / methyl state) by 0.84 (average midpoint of methyl oleate curve below) to obtain % yield.

2. Calibration Curves

2.1 Methyl Oleate



Me-Oleate

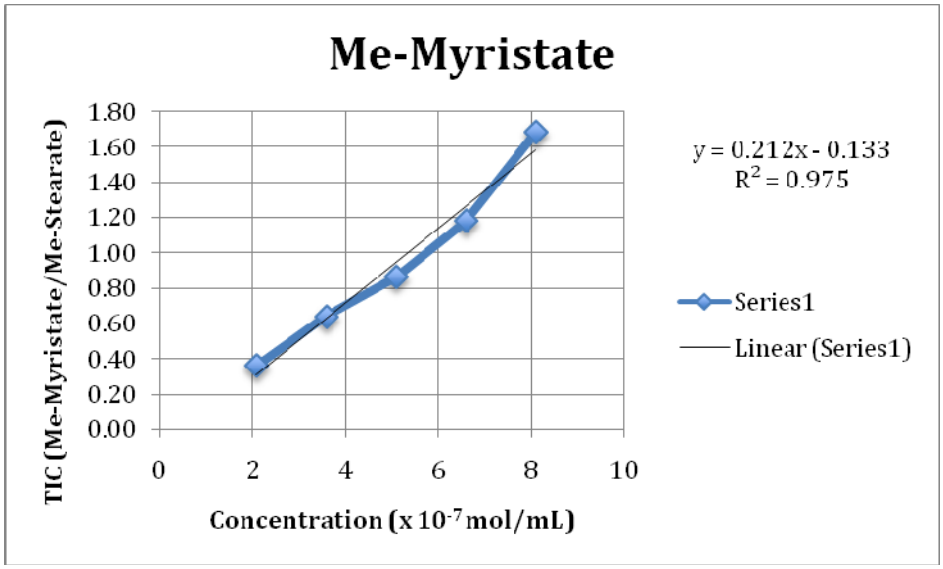
Conc.	Curve 1	Curve 2	Curve 3	Conc.	Ave
2.09	0.29	0.285	0.35	2.09	0.31
3.59	0.567	0.375	0.576	3.59	0.51
5.09	0.904	0.857	0.77	5.09	0.84
6.59	1.17	1.31	1.12	6.59	1.20
8.09	1.41	1.4	1.71	8.09	1.51

5.09 = .873 Error = 3.9 %

Percent Error = (0.873-0.84)/0.84

Percent Error = (estimate-actual)/actual

2.1 Methyl Myristate



Me-Myristate

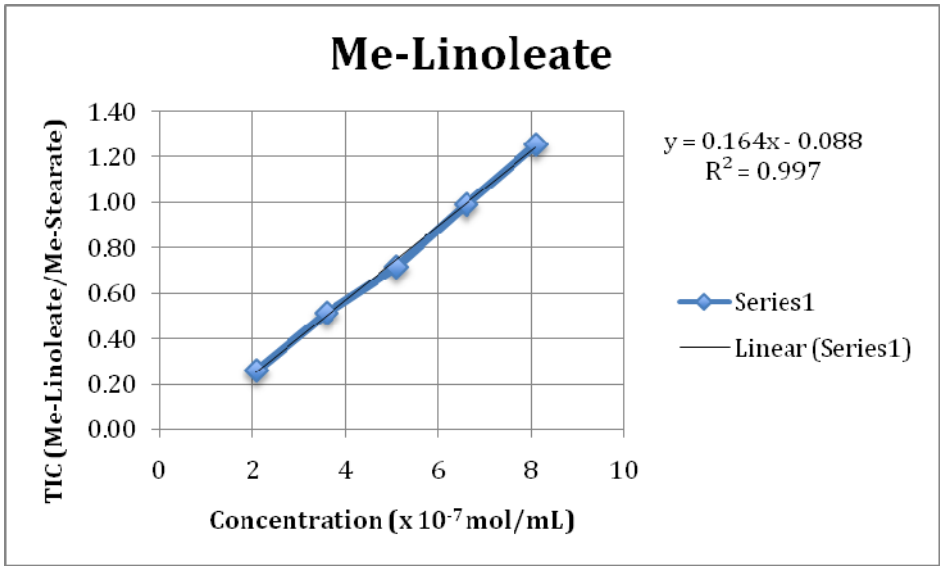
Conc.	Curve 1	Curve 2	Curve 3	Conc.	Ave
2.09	0.348	0.38	0.357	2.09	0.36
3.59	0.612	0.671	0.643	3.59	0.64
5.09	0.839	0.848	0.913	5.09	0.87
6.59	1.21	1.22	1.12	6.59	1.18
8.09	1.81	1.69	1.55	8.09	1.68

5.09 = .946 Error = 8.7 %

Percent Error = (0.946-0.87)/0.87

Percent Error = (estimate-actual)/actual

2.1 Methyl Linoleate



Me-linoleate

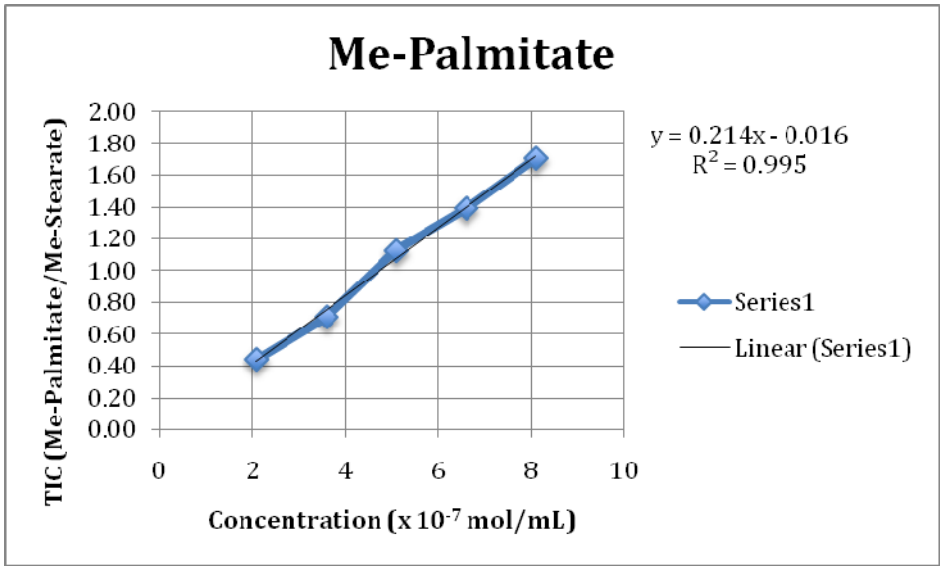
Conc.	Curve 1	Curve 2	Curve 3	Conc.	Ave
2.09	0.24	0.25	0.29	2.09	0.26
3.59	0.47	0.55	0.524	3.59	0.51
5.09	0.63	0.742	0.779	5.09	0.72
6.59	0.92	1.07	0.979	6.59	0.99
8.09	1.32	1.15	1.29	8.09	1.25

5.09 = .746 Error = 3.6 %

Percent Error = (0.746-0.72)/0.72

Percent Error = (estimate-actual)/actual

2.1 Methyl Palmitate



Me-Palmitate

Conc.	Curve 1	Curve 2	Curve 3	Conc.	Ave
2.09	0.48	0.41	0.43	2.09	0.44
3.59	0.74	0.63	0.75	3.59	0.71
5.09	0.98	1.32	1.08	5.09	1.13
6.59	1.55	1.31	1.32	6.59	1.39
8.09	1.74	1.71	1.66	8.09	1.70

5.09 = 1.07 Error = 5.3 %

Percent Error = (1.07-1.13)/1.13

Percent Error = (estimate-actual)/actual