ห้องสมุดงานวิจัย สำนักงานคณะกรรมการวิจัยแห่งชาติ



## รายงานการวิจัยฉบับสมบูรณ์

การออกแบบเครื่องปฏิกรณ์แบบต่อเนื่องสองขั้นตอนเพื่อผลิต เมทิลเอสเทอร์จากน้ำมันปาล์มดิบชนิดทีบรวมชนิดกรดสูง คณะวิศวกรรมศาสตร์ มหาวิทยาลัยสงขลานครินทร์ วิทยาเขตหาดใหญ่

หัวหน้าโดธงการวิจัย ธศ.กำพล ประทีปชัยกูร

งานวิจัยนี้ได้รับทุนอุดหนุนการวิจัย จากเงินงบประมาณแผ่นดิน ประจำปังบประมาณ 2554





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**Topic:** Design of Two-Stage Continuous Reactor for Producing Methyl Ester from High Free Fatty Acid Mixed Crude Palm Oil

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#### **ABSTRACT**

Generally, high quality of biodiesel in high productivity is required under the low cost investment. Therefore, the continuous stirred tank reactors (CSTR) are preferred in the investment because it is easy to fabricate and to control the product quality from liquid-liquid reaction under the low cost investment. The principle of kinetics, unit operation of chemical engineering and heat transfer are used to design CSTR for producing biodiesel from mixed crude palm oil (MCPO) via the two-stage process (esterification followed by transesterification). After finishing the design, the designed system was verified by simulating with The ASPEN PLUS Simulation Engine (chemical commercial program). Finally, the system was fabricated and operated. The results indicated that the efficiency of the two-stage continuous process in full system was lower than the design around 10 %. FFA could be reduced from 16-18 wt% to less than 1 wt% under the optimization of 23.04 v% of methanol, 2.07 v% of sulfuric acid, 2.22 min of retention time, and 793 rpm of stirrer speed (40 L/hr of oily solution yield and 15 L/hr of waste solution yield). The concentration of biodiesel at 95.70 wt% having yield around 43 L/hr and of glycerin around 12 L/hr was obtained under the test operated condition of 24 v% of methanol and 1 %wt/v of potassium hydroxide under 4 min of retention time of and 793 rpm of stirrer speed. After purification, it was found that 75.06% of biodiesel based on initial MCPO (84.44% of biodiesel based on acidified MCPO) was obtained.

<u>Keywords</u>: Biodiesel, Continuous reactor, CPO, Methyl ester, Mixed crude palm oil, The two-stage process

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#### **NOMENCLATURES**

A The initial regent

 $A_d$  The surface area of decanter (m<sup>2</sup>)

A<sub>i</sub> Area of inner surface of reactor (m)

A<sub>o</sub> Area of outer surface of reactor (m)

 $A_{\rm m}$  Logarithmic mean area (m<sup>2</sup>)

 $A_s$  Surface area (m<sup>2</sup>)

ASTM American standard test method

[A] The molar concentration of reagent A

[AL] The concentration of alcohol (mol/L)

The coefficient of reagent A, The reaction order of reagent A

a and FFA

a<sub>1</sub> The experiment determined constant

B The initial reagent

[B] The molar concentration of reagent B

The coefficient of reagent B, The reaction order of reagent B

and alcohol

C The product

 $C_A, C_{A0}$  The concentration of reagent A (mol/L)

C<sub>AL</sub> The concentration of alcohol (mol/L)

 $C_{B, C_{B0}}$  The concentration of reagent B (mol/L)

C<sub>C</sub>, C<sub>C0</sub> The concentration of product C (mol/L)

 $C_D, C_{D0}$  The concentration of product D (mol/L)

C<sub>DG</sub> The concentration of diglyceride (mol/L)

 $C_E$  The concentration of ester (mol/L)

C<sub>FFA</sub> The concentration of free fatty acid (mol/L)

C<sub>GL</sub> The concentration of glycerol (mol/L)

C<sub>MG</sub> The concentration of monoglyceride (mol/L)

C<sub>p</sub> Specific heat at constant pressure, KJ/kg·°C or KJ/kg·K

C<sub>TG</sub> The concentration of triglyceride (mol/L)

 $C_{WT}$  The concentration of water (mol/L)

CSTR, CSTRs Continuous stirred tank reactors

[C] The molar concentration of product C

#### NOMENCLATURES (Cont')

The coefficient of product C, The reaction order of product C

c and ester

D The product

Da Diameter of turbine impeller (cm)

D<sub>am</sub> The diameter of impeller (m)

D<sub>d</sub> Diameter of turbine impeller disc (cm)

D<sub>h</sub> Diameter of hole on separate plate (cm)

D<sub>i</sub> The inside diameter of reactor (m)

D<sub>o</sub> The outside diameter of reactor (m)

D<sub>r</sub> Diameter of reactor or tube (m)

D<sub>s</sub> Diameter of separate tank plate (cm)

D<sub>t</sub>, D<sub>tm</sub> The diameter of reactor or tank (cm, m)

DG Diglyceride

[D] The molar concentration of product D

[DG] The concentration of diglyceride (mol/L)

The coefficient of product D, The reaction order of product D

and water

E<sub>s</sub> Space between turbine and bottom tank (cm)

EN European test method

[E] The concentration of ester (mol/L)

e The thickness of turbine (cm)

 $F_A$ ,  $F_B$ ,  $F_C$ ,  $F_D$  The molar flow rate

 $F_{A0}$ ,  $F_{B0}$ ,  $F_{C0}$ ,  $F_{D0}$  The initial molar flow rate

F<sub>A1</sub> The molar flow rate (mol/min)

FFA Free fatty acid

[FFA] The concentration of free fatty acid (mol/L)

f The thickness of axle (cm)

G Space between baffle and wall (cm)

GL Glycerol

[GL] The concentration of glycerol (mol/L)

g The thickness of axle ring (cm)

H<sub>n</sub> Reactor height (m)

#### **NOMENCLATURES (Cont')**

H<sub>t</sub>, H<sub>tm</sub>

The height of reactor or tank (cm, m)

HOR', HOR"

Alcohol

H<sub>2</sub>O

Water

H<sub>2</sub>SO<sub>4</sub>

Sulfuric acid

1.

The average heat transfer coefficient for vertical tier of N

hhorizontal, n tube

horizontal tube (W/m·°C)

The arrange head to

hhorizontal, 1 tube

The average heat transfer coefficient for vertical tier of 1

horizontal tube (W/m.°C)

 $h_i, h_2$ 

The convection heat transfer coefficients inside (W/m<sup>2</sup>.°C)

 $h_0, h_1$ 

The convection heat transfer coefficients outside (W/m<sup>2</sup>.°C)

J

Baffle width (cm)

**KOH** 

Potassium hydroxide

k

Thermal conductivity, W/m·°C

 $k_a, k_1$ 

The rate coefficient of the forward reaction

 $k_{-a}, k_2$ 

The rate coefficient of the reverse reaction

Thermal conductivity of reactor material or tube wall

 $k_{m}$ 

 $(W/m^2.°C)$ 

 $k_{v}$ 

The amount of independent variables

L

Blade length (cm)

 $L_{d}$ 

The length of decanter (m)

Lj

The length of jacket (m)

 $L_{t}$ 

The thickness of reactor wall (m)

**MCPO** 

Mixed crude palm oil

ME

Methyl ester or biodiesel

MeOH

Methanol

MG

Monoglyceride

[MG]

The concentration of monoglyceride (mol/L)

m

Mass flow rate (kg/s)

N, Ns

The speed of stirrer (rpm, rps)

N

The number of horizontal tube vertical tier (tube)

 $N_{min}$ 

The minimum number of distillatory plate

#### xxii

#### NOMENCLATURES (Cont')

N<sub>RE</sub> Reynolds number

NaOH Sodium hydroxide

Nu Nusselt number

n The number of fin

P'<sub>A</sub> Vapor pressure of components A (N/m<sup>2</sup>)

 $P'_{B}$  Vapor pressure of components B  $(N/m^2)$ 

PBR Packed bed reactor

PFR Plug-flow tubular reactor

Pr Prandtl number

Q The mixing flow rate (m<sup>3</sup>/hr)

Q Rate of heat transfer of inner solution (KJ/s)

R, R', R", R" Alkyl group

R'COOH Organic acid

RCOOR', R'COOR" Ester

Re Reynolds number

r<sub>A</sub> The reaction rate of regent A

 $r_{AL}$  The reaction rate of alcohol (mol·L<sup>-1</sup>·min<sup>-1</sup>)

 $r_{A1}$  The reaction rate of the first reactor

 $r_{A2}$  The reaction rate of the second reactor

r<sub>DG</sub> The reaction rate of diglyceride (mol·L<sup>-1</sup>·min<sup>-1</sup>)

 $r_E$  The reaction rate of ester (mol·L<sup>-1</sup>·min<sup>-1</sup>)

r<sub>FFA</sub> The reaction rate of free fatty acid (mol·L<sup>-1</sup>·min<sup>-1</sup>)

 $r_{GL}$  The reaction rate of glycerol (mol·L<sup>-1</sup>·min<sup>-1</sup>)

r<sub>i</sub> Inner radius (m)

 $r_{MG}$  The reaction rate of monoglyceride (mol·L<sup>-1</sup>·min<sup>-1</sup>)

r<sub>o</sub> Outer radius (m)

 $r_{TG}$  The reaction rate of triglyceride (mol·L<sup>-1</sup>·min<sup>-1</sup>)

 $r_{WT}$  The reaction rate of water (mol·L<sup>-1</sup>·min<sup>-1</sup>)

S The optimum fin spacing (m)

SFT Saponification followed by transesterification

T<sub>1</sub> Temperature at outside reactor wall (°C)

#### xxiii

#### NOMENCLATURES (Cont')

 $T_{1\infty}$  Temperature of paraffin oil (°C)

Temperature of inside reactor wall (°C)

 $T_{2\infty}$  Temperature of solution in reactor (°C)

TG Triglyceride

TLC/FID Thin Layer Chromatography/ Flame Ionization Detector

TSP The two-stage process

[TG] The concentration of triglyceride (mol/L)

t The fin thickness (m)

t<sub>s</sub> The separation time (hr)

 $t_T$  The mixing time (s)

U The force convection relations (W/m<sup>2</sup>· °C)

The outer surface reactor overall heat transfer coefficient

 $U_o$  (W/m<sup>2</sup>· °C)

V The volume of reactor (L)

 $V_A$  The volume of axle (cm<sup>3</sup>)

 $V_{AR}$  The volume of axle ring (cm<sup>3</sup>)

V<sub>B</sub> The volume of four cylindrical baffles (cm<sup>3</sup>)

VBL The volume of six-blade (cm<sup>3</sup>)

VD The volume of disc (cm<sup>3</sup>)

V<sub>R</sub> The volume of reactor (cm<sup>3</sup>)

 $V_1$  The volume of the first reactor (L)

 $V_2$  The volume of the second reactor (L)

W Blade width (cm)

Ws The width surface of reactor (m)

WT Water

[WT] The concentration of water

X Conversion

X<sub>1</sub> The conversion of the first reactor

X<sub>2</sub> The conversion of the second reactor

x<sub>B</sub> Mole fraction in bottom product

 $x_D$  Mole fraction in overhead product

 $x_i, x_j$  Independent variables

#### xxiv

#### NOMENCLATURES (Cont')

x<sub>w</sub> Thickness of tube (m)

y Response

The heavy phase depth (a half of the liquid depth) and the  $Z_{A1}$ 

height of feed mixture entrance

Z<sub>A2</sub> The height of the heavy liquid overflow

 $$Z_{\text{T}}$$  The liquid depth (90 % of the full separated tank) and the

height of the light liquid overflow

 $\beta_0, \beta_i, \beta_{ij}, \beta_{ii}$  Coefficient

 $\Delta T$  Temperature difference of solution (°C)

 $\Delta T_{lm}$  The logarithm mean temperature difference solution

 $\Delta T_p$  Temperature difference of paraffin oil (°C)

α The center point

α<sub>AB</sub> Relative volatility of component A to component B

 $\rho$  The density of liquid (kg/m<sup>3</sup>)

PA The density of liquid of solution A (kg/m³)

 $\rho_B$  The density of liquid of solution B (kg/m<sup>3</sup>)

τ The retention time (min)

μ The viscosity of liquid (Pa.s)

 $\mu_t$  The total viscosity (cP)

v The volumetric flow (L/min)

 $v_k$  Kinematics viscosity,  $m^2/s$ 

 $v_m$  The mean velocity of liquid (m/s)

 $v_0$  The volumetric flow (L/min)

(1) Triglyceride

(2) Alcohol

(3) Ester

(4) Glycerol