Newcastle University
School of Engineering

ISSUE 8 | December 2017/January 2018

pig MONTHLY

process intensification group | www.pig.ncl.ac.uk | www.pinetwork.org

In This Issue

New Group Members

- Following the recent additions of John Dalton, Dr Chris O’Malley, Dr Mark Willis and Dr Jie Zhang to the group (following the formation of the new School of Engineering at Newcastle University), the PIG would like to officially welcome their associated students, visitors and researchers (project titles can be found on the *website*):
  - ***Prof Tinuade Afolabi*** (Visitor) | working with Dr Zhang
  - ***Dr Victor Grisales Diaz*** (Research Associate) | working with Dr Willis
  - ***Kazeem Alli*** (PhD Student) | supervised by Dr Zhang
  - ***Jeremiah Corrigan*** (PhD Student) | supervised by Dr Zhang
  - ***Chris Dixon*** (PhD Student) | supervised by Dr Willis and Dr O’Malley
  - ***Harry Laing*** (PhD Student) | supervised by Dr Willis and Dr O’Malley
  - ***Kai Liu*** (PhD Student) | supervised by Dr Zhang
  - ***Wissan Muhsin*** (PhD Student) | supervised by Dr Zhang
  - ***Kirstie Pemberton*** (EngD Student) | supervised by Dr O’Mal-ley (2nd sup)
  - ***Rousen Qi*** (PhD Student) | supervised by Dr Zhang
  - ***Paulius Rasiukas*** (PhD Student) | supervised by Dr Willis & Dr O’Malley
  - ***Shengkai Wang*** (PhD Student) supervised by Dr Zhang
  - ***Peng Zhang*** (PhD Student) supervised by Dr Zhang
  - ***Changhao Zhu*** (PhD Student) supervised by Dr Zhang
- The PIG would also like to welcome the following new PhD students who started in January 2018:
  - ***Abdullahi Adamu***, who is working with Dr Boodhoo on “Photocatalytic CO_2 reduction for chemical and fuel production petroleum technology”
  - ***Olatunde Akinbuja***, who is working with Dr Velasquez-Orta on “Algae-based microbial fuel cells for pollution conversion and monitoring”
- The group now comprises 15 academics, 10 research associates and 45 postgraduate research students.

Upcoming Conferences

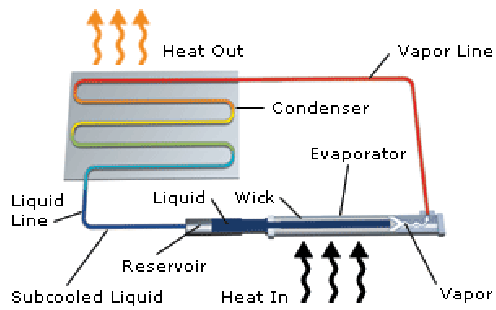
- **ChemEngDayUK2018** (27-28 March 2018, Leeds, UK). Early Bird Registration Deadline: 26th Feb 2018
- **2018 *AIChE* Spring Meeting** (22-26 April 2018, Orlando, Florida). Early Bird Registration Deadline: 12th March 2018
- **25th International Symposium on Chemical Reaction Engineering, ISCRE25** (20-23 May 2018, Florence, Italy). Early Registration Deadline: End of March 2018
- **8th European Meeting on Chemical Industry and Environment (EMChIE)** (13-15 June 2018, Nantes, France). Early Registration Deadline: 30th March 2018
- **16th International Heat Transfer Conference** (10-15 August 2018, Beijing, China). Registration Deadline: 30th June 2018
- **6th International Congress on Green Process Engineering (GPE)** (3-6 June 2018, Toulouse, France). Final Abstract Deadline: 2nd Feb 2018
- **X Minsk International Seminar: *Heat Pipes, Heat Pumps, Refrigerators, Power Sources*** (10-13 Sep 2018, Minsk, Belarus). Abstract Deadline: 28th Feb 2018
- **6th International Conference on Micro and Nano Flows, MNF2018** (9-12 Sep 2018, Atlanta, Georgia, US). Abstract Deadline: 30th March 2018
- **2018 *AIChE* Annual Meeting** (28 Oct-2 Nov 2018, Pittsburgh, PA, US). Abstract Deadline: 18th April 2018

PIG News

- Dr Thea Ekins-Coward recently joined a new project with Dr Boodhoo and Dr Velasquez-Orta: "Scalable engineering approaches for exploiting a novel biocomposite material applied to light-driven CO_2 absorption and utilization". The project is part of *C1Net*, one of 13 projects currently funded by BBSRC NIBB (Networks in Industrial Biotechnology and Bioenergy)
- Congratulations to James Hendry, who passed his EngD viva on 2nd February 2018. His thesis was titled: "Computational Fluid Dynamic Modelling of Benzene Abatement using Cryogenic Condensation"
- Congratulations to Jonathan McDonough, who passed his PhD viva (with no corrections) on 13th December 2017. His thesis was titled: "Process Development Using Oscillatory Baffled Mesoreactors"
- Several researchers from the group have recently had papers accepted for oral/poster presentations at the upcoming AIChE Spring conference and 14th Global Congress on Process Safety in Orlando (April 2018):
  - K.V.K. Boodhoo, A. Phan, V. Zivkovic, V. Eze, A. Mustaffar. *"Intensified by Design (IbD): Creating a Platform for Facilitating Process Intensification in Solids Handling Applications"* [Oral Presentation]
  - A. Mustaffar, A. Phan, K.V.K. Boodhoo. *"Hybrid Heat Pipe Screw Dryer: A Novel, Continuous and Highly Energy-Efficient Drying Technology"* [Oral Presentation]
  - A. Mustaffar, A. Phan, K.V.K. Boodhoo. *"Bi-Directional Thermal Control of Twin Screw Granulation Process Via a Specialised Annular Heat Pipe"* [Poster Presentation]
- On 30th January, two visitors from Torftech (Dan Groszek and Martin Groszek) met with David, Vlad, Richard and Jonathan to discuss the use of toroidal fluidized bed (Torbed®) technology in their EPSRC funded carbon capture project (see *issue 7* of the PIG newsletter for further details). This included discussions of material requirements for pilot-scale testing and data collection for rough estimations of equipment size/costs. Another possibility discussed was the use of 3D printing for the fabrication of small scale Torbed® units for sorbent screening. Finally, the opportunity was taken to discuss the results of a Torbed® used within the 'Intensified by Design' (IbD) HORIZON 2020 project in the context of its ability to handle solid particles, in this case for drying a pharmaceutical intermediate. A description of the Torbed® and its applications is available on the PIN website (24th PIN meeting, May 2015), where the *overheads* relating to a talk by Dan Groszek are presented.

Fully Funded PhD Project

Industrial Cooperative Awards in Science & Technology (CASE) provide PhD studentship funding through the EPSRC, where businesses take the lead in arranging projects with an academic partner of their choice. There is currently three years of funding available from this scheme for the following project: "*Next generation loop heat pipe wick technology for thermal management of Space and Terrestrial Applications*", a summary of which can be found *here*. This project will involve working at both Newcastle University and Aavid Thermacore (situated in Ashington, ~30 miles north of Newcastle). The project will be supervised at Newcastle University by Dr Richard Law and Prof David Reay, while Dr Ryan McGlen will supervise the work at Thermacore. Anyone interested in applying should contact Richard directly: richard.law2@ncl.ac.uk.



Aavid Thermacore, Ashington, UK

PIN & HEXAG Meetings

The date for the next ***Heat EXchanger Action Group (HEXAG)*** meeting is Tuesday 15th May 2018. The ***Process Intensification Network (PIN) meeting*** will be on the following day, Wednesday 16th May 2018. Both meetings will be held in the Research Beehive in the Old Library Building at Newcastle University. Please contact David Reay (DAReay@aol.com or David.Reay@newcastle.ac.uk) if you would like to present a talk.

Other Information

- Full contact details and research profiles for the PI group members can be found at the website: *http://pig.ncl.ac.uk*
- For enquires about collaborations or PhD study, see the website: *http://pig.ncl.ac.uk*
- If anyone would like to contribute any articles, or if anyone has any ideas regarding the newsletter please contact Jonathan McDonough: jonathan.mcdonough@ncl.ac.uk

Upcoming PIG Seminars

- Friday 9th Feb
  Valentine Eze
  TBD
- Friday 23rd Feb
  Abdul Rehman
  TBD
- Friday 2nd March
  Jonathan McDonough
  TBD
- Friday 9th March
  Muayad Al-Karawi
  TBD
- Friday 16th March
  Safaa Ahmed
  TBD
- Friday 23rd March
  Eka Utuk
  TBD

New Publications

- C. Ahoba-Sam, K.V.K. Boodhoo, U. Olsbye, K.J. Jens. Tailoring Cu Nanoparticle Catalyst for Methanol Synthesis Using the Spinning Disk Reactor. Materials 11(1) (2018) 154
- X. Christodoulou, T. Okoroafor, S. Parry, S.B. Velasquez-Orta. The use of carbon dioxide in microbial electrosynthesis: Advancements, sustainability and economic feasibility. Journal of CO_2 Utilization 18 (2017) 390-399
- T. Coward, H. Tribe, A.P. Harvey. Opportunities for process intensification in the UK water industry: A review. Journal of Water Process Engineering 21 (2018) 116-126
- R. Law, C. Ramshaw, D. Reay. Process intensification - Overcoming impediments to heat and mass transfer enhancement when solids are present, *via* the IbD project. Thermal Science and Engineering Progress 1 (2017) 53-58
- F. Li, J. Zhang, C. Shang, D. Huang, E. Oko, M. Wang. Modelling of a post-combustion CO_2 capture process using deep belief network. Applied Thermal Engineering 130 (2018) 997-1003
- O.L. do Nascimento, D. Reay, V. Zivkovic. Study of transitional velocities of solid-liquid micro-circulating fluidised beds by visual observation. Journal of Chemical Engineering of Japan (2018). Article in-press
- A. Umar, G. Caldwell, J.G.M. Lee. Foam flotation can remove and eradicate ciliates contaminating algae culture systems. Algal Research 29C (2018) 337-342

Recent PIG Seminars

- ***Luma Al-Saadi*** | 8th Dec
  "A study of process conditions required for high conversion of high free fatty acid/water triglyceride feedstocks to biodiesel"
- ***James Hendry*** | 15th Dec
  "Pressure Drop and Flooding in a Rotating Packed Bed"
- ***Akmal Bin Abdul Rahim*** | 12th Jan
  "Intensification of Epoxidation of Vegetable Oil"
- ***Long Duong*** | 12th Jan
  "Upgrading bio-oil from pyrolysis of biomass"
- ***Yi Zhang*** | 19th Jan
  "Development of a Miniaturised Three-Phase Fluidized Bed Bioreactor"
- ***Zakariah Adu Adejoh*** | 19th Jan
  "Particle Emissions from Biomass Boilers and Their Mitigation"
- ***Sahr Sana*** | 26th Jan
  "Solvent-antisolvent precipitation of starch nanoparticles in a spinning disc reactor"
- ***Faisal Saleem*** | 2nd Feb
  "Effect of carrier gas on the removal of a tar analogue in dielectric-barrier discharge reactor"

Biodiesel

Prof Adam Harvey

The PI group is well known for its work in developing new biofuel processes, particularly for the production of biodiesel. Indeed, it turns out that almost 40% of my publications have the word "biodiesel" in them somewhere! I started work on biodiesel as a postdoctoral researcher in Cambridge, when it was suggested that the biodiesel reaction would be a good fit for the oscillatory baffled reactor. Indeed it was, and we discovered that the typical reaction time in industry, of 1 to 2 h was far too long, and that we could produce it at the required conversion in 15 minutes. We have since gone well beyond this to demonstrate methods by which the reaction can be reduced to 2 min, but we have also investigated a range of other elements of the biodiesel process, including production of biodiesel directly from oilseeds and algae ("reactive extraction"), and a range of other feedstocks, catalyst development, and glycerol valorisation (particularly *in situ*). The brief descriptions following should give an idea of the breadth of research that has now developed within the group (and I count at least 12 graduated PhDs who have worked on biofuels in the PIG):

In situ transesterification of rapeseed for the production of biodiesel and secondary products

Akeem Babatunde, PhD, 2013-2017

This project centred on combining reactive extraction of oilseed with reactive coupling, to convert the glycerol by-product into polyglycerol *in situ*. The rationales for the combination were:

- Higher biodiesel yield due to removal of glycerol by product, which should push the transesterification reaction to the product side according to Le Chatelier's principle
- Lower methanol requirement because of the removal of glycerol
- Increased rate of reaction; 'rapid' biodiesel production as observed for the conventional reaction
- Waste product (glycerol) converted to a value added product: polyglycerol
- Reduced processing steps to reduce the overall capital cost of production

Literature screening identified the polyglycerol reaction as the best candidate for a complementary reaction. "Fast reactive extraction" was performed on rapeseeds, which achieved 90% FAME yield within 5 min of reaction. These *in situ* transesterifications were monitored online for the first time via FTIR. Co-production of dimethyl ether (DME) was demonstrated.

Overall, the basis of a biodiesel-based refinery with biodiesel, polyglycerol, DME and animal feed meal as products was established. However, these are not the only products because the waste streams are yet to be fully optimised. These areas are currently the subject of further investigation by Ibrahim Mohammed, who began his studies in 2017.

Reactive coupling of biodiesel with poly glycerol production

Ibrahim Mohammed, PhD, 2016-Present

The commercial production of biodiesel is on the increase. However, 10-18 wt% of the reactor product is crude. The crude glycerol might contain the catalyst used during the reaction, water reaction product, unreacted alcohol and triglyceride.

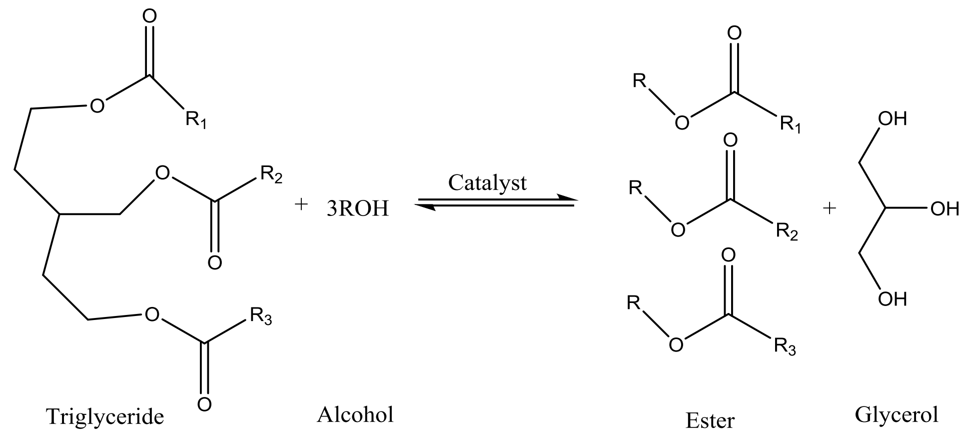


Figure 1. Transesterification of triglyceride with an alcohol

The applications of pure glycerol in food, cosmetics and pharmaceutical industries among others also increases with the demands of the products. However, the glycerol formed during biodiesel production requires various steps of purification before the coproduct will have an appreciable market value. The crude glycerol, rather than purification due to high cost and time consumption, has found other process that can convert it to added values chemicals. Among them is dehydration to form acrolein, fermentation to produce alcohol, digestion for biogas production, pyrolysis to biochar, steam reforming for hydrogen production and etherification for polyglycerol production. The application of polyglycerols in food, pharmaceutical, biomedical, cosmetics and drug industries among others has make it significant in the science and engineering of polymers. It is produced by the condensation of two or more glycerol molecules with catalyst to produce the oligomers of glycerol and water as a coproduct.

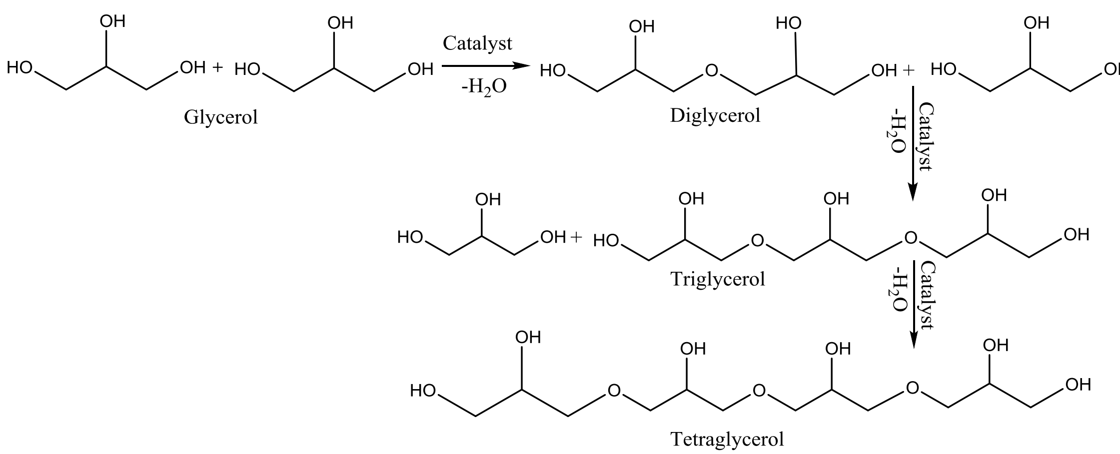


Figure 2. Polyglycerol production

Thermal dehydration of glycerol at an elevated temperature and atmospheric pressure have been applied by various literature for the polyglycerol production with a selected catalyst. However, the reaction has been reported to proceed in the absence of the a catalyst, but the conversion is slow and the yield relatively small.

The main aim of this work is to simultaneously produce biodiesel and convert the glycerol by-product into polyglycerol in a pressurized vessel.

Reactive extraction of microalgae in a foam column

Salihu Musa, PhD, 2016-Present

The aim of this work is to convert wet algae into biodiesel in a foam flotation column. To achieve this aim, three main objectives are to be explored: (1) generation of stable foam in the presence of methanol; (2) ensuring that there is enough methanol at the top of the column to allow for reactive extraction; and (3) the actual biodiesel production from the collected broth.

Reactive extraction requires that both oil extraction and transesterification of lipid to biodiesel occur simultaneously. *Chlorella Vulgaris* has been selected for the first phase of study while cetyltrimethyl ammonium bromide (CTAB), a cationic surfactant, is the algae collector. The generation of stable foams in the presence of methanol was successfully examined based on tesnsiometry and a threshold value of 50% methanol was discovered, beyond which foam stability could not be achieved. Also investigated, was the order and pattern of mixing based on which foam stability was disrupted by the counter flow when methanol was added from the top. Furthermore, the fate of methanol was investigated for the bottom-fed methanol, by a combination of refractometry, material balance, and factorial design of experiment. It was discovered therefrom that the following conditions favours high methanol composition at the top: high airflow combined with low feed flow (Figure 3); high feed flow combined with low airflow (Figure 3); low CTAB concentration over a long runtime (Figure 4); and high CTAB concentration at low feed flow (Figure 5). These conditions are more likely to warrant reactive extraction.

Ultimately, working at higher methanol regime (≤ 50%), combined with the optimised conditions may allow for the next stage, which is the reactive extraction. There is an alternative plan to do the reactive extraction in a separate unit located at the top of the column where methanol would be added as a separate stream.

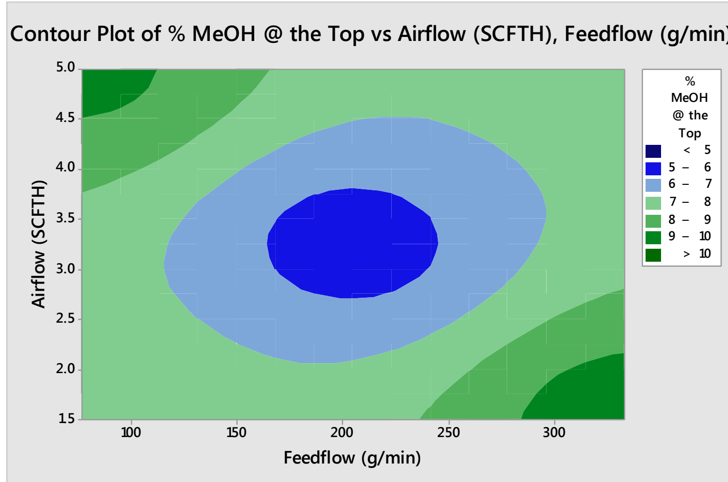


Figure 3: Interactive effect of airflow and feed flow on the composition of methanol in the foamate

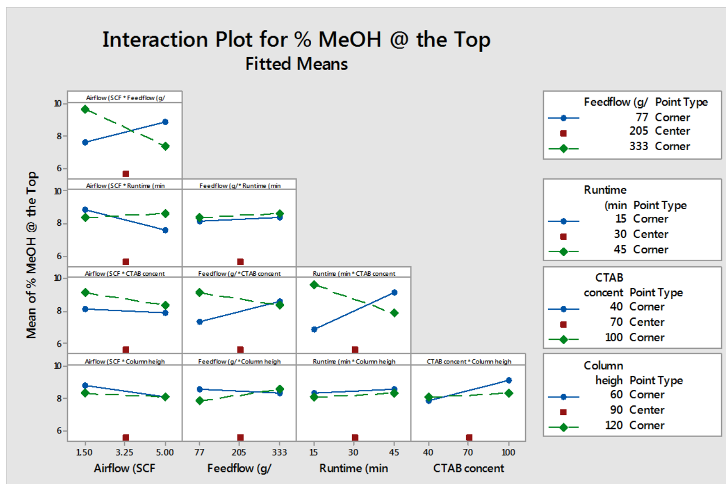


Figure 4. Combined effect of all process factors on the composition of methanol at the column top

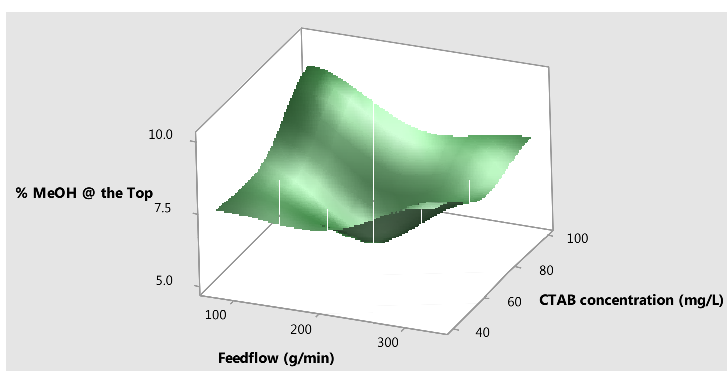


Figure 5. Contour plot of the effects of feed flow and CTAB conc. on methanol composition at the top

Strategies for biodiesel production from waste cooking oils via base-catalysed process

Dr Valentine Eze, Research Associate

Homogenous alkali-catalysed biodiesel production from waste cooking oil (WCO) requires acid-catalysed pre-treatment steps due to high free fatty acid (FFA) contents. The presence of high FFA and water causes saponification in a conventional biodiesel processing, and an FFA pre-treatment step is usually required for such feedstocks.

This study applied numerical and experimental techniques to investigate strategies to obtain high yields of fatty acid methyl esters (FAME) from one-step base-catalysed transesterification of WCO containing high levels of free fatty acids (up to 5.5 wt%) and 3 wt% of water by altering operating conditions. The process conditions were investigated for methanol to WCO molar ratios from 6:1 – 18:1, $NaOCH_3$ catalyst contents in the range of 0.5 wt% - 3 wt%, FFA contents from 1.5 wt% - 5.5 wt%, water contents of 0.12 wt% - 3 wt%, and reaction temperature of 60°C.

About 98% FAME yield in homogeneous $NaOCH_3$-catalysed transesterification was obtained within 5 min reaction time at methanol to WCO molar ratios of 12:1 – 18:1 and 3 wt% catalyst loading for WCO containing 1.53% FFA and 0.12 wt% water (Figure 6a). It was found that high molar ratios of methanol to oil (> 6:1) could prevent saponification. At 5.5 wt% FFA and 3 wt% water contents, ≥96.5% FAME yield was achieved at 18:1 molar ratio of methanol to WCO within 5 min, compared to only 62.8 ± 1.2% for a molar ratio of 6:1, as shown in Figure 6b. Mathematical modelling (MATLAB) was used to predict FAME yields at various conditions and these were validated using experimental data. Sets of conditions identified in this study can be used to rapidly produce biodiesel from low quality triglyceride sources in a single base-catalysed process step.

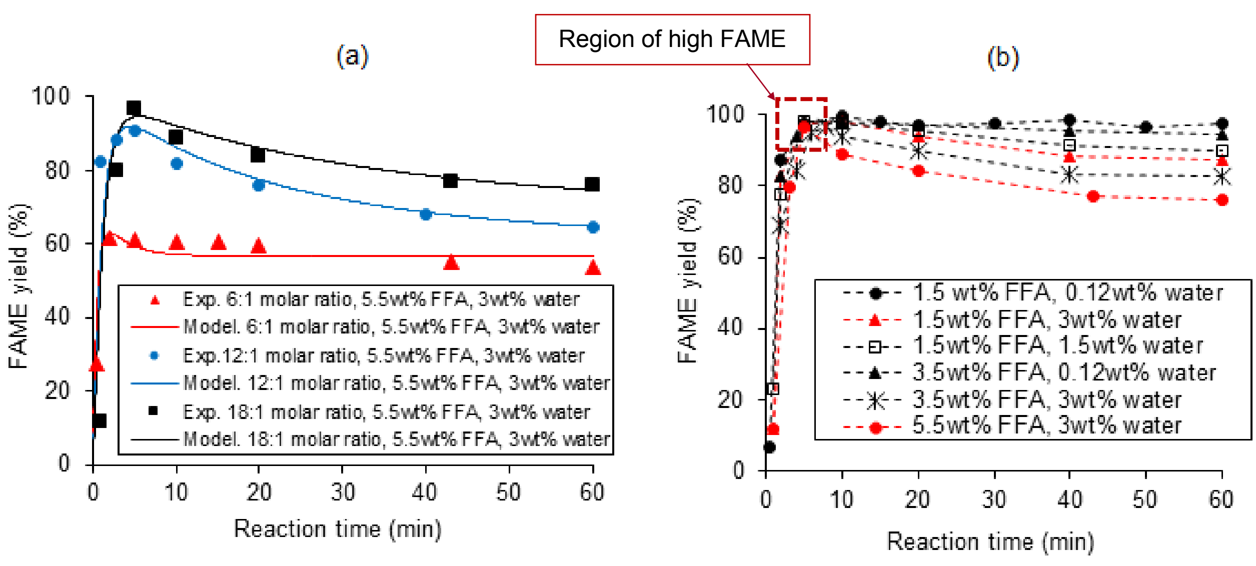


Figure 6. FAME yields at 60°C temperature, and 3 wt% $NaOCH_3$ for transesterification at (a) 18:1 methanol to WCO molar ratio, 0.12 wt% - 3 wt% water content and FFA levels of 1.5 wt% - 5.5 wt%, (b) WCO containing 5.5 wt% FFA and 3 wt% water at 6:1 - 18:1 methanol to WCO molar ratios