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PROCEEDING

INTERNATIONAL CONFERENCE AND WORKSHOP ON CHEMICAL ENGINEERING 2013

in conjuction with The 10th UNPAR National Chemical Engineering Conference

“Chemical Engineering Role for Sustainable Development”

Kuta, Bali
December 4-5th, 2013

INSTITUT
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SURAT TUGAS

No. : 172 /ST/LP3M-ITI/XI/2013

Pertimbangan : bahwa dalam rangka mengikuti Seminar Internasional Conference and Workshop on Chemical Engineering 2013 di Kuta - Bali, perlu dikeluarkan Surat Tugas.

Dasar : 1. Surat penerimaan abstrak dari Panitia Seminar tanggal 5 September 2013
2. Kepentingan ITI

DITUGASKAN

Kepada : Dr. Ir. Joelianingsih, MT

Untuk : 1. Mengikuti Seminar Internasional Conference and Workshop on Chemical Engineering sebagai Pemakalah yang berjudul " Semibatch Bubble Column Reactor Design for Biodiesel Production " dan " Biodiesel Production from Palm Frying Oil Using Sulphated Zircfonia Catalyst In a Bubble Column Reactor " pada tanggal 3 – 6 Desember 2013 bertempat di Kuta - Bali.
2. Melaporkan hasil tugas kepada Direktur LP3M ITI
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INTERNATIONAL CONFERENCE AND WORKSHOP ON CHEMICAL ENGINEERING UNPAR 2013

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CERTIFICATE OF ATTENDANCE

This is to certify that

Dr. Ir. Joelianingsih, M.T.

has presented oral paper during
International Conference and Workshop on Chemical Engineering 2013
with theme

"Chemical Engineering Role for Sustainable Development"

held in Kuta, Bali, Indonesia, December 4-5th, 2013

Chairman ICCE 2013

INTERNATIONAL CONFERENCE AND WORKSHOP ON CHEMICAL ENGINEERING UNPAR 2013

Dr. Henky Muljana, S.T., M.Eng.

PREFACE

Dear all participants,
First of all, we would like to welcome all of you into the 1st UNPAR's International Conference of Chemical Engineering and Process Safety Workshop where this event is also in conjunction with the 10th National Conference of Chemical Engineering. This year, on April 2013, we are celebrating our 20th anniversary. Therefore in this special year, we are extending our conference scope from national to international event. It is also our intention to introduce our Chemical Engineering Department in theInternational level.

We are choosing Chemical Engineering Role for Sustainable Development as the conference theme. We realize that chemical engineering plays an important role to ensure the sustainability in every aspects starting from alternative renewable feedstocks for energy and chemicals, alternative green processes until waste minimization. Strongly related with the sustainable process development, chemical engineer also have to deeply involved in the process safety in order to bring this sustainable technology in the industrial level. It is our wish that the conference and the workshop will provide an excellent forum for academia, industry and Indonesia's government to share information, discuss recent knowledgeand technological advancement, as well as provide an up-to-date perspective on the sustainable development around the globe.

We are very pleased to have lectures given by our special keynote speakers. The topics are surely important and will stimulate a fruitful discussion about the chemical engineering role in many aspects for sustainable development. We would like to sincerely thanks our keynote speakers, Prof Djoko Said Damardjati from Indonesian Agency for Agricultural Research and Development (IAARD), Ministry of Agriculture, Indonesia, Prof Haryadi from Gadjah Mada University, Prof Yudi Samyudia from Curtin University,Prof. Leon Janssen, Prof H.J. Heeres, and Prof F. Picchioni from University of Groningen, Assoc. Prof. Kim Jaehoon from Sungkyunkwan University, and Prof. Xiao Dong Chen from Soochow University. We would like to give a special thanks to Bapak Ir Yos Triadmodjo, M.M., for his willingness to share and to bring his 30 years Industrial experiences in the Process Safety workshop.

We are also grateful to the Indonesian Center for Estate Crops Research and Development Indonesian Agency for Agricultural Research and Development (IAARD), Ministry of Agriculture, Indonesia and Research Centre for Chemistry, Indonesian Institute of Science (LIPI) for supporting our 1st International Conference and Workshop. Last but not least, I would like to express a very special gratitude to the scientific committee and the organizing committee for their time, efforts and contribution to this event.

And finally, I hope that you enjoy this event and wish you a pleasant stay in Kuta, Bali.

Sincerely,

Dr.Henky Muljana, ST.,M.Eng
Chairman of the Organizing Comitteee,
Head of Chemical Engineering Department
Parahyangan Catholic University

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International Conference and Workshop on Chemical Engineering UNPAR 2013

Semibatch Bubble Column Reactor Design for Biodiesel Production

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Abstract

The application of bubble column reactors are widely spread out due to a number of advantages both in design and operations compared to other reactors. The first one is they have excellent heat and mass transfer characteristic. The second is low cost of operation and maintenance, and the last is high durability of the catalyst. Owing to the wide application area of bubble column reactors, the design and scale up of the reactors, the complexity of hydrodynamic and operational conditions have put on the attention of engineers. The performance of bubble column reactor is proven for transesterification process reported by Joelianingsih et. al.; nevertheless, the conversion and the yield of the product are still low. The reaction operated at atmospheric condition in the absence of catalyst will lead to more economic process for industrial application. Thus, the improvement of reactor design is necessary especially in order to increase the yield and conversion of the product. The bubble column reactor is designed with height to diameter ratio of 5 equipped with vaporizer, superheater and condenser. The material for all the equipments is made of stainless steel (316 SS) except for vaporizer is made of 304 SS. The performance of bubble column reactor for methyl esterification is increased both on the quality and the quantity of the biodiesel product. The analysis on quality of biodiesel showed that the free glycerol contents, monoglycerides, diglycerides, and triglycerides has satisfied both EN 14214 standard and SNI 718:2012. The quantity of the product is improved as showed by higher yield obtained. The future work shall be focused on utilizing various spargers to gain optimum results and also attaining continuous methanol separation setup from end product for recycling.

Keywords: bubble column; design ratio; yield; conversion; biodiesel.

1. Introduction

The general types of multiphase reactor comprise of three main categories namely the fluidized bed reactor, the trickle bed reactor (fixed or packed bed), and the bubble column reactor. In principle, a bubble column reactor is a cylindrically vessel equipped with gas sparger at the base of reactors to dispense gas in the form of bubbles into either liquid phase or solid-liquid phase. The bubble column reactors are intensively used as multiphase reactor and contactor in chemical, petrochemical, biochemical and metallurgical industries [1]. These typical reactors are especially utilized in chemical processing such as oxidation, chlorination, polymerization, alkylation, and hydrogenation, in producing synthetic fuel, in biochemical process, and waste water treatment [2,3].

The application of bubble column reactors are widely spread out due to a number of advantages both in design and operations compared to other reactors. The first one is they have excellent heat and mass transfer characteristic. The second is low cost of operation and maintenance, and the last is high durability of the catalyst [1]. Furthermore, continuous catalyst supply and withdrawal ability and plug-free operation are their superiority among other reactors [3]. Owing to the wide application area of bubble column reactors, the design and scale up of the reactors, the complexity of hydrodynamic and operational conditions have put on the attention of engineers.

Joelianingsih, et al. [4] reported the performance of bubble column reactor for uncatalyzed methyl esterification of free fatty acids. The performance of bubble column reactor is proven for esterification process; nevertheless, the conversion and the yield of the product are still low. The reaction operated at atmospheric condition in the absence of catalyst will lead to more economic process for industrial

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application. Thus, the improvement of reactor design is necessary especially in order to increase the yield and quality of the product.

2. The bubble column reactors concept and technology

The main interest of bubble column reactor studies are focused on design and scale-up, hydrodynamics and regime analysis and characteristics parameters. This study mainly focused on design and scale-up of bubble column reactor for biodiesel productions in order to increase conversion rate of methyl esterification/transesterification.

In general, the design and scale-up of bubble column reactors rely on at least three characteristic: (i) heat and mass transfer (ii) mixing (iii) chemical kinetics. Although the construction of bubble column is straightforward, precise and successful design and scale-up require an improved understanding of multiphase fluid dynamics and its influences. Industrial bubble column usually operate with a length to diameter ratio of at least 5 [1].

Shah et al. [2] reported that the effect of column diameter is insignificant on gas holdup when larger than 10-15 cm. On the other hand, Luo et al. [5] reported that the column height effect is negligible when the height is higher than 1-3 m and the the aspect ratio is larger than 5. Vandu and Krishna [6] reported that $k_l a/\square_g$ demonstrated a slight increase with column diameter. The volumetric mass transfer coefficient, $k_l a$ increases with gas velocity, gas density and pressure while it decreases with increasing solid concentration and liquid viscosity. The presence of large bubbles should be avoided in industrial columns for effective mass transfer [7].

Table 1. Mass transfer coefficient correlations for gas-liquid bubble columns

Researcher	Correlation	Reference
Akita and Yoshida	$\frac{k_l a D_T^2}{D_{AB}} = 0.6\left(\frac{\nu_l}{D_{AB}}\right)^{0.5}\left(\frac{g D_T^2 \rho_l}{\sigma}\right)^{0.62}\left(\frac{g D_T^3}{\nu_l^2}\right)^{0.31}\varepsilon_g^{1.1}$	[8]
Shah et al.	$k_l a = 0.467 V_g^{0.82}$	[2]
Kang et al.	$k_l a = K \times 10^{-3.08}\left(\frac{D_T V_g \rho_L}{\mu_l}\right)^{0.254}$ where K is the correlation dimension	[9]

Heat transfer in bubble column reactor is important since many chemical reactions are usually involved with energy supply (endothermic) or energy removal (exothermic) process. Hence, the heat transfer from the reactor wall and inserted coils became interesting discussion in many literatures [10]. Many hydrodynamic studies examined the heat transfer between the heating objectives and the system flow to understand the effect of hydrodynamic on the heat transfer for improving the design and operation of bubble column reactors [11]. It can be stated that the heat transfer coefficient increases with increasing temperature, but decreases as a function of liquid viscosity and particle density [7].

Bubbles size and distribution also important in bubble column operations. Their holdup contribution and rise velocities have significant impact on altering the hydrodynamics, as well as heat and mass transfer coefficient in bubble column reactors. The distribution of bubbles firstly influence by the bubbles formulation by the sparger.

For any given gas sparger size with pre-determined number of openings or holes size, the gas initial force at the sparger orifice is related to the surface tension forces. This relationship is best described by Weber number (We), which is often used to design the gas sparger. The Weber number for gas is given as follow:

$$We = \frac{\rho_G U_{G,O}^2 d_O}{\sigma} = \frac{\rho_G U_G^2 D_C^4}{N_O^2 d_O^3 \sigma} \quad (1)$$

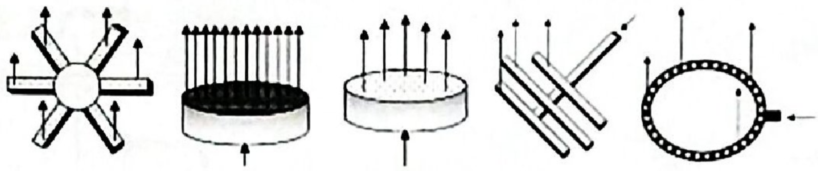

Fig 1. Sparger types utilized in bubble column reactor (a) spider shape (b) porous plate (c) perforated plate (d) multiple orifice nozzle (e) perforated ring [12]

3. Design and scale-up

In this project, the design of bubble column reactor is straight forward with height to diameter ratio is 3 to 5 (more preferably 5). The bubble column reactor setup equipped with a methanol vaporizer, a superheater, and a condenser. All main materials are made of stainless steel 316 SS except for vaporizer made of 304 SS. The flow rate of methanol controlled by Chengfeng Flowmeter LZB-DK800-4 and the temperature system is controlled by automatic PID temperature controller YFYB (Type XMTG) equipped with thermocouple type-K. Overview of the setup are shown in Fig 2 to Fig. 4

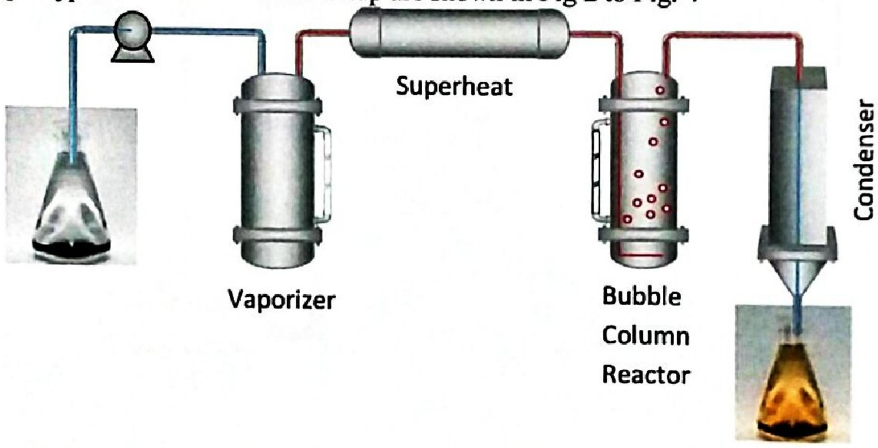

Fig 2. Schematic diagram of the bubble column reactor setup equipped with vaporizer, superheater, and condenser

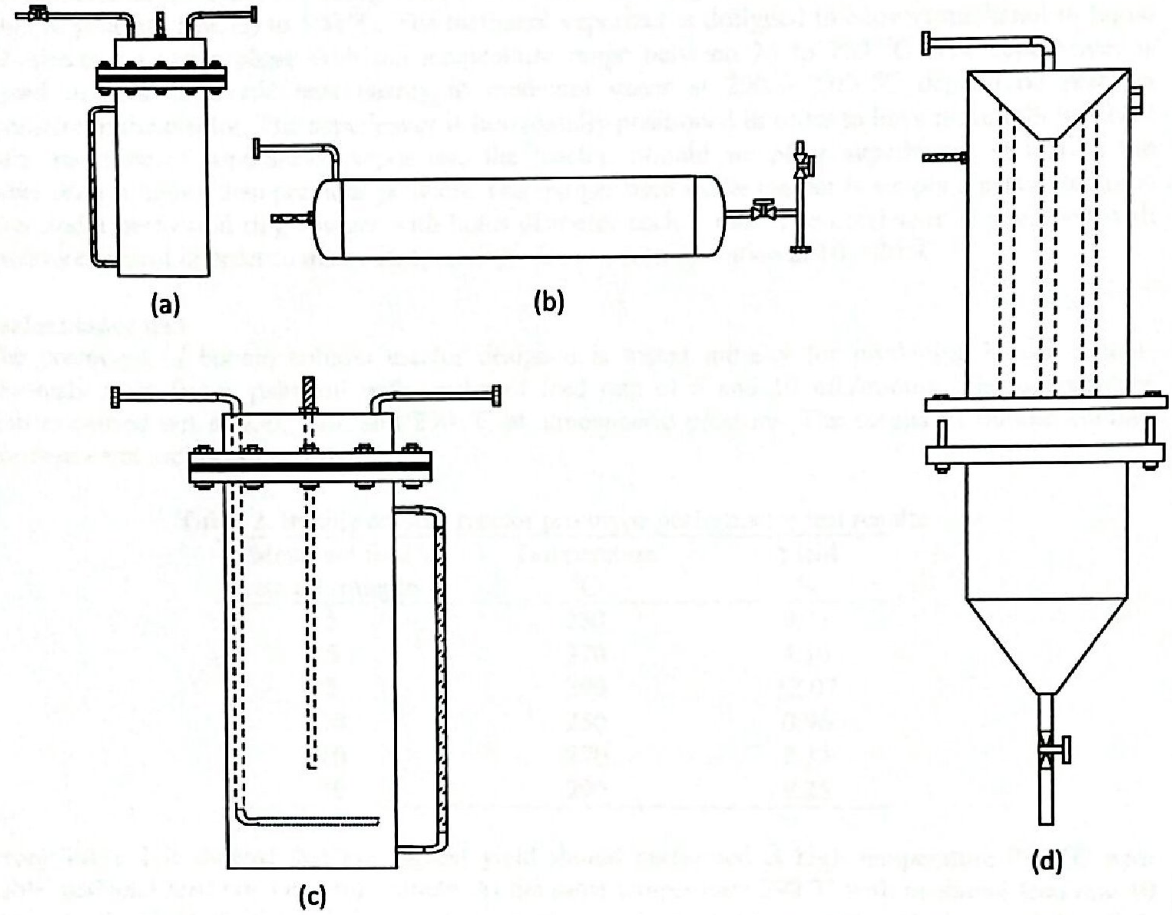

Fig 3. **The bubble column reactor designed (a) vaporizer (b) superheater (c) reactor (d) condenser**

Fig 4. **The bubble column reactor setup and rotary evaporator for final product separation from methanol**

All the heating elements are using band heater built on design with power supply 500 to 750 watt capable to generate heat up to 500 °C. The methanol vaporizer is designed to convert methanol in liquid phase directly to vapor phase with the temperature range between 75 to 250 °C. The superheater is designed to give additional heat energy to methanol vapor at 250 – 500 °C depend on reaction temperature in the reactor. The superheater is horizontally positioned in order to have more efficient heat transfer and flow of superheated vapor into the reactor. Should we place superheater vertically; the pressure drop is higher than previous position. The sparger used in the reactor is simply a perforated tube sparger and a perforated ring sparger with holes diameter each 1 mm. The condenser is equipped with temperature control in order to maintain operating condensation operation at 10 – 20 °C.

4. Performance test

The prototype of bubble column reactor designed is tested initially for producing biodiesel non-catalytically from frying palm oil with methanol feed rate of 5 and 10 mL/minute. The temperature condition carried out at 250, 270, and 290 °C at atmospheric pressure. The results of bubble column performance test are shown in Table 2.

Table 2. Bubble column reactor prototype performance test results

Methanol feed rate mL/minute	Temperature °C	Yield %
5	250	0.71
5	270	4.16
5	290	12.07
10	250	0.96
10	270	2.32
10	290	9.25

From Table 1 it showed that the highest yield should performed at high temperature 290 °C with suitable methanol feed rate i.e. 5 mL/minute. At the same temperature 290 °C with methanol feed rate 10 mL/minute, the yield of product is lower due to short contact time between methanol vapor and the oil. It is found that the number of bubbles formed at low methanol feed rate i.e. 5 mL/minute are much more uniform and even. Therefore, the contact between the reactants particles are more frequent and in results more reactants converted into the product. The formation of bubbles at high feed rate of methanol i.e. 10 mL/minute, resulting an excessive interfacial contact between reactants particles. The bubbles size are larger and more turbulence. For high methanol feed rate, an additional roof perforated plate should be placed to increase gas hold up and accordingly increase contact time between the reactants.

The biodiesel product were analyzed by gas chromatography (GC 2010 Shimadzu) with modified EN 14105 standard methods [13] and presented in Fig 5 to Fig 8.

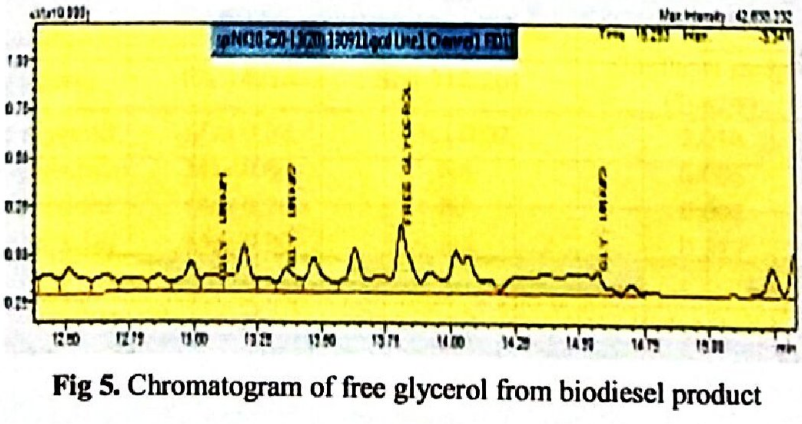

Fig 5. Chromatogram of free glycerol from biodiesel product

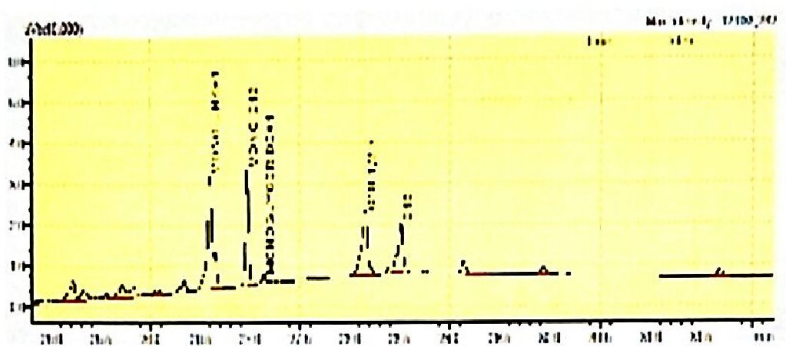

Fig 6. Chromatogram of monoglycerides from biodiesel product

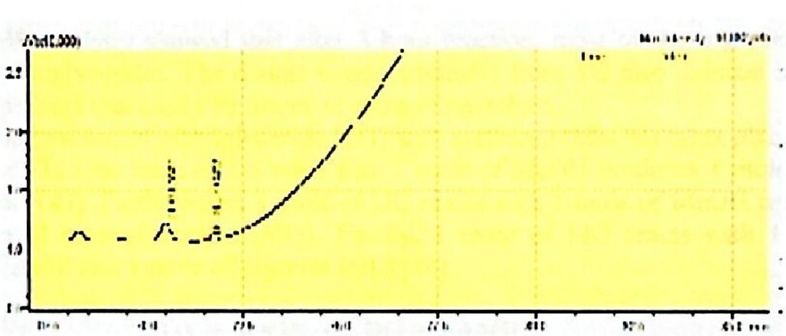

Fig 7. Chromatogram of diglycerides from biodiesel product

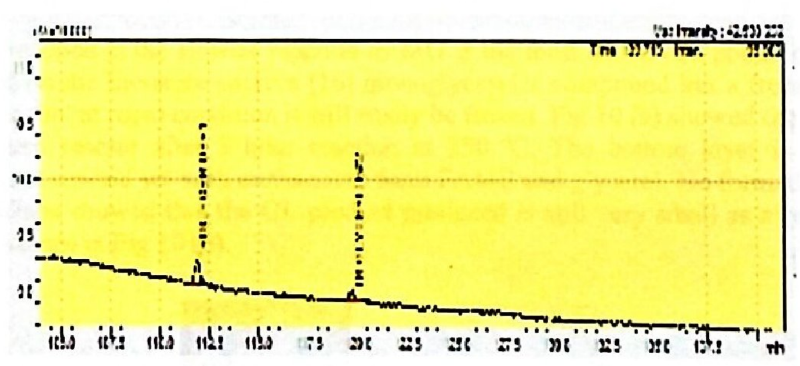

Fig 8. Chromatogram of triglycerides from biodiesel product

Table 3. Biodiesel product analysis

Parameter	EN 14214	SNI 718:2012	Biodiesel sample (%w/w)
Free glycerol	Max 0.02	Max 0.02	0.018
Monoglycerides	Max 0.80	NA	0.005
Diglycerides	Max 0.20	NA	0.001
Triglycerides	Max 0.20	NA	0.017

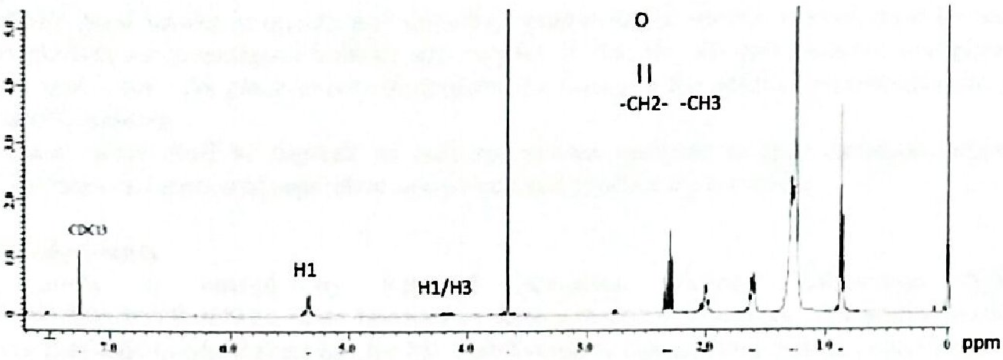

Fig 9. ^{1}H-NMR spectrum of reactant in the bubble column reactor after 3 hr reaction at 250 °C reaction condition

From the ^{1}H-NMR analysis showed that after 3 hour reaction, most of the triglycerides are already breaking out into monoglycerides. The double bonds originally from TG also reduced into straight chain (alkanes) resulting product that easily be frozen at room temperature.

Transesterification reaction of oil/triglyceride (TG) with methanol (MeOH) takes place in 3 stages as in equation (1), (2) and (3). One mole of TG react with 1 mole of MeOH produces 1 mole of FAME and 1 mole of diglycerides (DG). Furthermore, 1 mole of DG reacts with 1 mole of MeOH produces 1 mole of FAME and 1 mole of monoglycerides (MG). Finally, 1 mole of MG reacts with 1 mole of MeOH produces 1 mol of FAME and 1 mole of glycerol (GL) [14].

$$TG + MeOH \leftrightarrow DG + FAME \quad (1)$$
$$DG + MeOH \leftrightarrow MG + FAME \quad (2)$$
$$MG + MeOH \leftrightarrow GL + FAME \quad (3)$$

The third phase reaction is the slowest reaction as MG is the most stable compound compared to DG and TG [15]. Based on the literature sources [16] monoglycerides compound has a freezing point above room temperature so that at room condition it will easily be frozen. Fig 10 (a) showed the liquid condition in the bubble column reactor after 3 hour reaction at 250 °C. The bottom layer is monoglycerides compound that has not reacted yet with methanol to form FAME and glycerol. No formation of two layers in the reaction products showed that the GL product produced is still very small as a result of the slow reaction stage 3 as shown in Fig 10 (b).

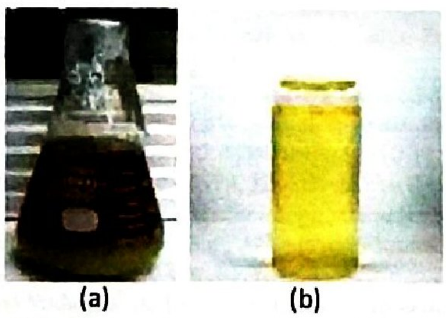

Fig 10. (a) The liquid condition in the bubble column reactor, and (b) biodiesel product after 3 hr reaction at 250 °C reaction condition

5. Conclusion

The bubble column reactor designed performance for methyl esterification is increased both on the quality and the quantity of the biodiesel product compared to previous work done by Joelianingsih et al.. The analysis on quality of biodiesel showed that the free glycerol contents, monoglycerides, diglycerides, and triglycerides has satisfied both EN 14214 standard and SNI 718:2012.

To obtain good results in quality and quantity, sparger in the reactor system must be redesigned in order to produce more methanol bubbles and smaller so that the reaction between two phases (oil and methanol vapor) can take place evenly throughout the liquid in the reactor, accordingly the yield of oil products will increase.

The future work shall be focused on utilizing various spargers to gain optimum results and also attaining continuous methanol separation setup from end product for recycling.

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