

Process Optimization for Biodiesel Production from Animal Fat via Two-step Catalyzed Process

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Introduction

Introduction



The leather industry is an industry which generates a large amount of solid and liquid wastes. Most of the solid wastes originate from the pre-tanning processes while half of it comes from the fleshing step. Raw fleshing wastes which mainly consist of protein and fat have almost no recovery option and the disposal is costly. Animal fat waste can produce biodiesel for renewable energy.

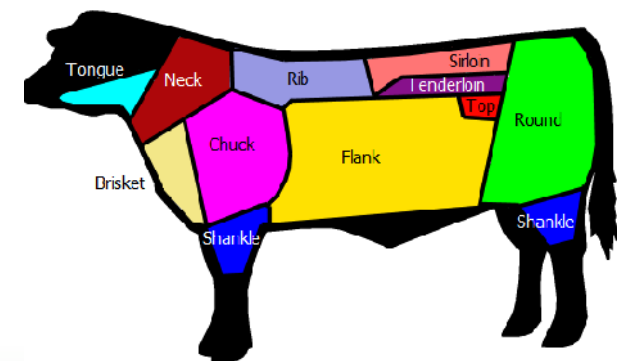


Introduction



❖ Response Surface Methodology (RSM)

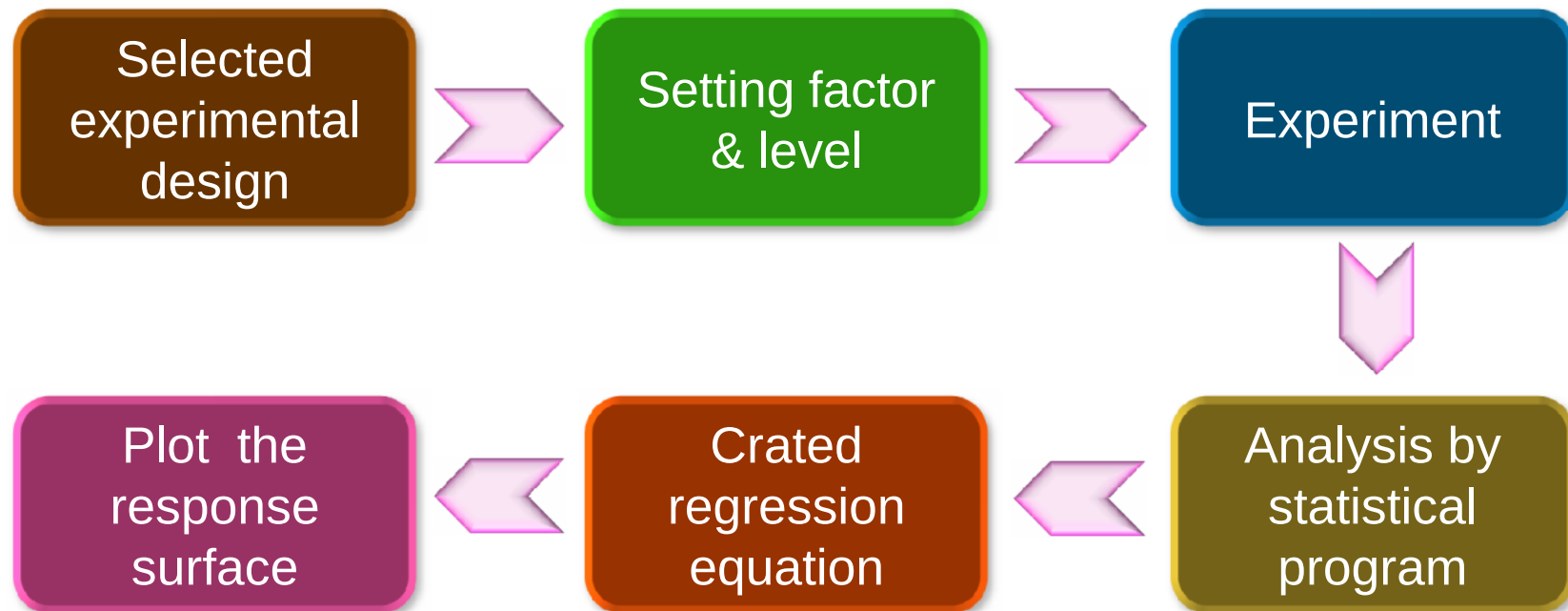
- Statistical and mathematical techniques
 - Developing & improving processes
 - Optimizing processes



Introduction



❖ Optimization process by RSM



Introduction



❖ Regression equation

$$Y = b_0 + \sum_{i=1}^4 b_i x_i + \sum_{i=j}^3 \sum_{j=i+1}^4 b_{ij} x_{ij}$$

$$Y = b_0 + \sum_{i=1}^4 b_i x_i + \sum_{i=1}^4 b_{ii} x_i^2 + \sum_{i=j}^3 \sum_{j=i+1}^4 b_{ij} x_{ij}$$

$$Y = b_0 + \sum_{i=1}^5 b_i x_i + \sum_{i=1}^5 b_{ii} x_i^2 + \sum_{i=1}^5 b_{iii} x_i^3 + \sum_{i < j}^4 \sum_{j=1}^5 b_{ij} x_i x_j + \sum_{i < j}^3 \sum_{j < k}^4 \sum_{k=1}^5 b_{ijk} x_i x_j x_k$$



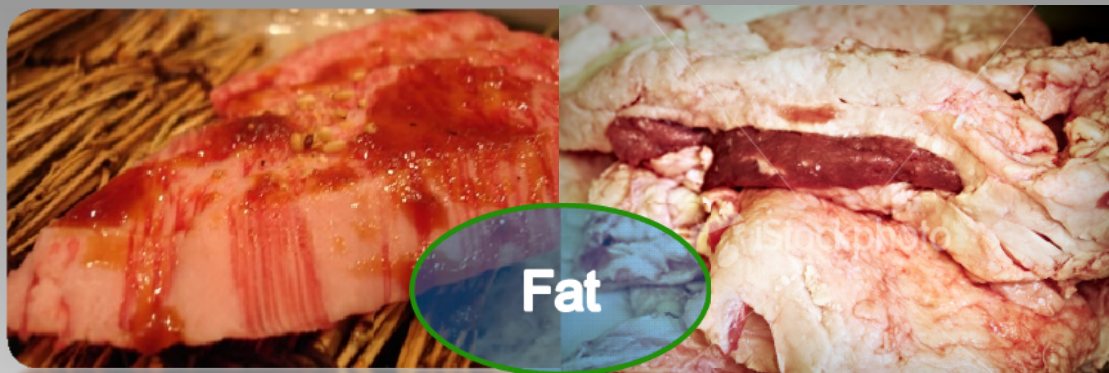
Objective

Objectives



- **To generate biodiesel from animal fat via two-step catalyzed process.**
- **To study the optimize condition of the esterification step by response surface methodology (RSM).**

Materials



Heat



Animal fat of beef tallow from tanning industrial



Experimental Section

Biodiesel Production from Animal Fat via Two-step Catalyzed Process



Esterification
Process



Excess
Alc.

AV<2mg/g
of Oil



Transesterification
Process



Biodiesel

Glycerol

Experimental Section



Method of ASTM D 664

$$\text{Acid Value (mg KOH/g)} = \frac{(A - B) \times N \times 56.1}{W}$$

Vol
KOH

Vol
KOH

Conc. of KOH

Weight of sample



Method



❖ Experimental design

- 5- level-3-factor central composite design (CCD)

Table 1 Coded and actual levels of variables for design of experiment

Variable			Coded value				
			-1.68	-1	0	1	1.68
			Actual value				
Methanol to animal fat ratio	(M)	(v/w)	0.45	0.75	1.205	1.66	1.96
H ₂ SO ₄ concentration	(C)	(% w/w)	0.48	1.50	3.00	4.50	5.52
Reaction time	(T)	(hr)	0.32	1.00	2.00	3.00	3.68

Method



Table 2 Experimental design of 5-level, 3-factor CCD

RUN	M		C		T	
	Co	A	Co	A	Co	A
1	-1	0.75	-1	1.50	-1	1.00
2	-1	0.75	-1	1.50	1	3.00
3	-1	0.75	1	4.50	-1	1.00
4	-1	0.75	1	4.50	1	3.00
5	1	1.66	-1	1.50	-1	1.00
6	1	1.66	-1	1.50	1	3.00
7	1	1.66	1	4.50	-1	1.00
8	1	1.66	1	4.50	1	3.00
9	-1.68	0.441	0	3.00	0	2.00
10	+1.68	1.969	0	3.00	0	2.00

RUN	M		C		T	
	Co	A	Co	A	Co	A
11	0	1.205	-1.68	0.48	0	2.00
12	0	1.205	+1.68	5.52	0	2.00
13	0	1.205	0	3.00	-1.68	0.32
14	0	1.205	0	3.00	+1.68	3.68
15	0	1.205	0	3.00	0	2.00
16	0	1.205	0	3.00	0	2.00
17	0	1.205	0	3.00	0	2.00
18	0	1.205	0	3.00	0	2.00
19	0	1.205	0	3.00	0	2.00
20	0	1.205	0	3.00	0	2.00

When M = Ratio of methanol; C = H₂SO₄ concentration; T = Reaction time
Co = Code value; A = Actual value



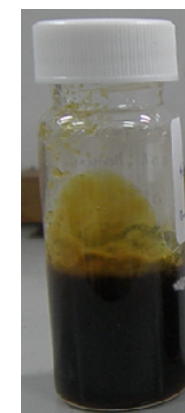
Result & Discussion

Physical and chemical properties of animal fat



Table 3. Fatty acid composition of beef tallow.

Fatty acid	Formula	Structure	wt %
Decanoic	$C_{10}H_{20}O_2$	10:0	0.07
Lauric	$C_{12}H_{24}O_2$	12:0	0.13
Myristic	$C_{14}H_{28}O_2$	14:0	3.51
Palmitic	$C_{16}H_{32}O_2$	16:0	26.50
Palmitoleic	$C_{16}H_{30}O_2$	16:1	7.77
Steric	$C_{18}H_{36}O_2$	18:0	19.24
Oleic	$C_{18}H_{34}O_2$	18:1	40.10
Linoleic	$C_{18}H_{32}O_2$	18:2	1.68
Linolenic	$C_{18}H_{30}O_2$	18:3	0.40
Arachidic	$C_{20}H_{40}O_2$	20:0	0.51
Behenic	$C_{22}H_{44}O_2$	22:0	0.10



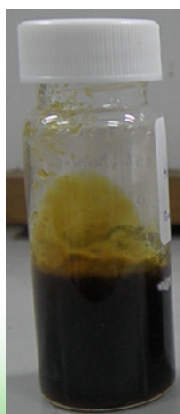
Raw material

Physical and chemical properties of animal fat



Table 4. Physical and chemical properties of animal fat.

Properties	Unit	Method	Amount
Density @ 15 °C	(g/cm ³)	ASTM D1293	0.8928
Kinematic viscosity @ 40 °C	(cSt)	ASTM D445	32.27
Acid value	(mg KOH/g)	ASTM D664	78.80
Water content	(%wt.)	-	0.32
Average molecular weight	g/mole	-	851



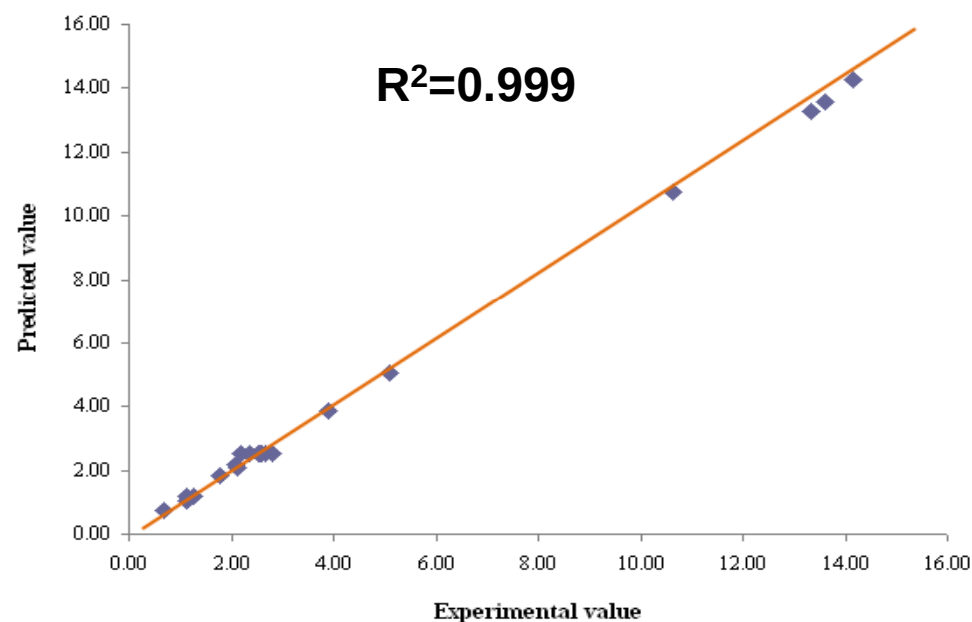
Raw material

Optimization of esterification step



Table 5. Central composite design (CCD) arrangement and response for esterification step.

Treatment	Acid value (mg KOH/g)	
	Experimental	Predicted
1	2.59	2.54
2	2.12	2.06
3	13.61	13.56
4	13.34	13.29
5	1.12	1.06
6	5.11	5.05
7	3.89	3.84
8	1.26	1.20
9	10.65	10.73
10	0.69	0.76
11	1.12	1.19
12	14.16	14.25
13	1.77	1.85
14	2.10	2.17
15	2.82	2.52
16	2.58	2.52
17	2.19	2.52
18	2.54	2.52
19	2.36	2.52
20	2.66	2.52



❖ Lack of Fit

Test	Calculated	Tabular
$F_{0.05(1,5)}$	1.18*	6.61

* Significant at 95% confidence level

Result & Discussion



❖ Model fitting

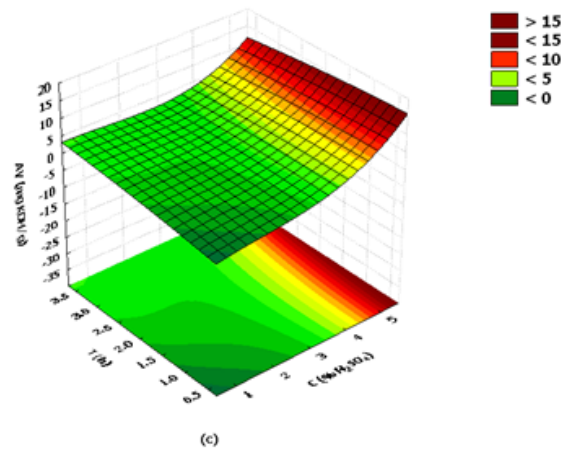
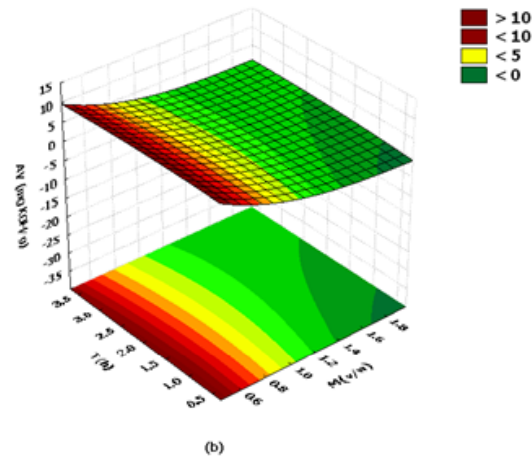
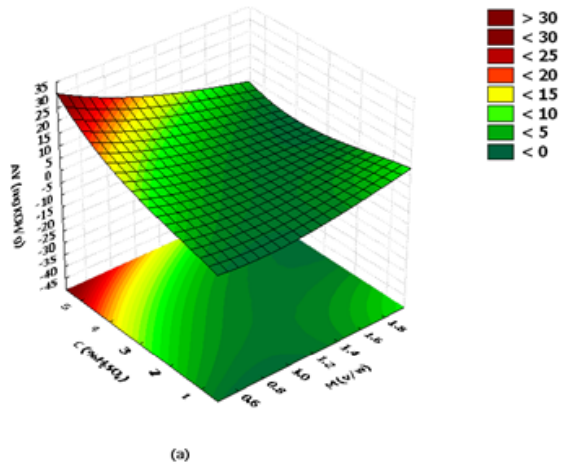
$$Y = b_0 + \sum_{i=1}^5 b_i X_i + \sum_{i=1}^5 b_{ii} X_i^2 + \sum_{i=1}^3 b_{iii} X_i^3 + \sum_{i < j}^2 \sum_{j=1}^3 b_{ij} X_i X_j + \sum_{i < j}^2 \sum_{j < k}^3 \sum_{k=1}^3 b_{ijk} X_i X_j X_k$$

$$\begin{aligned} \text{Acid value} = & 9.865 - 25.159M + 5.033C - 2.682T \\ & + 14.615M^2 - 0.990C^2 - 0.248T^2 - 1.773MC \\ & + 4.324MT + 0.970CT - 2.514M^3 + 0.201C^3 \\ & + 0.011T^3 - 1.249MCT \end{aligned}$$

Result & Discussion



❖ Effect of parameters



Response surface plot showing the effect parameters

(a) H_2SO_4 concentration vs. ratio of methanol

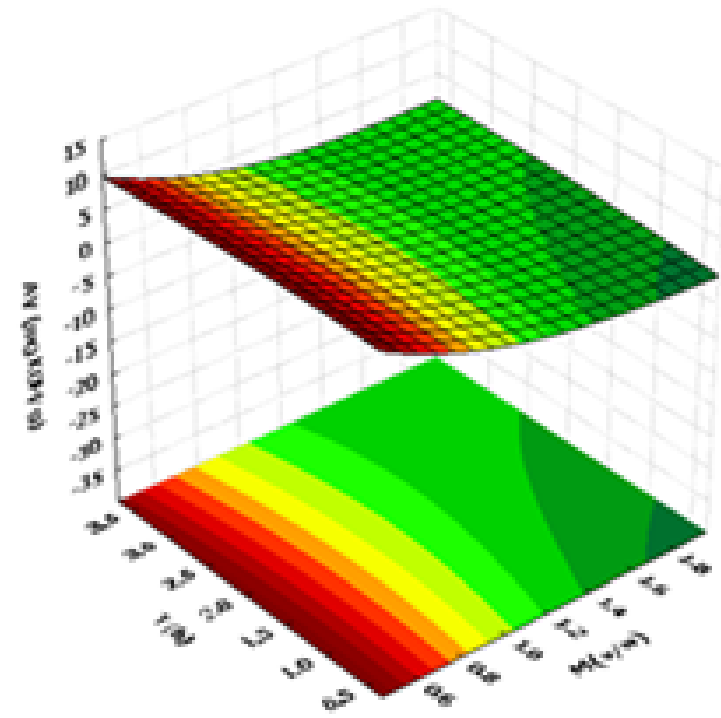
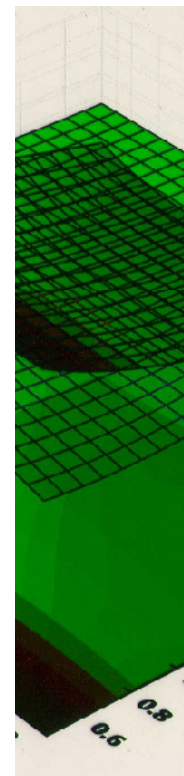
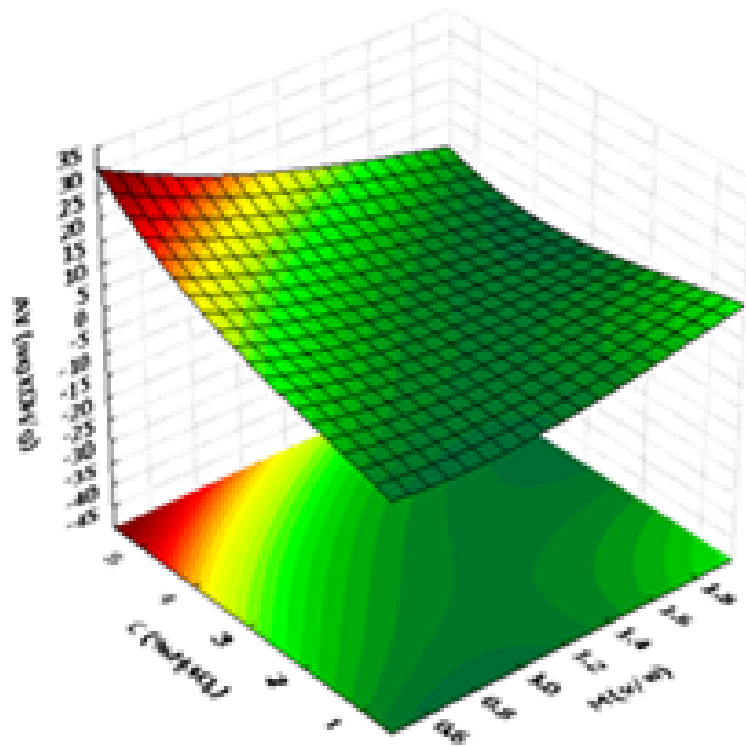
(b) Reaction time vs. ratio of methanol

(c) Reaction time vs. H_2SO_4 concentration

Result & Discussion



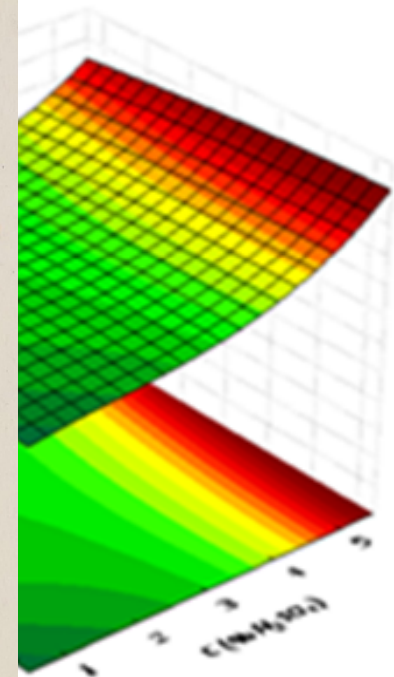
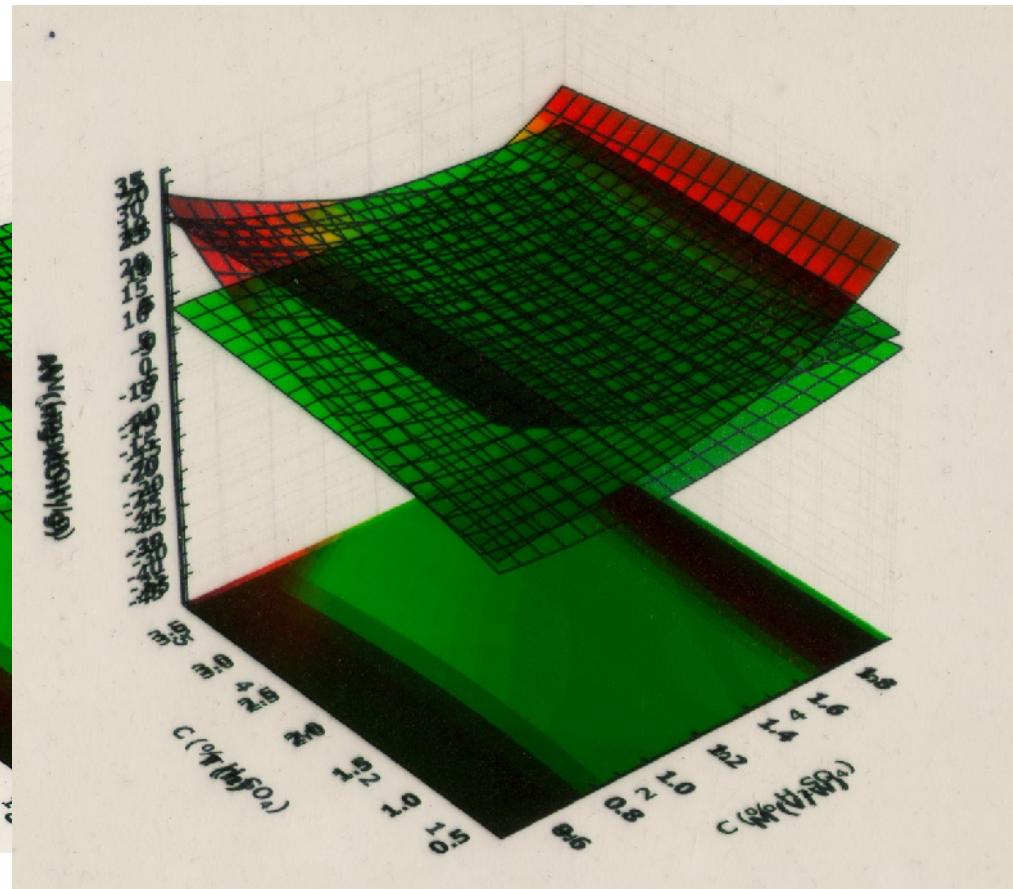
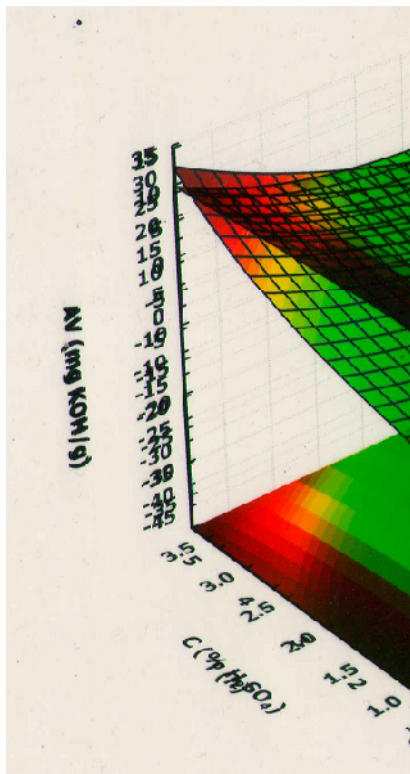
❖ Optimum condition



Result & Discussion



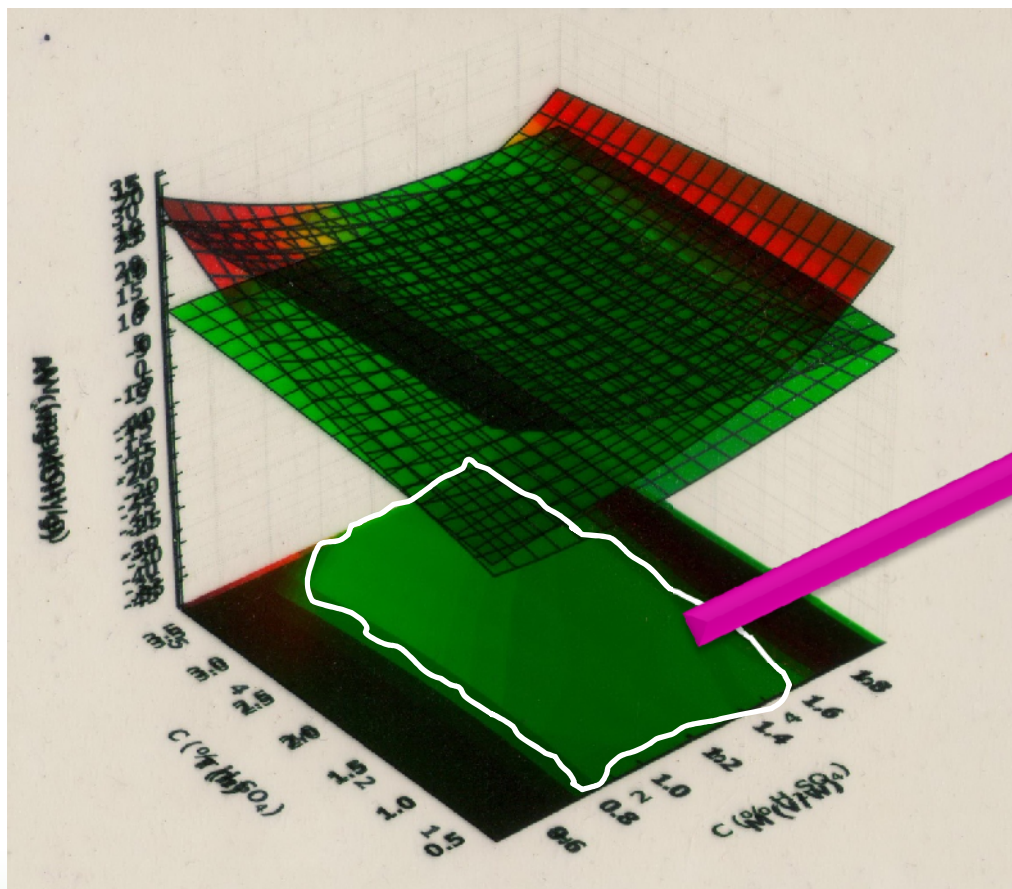
❖ Optimum condition



Result & Discussion



❖ Optimum condition



0.24 – 0.40%(w/w) of acid
1 – 2.5 h of reaction time
1.0 – 1.4 ml (v / w) of
volume of methanol

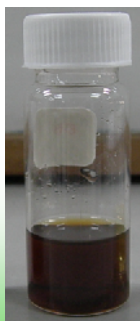
Result & Discussion



❖ Optimum condition

Table 6 Solutions of optimum conditions

Experiment No.	Optimized condition			Acid value (mg KOH/g)	
	M (v/w)	C (% w/w)	T (h)	Experiment	Prediction
1	1.1	2.4	1.0	1.83	1.87
2	1.2	2.7	1.0	1.79	1.80
3	1.3	3.0	1.2	1.77	1.87
4	1.4	3.2	1.5	1.78	1.80
5	1.4	3.3	1.0	1.84	1.81



AV<2mg/g of Oil

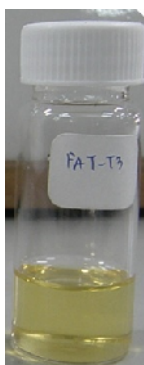
Transesterification step and biodiesel properties



The transesterification reaction was 1.50 h at 60°C, methanol-to-oil mole ratio was 6:1 and 1% KOH.

Table 7 The physical and chemical properties of animal fat methyl ester

Properties	Unit	Animal fat biodiesel (FAME)	ASTM D 6751	EN 14214
Density @15°C	g/cm ³	0.8566	-	0.86-0.96
Viscosity @ 40°C	cSt	5.06	1.9-6.0	3.50-5.00
Flash point	°C	170	120 min	130 min
Acid value	mg KOH/g	0.53	0.80 max	0.50 max
Total glycerin	%wt.	0.07	0.24 max	0.25 max
Free glycerin	%wt.	0.00	0.02 max	0.02 max
Monoglycerin	%wt.	0.11	-	0.80 max
Diglycerin	%wt.	0.23	-	0.20 max
Triglycerin	%wt.	0.02	-	0.20 max



Biodiesel



Conclusion

Conclusion



- ❖ The result of transesterification step
 - methyl ester content at 86.10 % and most properties of animal fat methyl ester met the ASTM D6751 and EN 14214 biodiesel standard.

Acknowledgement



Excellence Oil Palm Kasetsart University, Thailand



Biodiesel station, Vehicle, Building and Physical Plant
Division Kasetsart University, Thailand



GREEN Energy FOR COMMUNITY
BIODIESEL





Thank You

