

REFERENCES

- Boey, P.L., Maniam, G.P., Hami, S.A., (2009) Biodiesel production via transesterification of palm olein using waste mud crab (*Scylla serrata*) shell as a heterogeneous catalyst. Bioresource Technology, 100, 6362–6368.
- Boßelmann, F., Romanob, P., Fabritius H., Raabe, D., and Epple, M. (2007) The composition of the exoskeleton of two crustacea: The American lobster *Homarus americanus* and the edible crab *Cancer pagurus*. Thermochemica Acta, 463, 65–68.
- Canakci, M. and J. Van Gerpen, (2001) Biodiesel Production from Oils and Fats with High Free Fatty Acids. Trans. ASAE, 44, 1429.
- Freedman, B., Butterfield, R.O., and Pryde, E.H. (1986) Transesterification kinetics of soybean oil. Journal of the American Oil Chemist's Society, 63, 1375-1380.
- Galtsoff, P.S., (1964). The American oyster, *Crassostrea virginica* Gmelin. Fishery Bulletin, U.S. fish and wildlife services, 64, 1–480.
- Gelbard G., Bres O., Vargas R.M., Vielfaure F., and Schuchardt U.F., (1995). ¹H nuclear magnetic resonance determination of the yield of the transesterification of rapeseed oil with methanol. Journal of the American Oil Chemist's Society, 72, 1239–1241.
- Goff, M. J., N. S. Bauer, S. Lopes, W. R. Sutterlin, and G. J. Suppes, (2004) Acid-Catalyzed Alcoholysis of Soybean Oil, *J. Am. Oil Chem. Soc.*, 81, 415.
- Knothe, G., (2005) Dependence of biodiesel fuel properties on the structure of fatty acid alkyl esters. Fuel Processing Technology, 86, 1059– 1070.
- Liu X., He H., Wang Y., and Zhu S., (2007) Transesterification of soybean oil to biodiesel using SrO as a solid base catalyst, Catalysis Communications, 8, 1107–1111.
- Liu, X., He, H., Wang. Y., Zhu, S., and Piao. X. (2008) Transesterification of soybean oil to biodiesel using CaO as a solid base catalyst. Fuel, 87, 216–221.

- MacLeod, C.S., Harvey, A.P., Lee, A.F., and Wilson, K. (2008) Evaluation of the activity and stability of-alkali-doped metal oxide catalysts for application to an intensified method of biodiesel production. Chemical Engineering Journal, 135, 63–70.
- Marchetti J.M., Miguel V.U., Errazu A.F., (2007) Heterogeneous esterification of oil with high amount of free fatty acids, Fuel, 86, 906–10.
- Meher , L.C., Vidya, S. Dharmagadda, S., and Naik, S.N., (2006) Optimization of alkali-catalyzed transesterification of Pongamia pinnata oil for production of biodiesel, Bioresource Technology, 97, 1392–1397.
- Ngamcharussrivichai, C., Totarat, P., and Bunyakiat, K. (2008) Ca and Zn mixed oxide as a heterogeneous base catalyst for transesterification of palm kernel oil, Applied Catalysis A: General, 341, 77–85.
- Phan, A.N., Phan, T.M. (2008) Biodiesel production from waste cooking oils. Fuel, 87, 3490–3496.
- Rødde, R.H., Einbu, A., and Varum, K.M. (2008) A seasonal study of the chemical composition and chitin quality of shrimp shells obtained from northern shrimp (*Pandalus borealis*). Carbohydrate Polymers, 71, 388–393.
- Schuchardt U.F., Serchelia, R., and Vargas R.M., (1998). “Tranesterification of vegetable oils: a review.” Journal of the Brazilian Chemical Society, 9, 199–210.
- Saetang. R. (2008) Evaluation of Heterogeneous Catalyst for Biodiesel Production. Master Thesis. The Petroleum and Petrochemical College. Bangkok, Chulalongkorn University.
- Stadelman, W.J., (2000). Eggs and egg products. In: Francis, F.J. (Ed.), Encyclopedia of Food Science and Technology, second ed. John Wiley and Sons, New York, 593–599.
- Ting, W.J., Huang, C.M., Giridhar, N., Wu, W.T. (2008) An enzymatic/acid-catalyzed hybrid process for biodiesel production from soybean oil. Journal of the Chinese Institute of Chemical Engineers, 39, 203–210.

- Viriya-empikul, N., Krasae, P., Puttasawat, B., Yoosuk, B., Chollacoop, N., Faungnawakij, K. (2010) Waste shells of mollusk and egg as biodiesel production catalysts. Bioresource Technology, 101, 3765–3767.
- Wei, Z., Xu, C., and Baoxin Li, B. (2009) Application of waste eggshell as low-cost solid catalyst for biodiesel production. Bioresource Technology, 100, 2883–2885.
- Wheaton, F. (2007) Review of the properties of Eastern oysters, *Crassostrea virginica* Part I. Physical properties. Aquacultural Engineering, 37, 3–13.

APPENDICES

Appendix A Temperature Controller Calibration

Two types of heating tapes were evaluated to establish the actual heating rate used during the transesterification reaction for each of the experimental conditions. The heating tapes were connected to the Digi-Sense® Temperature Controller, which was set up with the ramp/soak mode at a heating rate of $5^{\circ}\text{C}\cdot\text{min}^{-1}$.

Figure A1 shows the temperature profile as a function of time for heating tape I, set up at a maximum temperature of 45°C . This graph shows that the actual temperatures measured by the thermocouple (square symbols) as a function of time are different from the temperature profile set up at the temperature controller (cross symbol). A maximum temperature overshoot of 52°C was observed for few minutes followed by a progressive reduction until the temperature reaches 45°C .

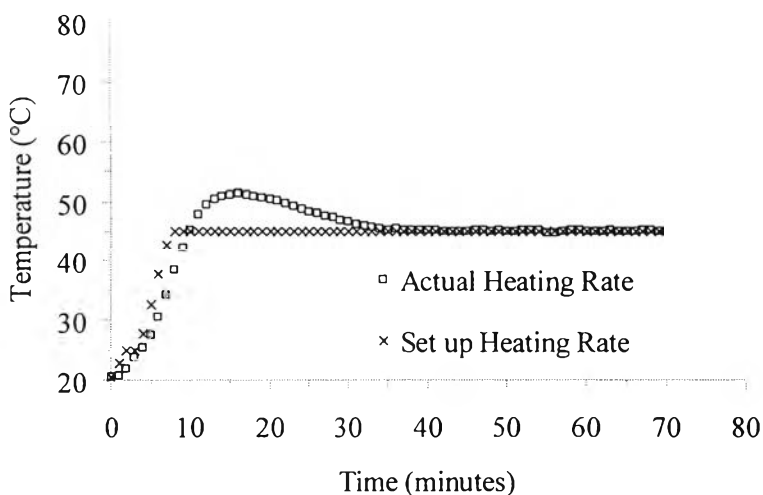


Figure A1 Temperature profile obtained for heating tape I for a setting temperature of 45°C . The square symbols represent the actual temperature and cross symbols represent the set temperature in the temperature controller.

Figures A2 and A3 show the same behaviour observed previously. The heating tape response indicates that temperature overshoot takes place for few minutes

and then, it levels off to the set temperature. The same procedure was repeated using the heating tape II to determine the heating rate.

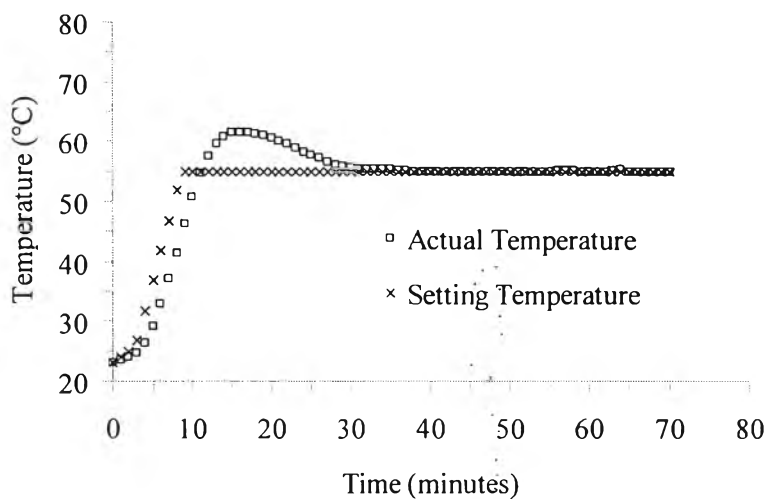


Figure A2 Temperature profile obtained for heating tape I for a setting temperature of 55°C. The square symbols represent the actual temperature and cross symbols represent the set temperature in the temperature controller.

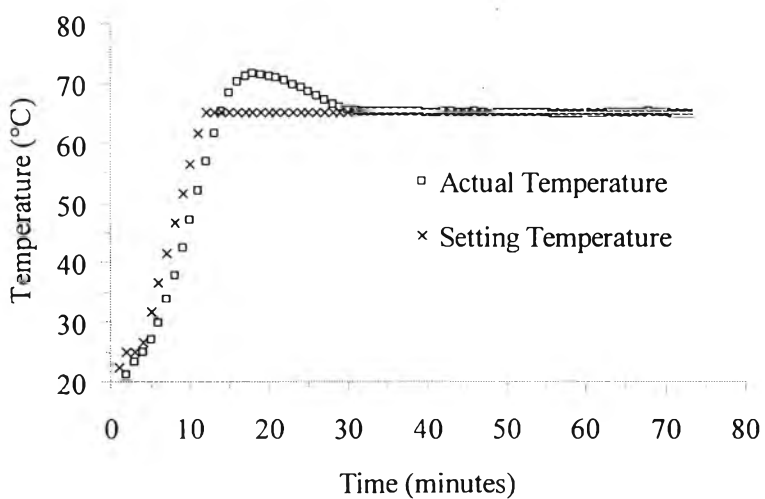


Figure A3 Temperature profile obtained for heating tape I for a setting temperature of 65°C. The square symbols represent the actual temperature and cross symbol represent the set temperature in the temperature controller.

Figures A4 to A6 indicate that in all cases, temperature overshooting takes place. However, heating tape type II shows lower temperature overshooting at the same heating rate compared to type I. Consequently, heater type II was chosen to conduct the experimental work to ensure a lower temperature overshooting during the reaction period.

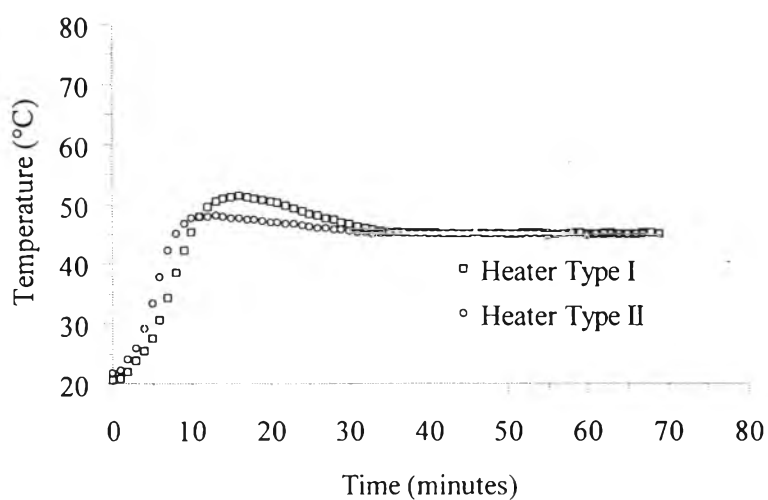


Figure A4 Comparison between type I and type II actual temperature profile for heating tape type I and type II at 45°C at the heating rate of 5°C.min⁻¹. The square symbols and diamond symbols represent the temperature profile of Heater Type I and Type II respectively.

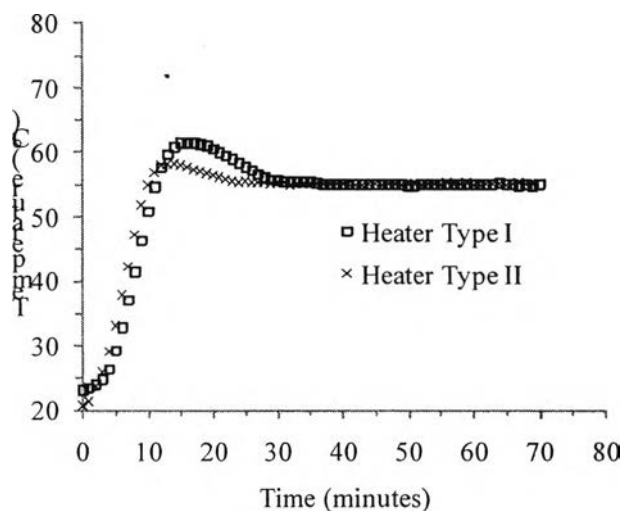


Figure A5 Comparison between type I and type II actual temperature profile for heating tape type I and type II at 55°C and at a heating rate of 5°C.min⁻¹. The squares symbols and cross symbols represent the temperature profile of Heater Type I and Type II respectively.

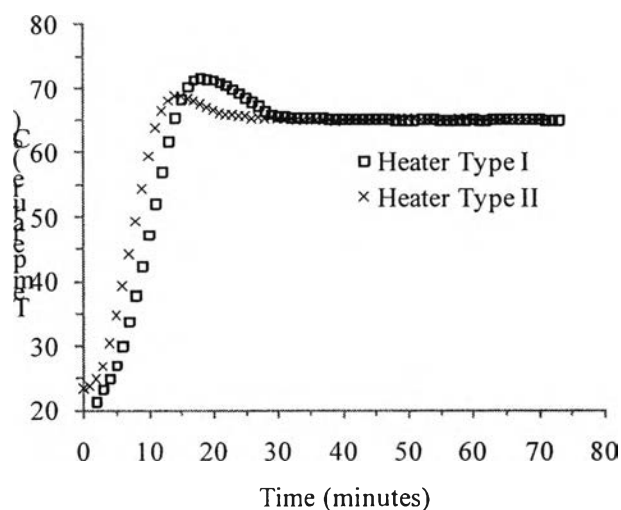


Figure A6 Comparison between type I and type II actual temperature profile for heating tape type I and type II at 65°C at a heating rate of 5°C.min⁻¹. The squares symbols and cross symbols represent the temperature profile of Heater Type I and Type II respectively.

Appendix B Calibration of $^1\text{H-NMR}$ for biodiesel yield determination

A calibration curve was obtained for reliable results from $^1\text{H-NMR}$ analysis. A biodiesel standard, which contains 99.8% of fatty acid methyl ester derived from canola oil, was used and mixed with consumable grade canola oil to get a series of biodiesel concentrations as follows: B0 which is canola oil at 100%wt concentration, B20 which has 20%wt of biodiesel standard mixed with 80%wt of canola oil, B40 which has 40%wt of biodiesel standard mixed with 60%wt of canola oil, B50 which has 50%wt of biodiesel standard mixed with 50%wt canola oil, B60 which has 60%wt of biodiesel standard mixed with 40%wt canola oil, B80 which has 80%wt of biodiesel standard mixed with 20%wt of canola oil, and B100 which contains 100%wt of the biodiesel standard. The actual weight of each runs were calculated and compared to the results obtained from $^1\text{H-NMR}$ analysis.

A biodiesel standard, which contains 99.8% of fatty acid methyl ester derived from canola oil, was used and mixed with consumable grade canola oil to get a series of biodiesel concentrations as shown in Table A1.

Table B1 Actual compositions of biodiesel standard for yield determination by $^1\text{H-NMR}$ analysis calibration

	Weight of (g)		Total (g)	Actual % of B100
	B100	Canola Oil		
B0	0	1.002	1.002	0.00
B20	0.201	0.800	1.001	20.08
B40	0.403	0.598	1.001	40.26
B50	0.502	0.503	1.005	49.95
B60	0.604	0.400	1.004	60.16
B80	0.800	0.203	1.003	79.76
B100	1.006	0	1.006	100.00

Table B2 Percentage of biodiesel yield obtained from $^1\text{H-NMR}$ analysis

	%Yield calculated from NMR		Average
	Run 1	Run 2	
B0	0	0	0
B20	18.99	19.13	19.06
B40	39.49	39.72	39.60
B50	48.63	49.23	48.93
B60	61.37	61.55	61.46
B80	79.83	80.42	80.12
B100	100.63	100.45	100.54

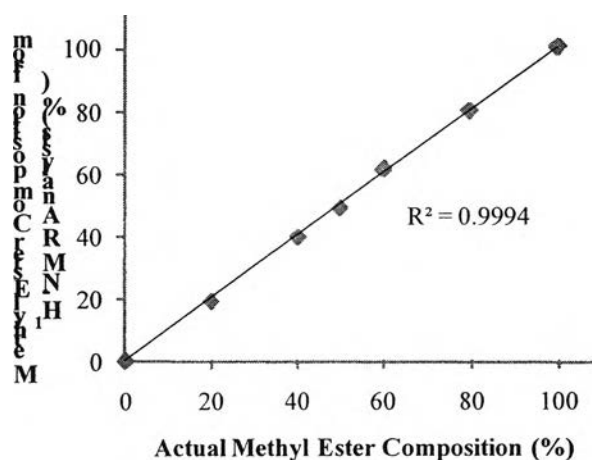
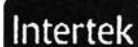
**Figure B1** Biodiesel standard calibration curve obtained from $^1\text{H-NMR}$ analysis.

Figure B1 shows the biodiesel concentration calibration curve. The biodiesel yield calculated from $^1\text{H-NMR}$ analysis corresponded to the actual fatty acid methyl ester contained in the mixture. The calibration curve shows a coefficient of determination of $R^2 = 0.9994$. The mixture B0 does not show an ester peak in the $^1\text{H-NMR}$ spectrum because it contains only pure canola oil. The other biodiesel standard samples: B20, B40, B50, B60, B80, and B100 showed the following biodiesel concentrations 19.104%, 39.603%, 48.928%, 61.460%, 80.124%, and 100.473% respectively.

Appendix C Analytical Data for B100 Used for $^1\text{H-NMR}$ Analysis Calibration Obtained from the Third Party Company



Report of Analysis

Client: Eastern Greenway Oils Inc.
14270 Route 2
Waterville, Carleton County
New Brunswick, Canada
E7P 1C4

Lab Report no.: CA120-4241-2
Report date: March 3, 2008

Submitted on: February 20, 2008
Tested on: February 29, 2008
Customer Product
Description: Biodiesel
Sample identification: B-100 ??

Attention: Brian Antworth

TESTS	UNITS	METHODS	SPECIFICATIONS ⁽¹⁾	RESULTS
Paraffins	% wt	GCMS Confirmation	---	< 1.0
Fatty Acid Methyl Esters (FAME)	% v/v	EN 14078	---	99.8

(1) Specifications not provided by the client

Megan Clarke

Mikhar Chughtai, Laboratory Manager

The information contained herein is based on laboratory tests and observations performed by Intertek Caleb Brett. This sample (or these samples) was or were submitted by the client solely for testing. Intertek Caleb Brett disclaims any and all liability for damage or injury which results in the use of the information contained herein; and nothing contained herein shall constitute a guarantee, warranty or representation by Intertek Caleb Brett with respect to the accuracy of the information, the sample, products or items described, or their suitability for use for any specific purpose. This report is for the exclusive use of the client and may only be reproduced in full by written permission of Intertek Caleb Brett. Unless otherwise instructed, all samples pertaining to this report will be discarded 60 days after the issuing date of the report.

Intertek Caleb Brett
651 Burlington Street East, Hamilton, Ontario, Canada L8L 4J5
Telephone 905-529-0090 Fax 905-529-5989 e-mail aison.gee@intertek.com

Figure C1 Report of analysis for B100 from Interlek

CURRICULUM VITAE



Name: Mr. Naravit Leaukosol

Date of Birth: November 6, 1984

Nationality: Thai

University Education:

2003–2006 Bachelor of Science Degree Program in Industrial Chemistry
at Chiangmai University, Chiangmai, Thailand

Bachelor's thesis: *On the Gas Fluidization of Homogeneous Bed Expansion*

Proceedings:

1. Leaukosol, N.; Rirksomboon, T.; Romero-Zerón, L.; Jongpatiwut, S.; and Steward, F.R. (2010, April 22) Formulation of Heterogeneous Catalysts from Natural and Synthetic Materials for Biodiesel Production. Proceedings of the 16th PPC Symposium on Petroleum, Petrochemical, and Polymers, Bangkok, Thailand.

Presentations:

1. Leaukosol, N.; Rirksomboon, T.; Romero-Zerón, L.; Jongpatiwut, S.; and Steward, F.R. (2009, May 15) Biodiesel Production from Canola Oil Using Heterogeneous Catalysts. Poster presentation at the Annual Graduate Student Conference 2009, (Best Poster Presentation Award), Fredericton, New Brunswick, Canada.
2. Leaukosol, N.; Rirksomboon, T.; Romero-Zerón, L.; Jongpatiwut, S.; and Steward, F.R. (2010, April 22) Formulation of Heterogeneous Catalysts from Natural and Synthetic Materials for Biodiesel Production. Poster presentation at the 16th PPC Symposium on Petroleum, Petrochemical, and Polymers, Bangkok, Thailand.