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APPENDICES

APPENDIX A THE STATISTICAL ANALYSIS OF THE REGRESSION MODEL FOR SCALE-UP REACTOR

Table A1. The statistical values of the regression model for scale-up reactor from Design Expert ® 6.0 software

Statistical term	value
Overall mean	64.36
Overall standard deviation	4.75
C.V.	7.38
R^2	0.9732
Adjusted R^2	0.9655
PRESS	938.67
Predicted R^2	0.9468
Adequate Precision	40.11

Overall mean (Mean) and standard deviation (SD) are calculated from the ME content of all experimental conditions. The coefficient of variation (C.V.) is a measure of unexplained or residual variation of the data relative to the size of mean. The variation expressed as a percentage of the overall mean and standard deviation, as shown in Equation A1.

$$\text{C.V.} = \frac{\text{SD}}{\text{Mean}} \times 100 \qquad \text{(A1)}$$

Coefficient of determination (R^2) is a measure of the amount of variation around the mean explained by the model, while adjusted R^2 is the ordinary R^2 value which is adjusted by the number of terms in the regression model. The adjusted R^2 was slightly lower than the R^2 that indicated the excluding of interaction terms has no significant impact on prediction of the model.

Predicted Residual Sum of Squares (PRESS) is a measure of how well the model predicts the responses in a new experiment and employ to calculate the predicted R^2. Small values of PRESS are desirable. The predicted R^2 of 0.9468 is in reasonable agreement, which differ less than 0.2, with the Adjusted R-Squared of 0.9655.

Adequate precision measures the signal to noise ratio and the value greater than 4 is desirable. The regression model has adequate precision of 40.11 which indicates that it will give reasonable performance in prediction within the design space.

The estimated coefficients and its standard error for the regression model for scale-up reactor are shown in Table A2.

Table A2. The estimated coefficients and its standard error in the regression model for scale-up reactor at ±95% confident interval from Design Expert ® 6.0 software

Factor	Estimated coefficient	Degree of freedom	Standard Error of estimated coefficient	Estimated coefficient at 95% confident interval	
				Low	High
Constant	75.90	1	1.86	72.03	79.78
A-T	21.02	1	1.21	18.50	23.54
B-P	9.60	1	1.00	7.52	11.68
C-MeOH:Oil	13.78	1	0.97	11.76	15.79
A^2	-16.89	1	1.01	-18.98	-14.80
B^2	-3.72	1	0.75	-5.27	-2.16
C^2	-4.57	1	0.78	-6.19	-2.95

From Table A2, it was clear that the standard errors are approximately less than 10% of their estimated coefficients. Therefore, this regression model was adequate to predict the methyl ester content for scale-up reactor.

Observed and predicted values, residual, standardized residual and Cook's distance are illustrated in Table A3. The residual, which represents the random or unexplained error in experiments, is a different between observed and predicted values. The standardized residual is the residual divided by the estimated standard deviation of the residual.

Table A3. The residual analysis of actual and predicted value for the regression model for scale-up reactor

Run Order	Actual Value	Predicted Value	Residual	Standardized Residual	Cook's Distance
1	76.34	74.10	2.24	0.51	0.01
2	54.72	53.95	0.76	0.18	0.00
3	83.23	80.05	3.19	0.78	0.03
4	78.25	75.42	2.83	0.65	0.01
5	38.96	47.46	-8.50	-2.37	0.60
6	72.97	75.90	-2.93	-0.67	0.01
7	32.54	35.10	-2.56	-0.62	0.02
8	40.49	44.89	-4.40	-1.07	0.05
9	3.16	0.92	2.24	0.56	0.02
10	91.46	96.17	-4.71	-1.05	0.02
11	80.63	75.60	5.02	1.15	0.03
12	21.60	20.21	1.39	0.34	0.01
13	35.86	36.39	-0.52	-0.16	0.00
14	60.41	62.56	-2.15	-0.53	0.01
15	65.77	65.97	-0.20	-0.05	0.00
16	2.10	4.85	-2.75	-0.80	0.08
17	82.37	82.09	0.27	0.08	0.00
18	78.25	74.28	3.97	0.91	0.02
19	75.50	64.10	11.40	2.85	0.47
20	80.91	83.35	-2.44	-0.55	0.01
21	85.10	88.96	-3.86	-0.88	0.02
22	87.40	92.28	-4.88	-1.11	0.03
23	88.90	90.51	-1.61	-0.41	0.01
24	76.70	70.68	6.02	1.41	0.07
25	76.00	71.47	4.53	1.06	0.04
26	74.70	77.97	-3.27	-0.76	0.02
27	78.00	80.31	-2.31	-0.53	0.01
28	79.70	76.46	3.24	1.02	0.19

From Table A3, it was clear that the residual and standardized residual were represented as the random error with a normal distribution. The normality and randomness of the residuals can be checked as show in Figure A1 - A3. Furthermore, all the cook's distance are less than unity shows that there are no recording errors and the experimented points is not far from the remaining cases.

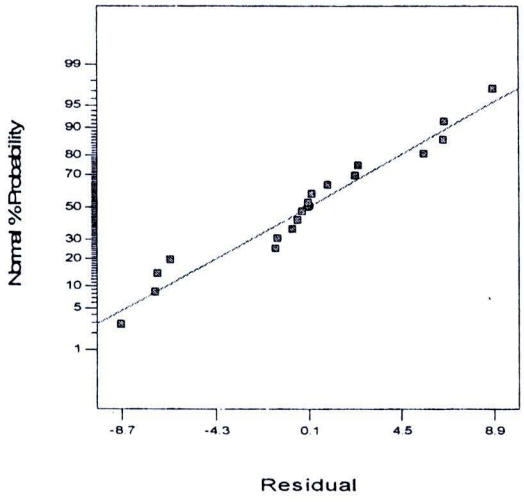

Figure A1. The normal plot of the residuals.

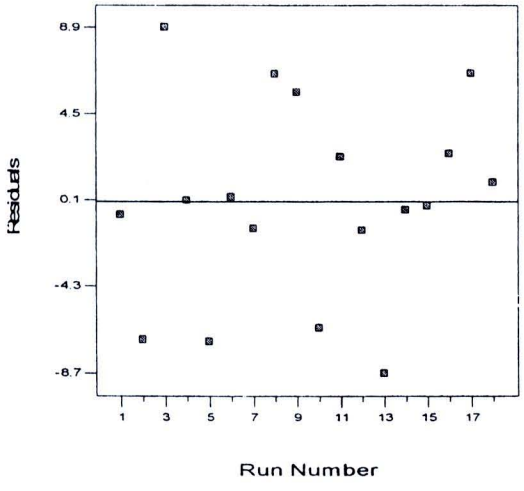

Figure A2. The relationship between residuals and run number.

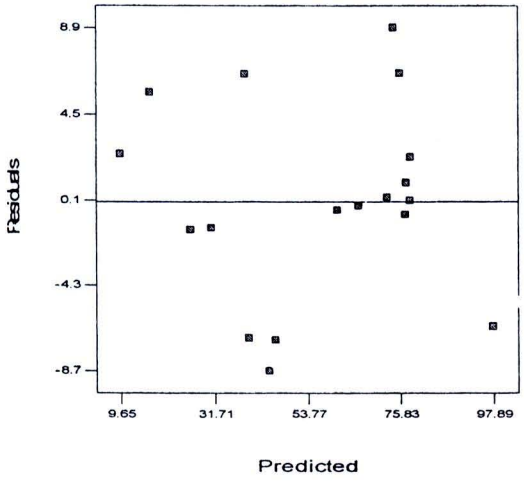

Figure A3. The relationship between residuals and predicted values.

APPENDIX B THE EXAMPLES OF PROGRAMING CODE FOR MATLAB ® SOFTWARE WITH SIMULIS TOOLBOX

Function B1. Code for creates Simulis calculator in Matlab ® software

```
function [] = CreateSimulisCal
% Create a Simulis Calculator
BDF = stCALCreate;
fprintf('\n');
% Edit the parameters of the Simulis Calculator
modified = stCALEdit(BDF);
if modified
    fprintf('The Simulis Calculator Object has been modified.\n');
else
    fprintf('The Simulis Calculator Object has NOT been modified.\n');
end
fprintf('\n');
% Edition of the INPUT unit system of the Simulis Calculator Object
modifiedInput = stCALSystemEdit(BDF,1);
if modifiedInput
    fprintf('The INPUT unit system of the Simulis Calculator Object has been
modified.\n');
else
    fprintf('The INPUT unit system of the Simulis Calculator Object has NOT
been modified.\n');
end
fprintf('\n');
% Edition of the OUTPUT unit system of the Simulis Calculator Object
modifiedOutput = stCALSystemEdit(BDF,2);
if modifiedOutput
    fprintf('The OUTPUT unit system of the Simulis Calculator Object has
been modified.\n');
else
    fprintf('The OUTPUT unit system of the Simulis Calculator Object has NOT
been modified.\n');
end
fprintf('\n');
% Save the Simulis Calculator Object in a text file
fprintf('The Simulis Calculator Object is saved in "BDFCal.txt".\n');
fprintf('Have A Nice Day!!! \n');
txt = stCALSaveToText(BDF);
fid = fopen('BDFCal.txt','w+');
fprintf(fid,'%s',txt);
fclose(fid);
fprintf('\n');
% Free the Simulis Calculator
stCALFree(BDF);
end
```

Function B1. Code for loads, edit and save Simulis calculator in Matlab ® software

```
function [] = LoadSimulisCal
% Create a blank Simulis Calculator
BDFCalEd = stCALCreate;
fprintf('\n');
% Load the Simulis Calculator from a text file
fid = fopen('BDFCalEd.txt','r');
txt = '';
continueRead = 0;
while (continueRead==0)
    line = fgets(fid);
```

```
    if (line == -1)
        continueRead = 1;
    else
        txt=[txt line];
    end
end
fclose(fid);
stCALLoadFromText(BDFCalEd,txt);
fprintf('The Simulis Compounds Object has been loaded from the file called
"compounds.txt".\n');
fprintf('\n');
% Edition of the parameters of the Simulis Calculator Object
modified = stCALEdit(BDFCalEd);
if modified
    fprintf('The Simulis Calculator Object has been modified.\n');
else
    fprintf('The Simulis Calculator Object has NOT been modified.\n');
end
fprintf('\n');
% Edition of the INPUT unit system of the Simulis Calculator Object
modifiedInput = stCALSystemEdit(BDFCalEd,1);
if modifiedInput
    fprintf('The INPUT unit system of the Simulis Calculator Object has been
modified.\n');
else
    fprintf('The INPUT unit system of the Simulis Calculator Object has NOT
been modified.\n');
end
fprintf('\n');
% Edition of the OUTPUT unit system of the Simulis Calculator Object
modifiedOutput = stCALSystemEdit(BDFCalEd,2);
if modifiedOutput
    fprintf('The OUTPUT unit system of the Simulis Calculator Object has
been modified.\n');
else
    fprintf('The OUTPUT unit system of the Simulis Calculator Object has NOT
been modified.\n');
end
fprintf('\n');
% Save the Simulis Calculator Object in a text file
fprintf('The Simulis Calculator Object is saved in "BDFCalEd.txt".\n');
txt = stCALSaveToText(BDFCalEd);
fid = fopen('BDFCalEd.txt','w+');
fprintf(fid,'%s',txt);
fclose(fid);
fprintf('\n');
% Free the Simulis Calculator
stCALFree(BDFCalEd);
end
```

Function B3. Code for show the compound names Simulis calculator in Matlab ® software

```
function ShowCompName
%Create the blank Simulis Calculator
BDFCalEd = stCALCreate;
fprintf('\n');
% Load the Simulis Calculator from a text file
fid = fopen('BDFCalEd.txt','r');
txt = '';
continueRead = 0;
while (continueRead==0)
    line = fgets(fid);
    if (line == -1)
        continueRead = 1;
```

```
    else
        txt=[txt line];
    end
end
fclose(fid);
stCALLoadFromText(BDFCalEd,txt);
%Show Component Name
cmpdCount = stCALCompoundCount(BDFCalEd);
if (cmpdCount==0)
    fprintf('Error: Where are your compounds? \n');
else
    fprintf('- Number of compounds = %d\n',cmpdCount);
    fprintf('\n');
    displayName = '';
    for i=1:cmpdCount
        displayName = strvcat(displayName,
stCALCompoundDisplayName(BDFCalEd,i));
        fprintf('%d - %s\n',i,strtrim(displayName(i,:)));
    end
end
```

Function B4. Code for calculates the initial compressibility factor and molar volume of mixture by Simulis calculator in Matlab ® software

```
function [zm0 vm0] = z0v0Cal(T,P,x)
%Create the blank Simulis Calculator
BDFCalEd = stCALCreate;
% Load the Simulis Calculator from a text file
fid = fopen('BDFCalEd.txt','r');
txt = '';
continueRead = 0;
while (continueRead==0);
    line = fgets(fid);
    if (line == -1);
        continueRead = 1;
    else
        txt=[txt line];
    end
end
fclose(fid);
stCALLoadFromText(BDFCalEd,txt);
% Calculate z0 and v0 from Simulis Calculator
[zmL zmV] = stCALZm(BDFCalEd,T,P,x,0,0,0);
[vmL vmV] = stCALVm(BDFCalEd,T,P,x,0,0,0);
end
```

Function B5. Code for calculates the compressibility factor of mixture in Matlab ® software

```
function [zmL zmV CalT VapRat] = zCal(T,P,X,F0)
%Create the blank Simulis Calculator
BDFCalEd = stCALCreate;
% Load the Simulis Calculator from a text file
fid = fopen('BDFCalEd.txt','r');
txt = '';
continueRead = 0;
while (continueRead==0);
    line = fgets(fid);
    if (line == -1);
        continueRead = 1;
    else
        txt=[txt line];
```

```
    end
end
fclose(fid);
stCALLoadFromText(BDFCalEd,txt);
% Generate mole fraction vector
FA0 = F0(1);
FB0 = F0(2);
FA = FA0*(1 - X);
FB = FA0*((FB0/FA0) - 3*X);
FC = FA0*((0/FA0) + 3*X);
FD = FA0*((0/FA0) + X);
F = [FA FB FC FD];
x = F/sum(F0);
% Calculate z from Simulis Calculator
[zmL zmV CalT VapRat] = stCALZm(BDFCalEd,T,P,x,0,0,0);
end
```

Function B6. Code for writes Equation 6.6 in Matlab ® software

```
% dX/dL = -k(T)*X(L)*z(T,P,L)*P(L)
function dXdL = KEPode(X,kA,FA0,FB0,z0,T,P,F0)
dXdL = kA*(1-X)^0.9565.*((FB0/FA0)-3*X)^1.0493*(z0/(zCal(T,P,X,F0)))^2.0058;
```

Function B7. Code for solves Equation 6.6 in Matlab ® software

```
function [MECont] = ModelAutoRun(T,P,WA0,WB0)
% Reactor design
    OD = 1/8; % [=] in
    thk = 0.029; % [=] in
    D = (OD - 2*thk)*2.54; % [=] cm
    A = D*D*pi()/4; % [=] cm2
    RL = 8000; % Reactor lenght [=] cm
% Operating condition
    T = T + 273; %[=] K
    R = 8.314; %[=] j/mol.K
    % A = Oil, B = MeOH % W0 = mass flow rate
    WA0 = WA0/60; % [=] g/s
    WB0 = WB0/60; % [=] g/s
    % F0 = Mol flow rate
    FA0 = WA0/850; % [=] mol/s
    FB0 = WB0/32; % [=] mol/s
    F0 = [FA0 FB0 0 0]; % [=] mol/s
    % v0 = total vol flow rate at REACTING CONDITION
    % V = molar volume of mixture and v0 = V*F0
    x0 = F0./sum(F0);
    [z0 Vm] = z0v0Cal(T,P,x0);
    v0 = Vm*sum(F0); % [=] cm3/s
% Song (2008) The Journal of Supercritical Fluids, 44(3),pp. 356-363
    k = 4.3376e8*exp(-1.0527e5/(R*T)); %[=] mL/mol.s
% Solve ODE
    kA = (k*A*FA0^1.0058)/(v0^2.0058); % [=] 1/cm
    Lspan = [0 RL];
    IC = 0; % X(t=0) = 0
    [L X] = ode45(@(L,X) KEPode(X,kA,FA0,FB0,z0,T,P,F0),Lspan,IC);
% Calculate Methyl ester content
    theta = F0./F0(1);
    F = [FA0*(1-X) FA0*(theta(2)-3*X) FA0*(theta(3)+3*X) FA0*(theta(4)+X)];
    x = F/sum(F);
% Set xMeOH = xGlyOH = 0 (after MeOH and glycerol were separated)
    x(2) = 0;
    x(4) = 0;
    MECont = 100*x(3)/sum(x);
```

BIOGRAPHY

The author who is responsible for this dissertation is Mr. Ruengwit Sawangkeaw. He was born on 24th January, 1981 at Lopburi Province, Thailand. He graduated with Bachelor of Applied Science in Industrial Chemistry (1998 – 2002) from the Faculty of Applied Science, King Mongkut's Institute of Technology North Bangkok, Thailand. He completed Master of Science in Chemical Technology (2002 – 2005) from the Department of Chemical Technology, Faculty of Science, Chulalongkorn University, Thailand.

Ruengwit started his doctorate program in second semester of the 2005 academic year in Department of Chemical Technology, Faculty of Science, Chulalongkorn University. He received financial support from Clean and Green Fuel Research Unit, and Graduate School, Chulalongkorn University in the first and second year. He was a co-author in the article names "Continuous production of biodiesel via transesterification from vegetable oils in supercritical methanol" which was accepted to publish in "Energy and Fuels". His first paper entitles "Effect of co-solvents on production of biodiesel via transesterification in supercritical methanol" accepted for publication in "Green chemistry".

Ruengwit was also received complementary financial grant from Chulalongkorn University Dutsadi Phiphat Scholarship do research at Institut National Polytechnique de Toulouse, Laboratoire de Génie Chimique in France for eight months. His second article on "A review of laboratory-scale research on lipid conversion to biodiesel with supercritical methanol (2001-2009)" accepted for publication in "The Journal of Supercritical Fluids". He also presented his work at 1 conference in Malaysia and 3 conferences in Thailand.