



SYNTHESIS WAX ESTERS FROM PALM FATTY ACID DISTILLATE AND OLEYL ALCOHOL OVER AMBERLYST15

Presented By

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Thursday, Nov 26th, 2009, 13.15 – 13.30 p.m

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Outline



1 Introduction

2 Experiments

3 Results and discussion

4 Conclusions



INTRODUCTION

Wax esters



Wax esters are long chain esters which are generated from the reaction between long chain fatty acid and long chain alcohol that consist of carbon atom more than 12 atoms

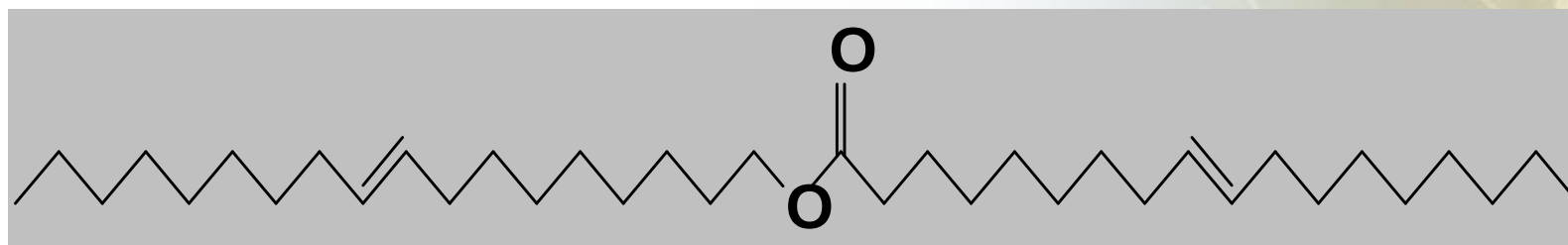
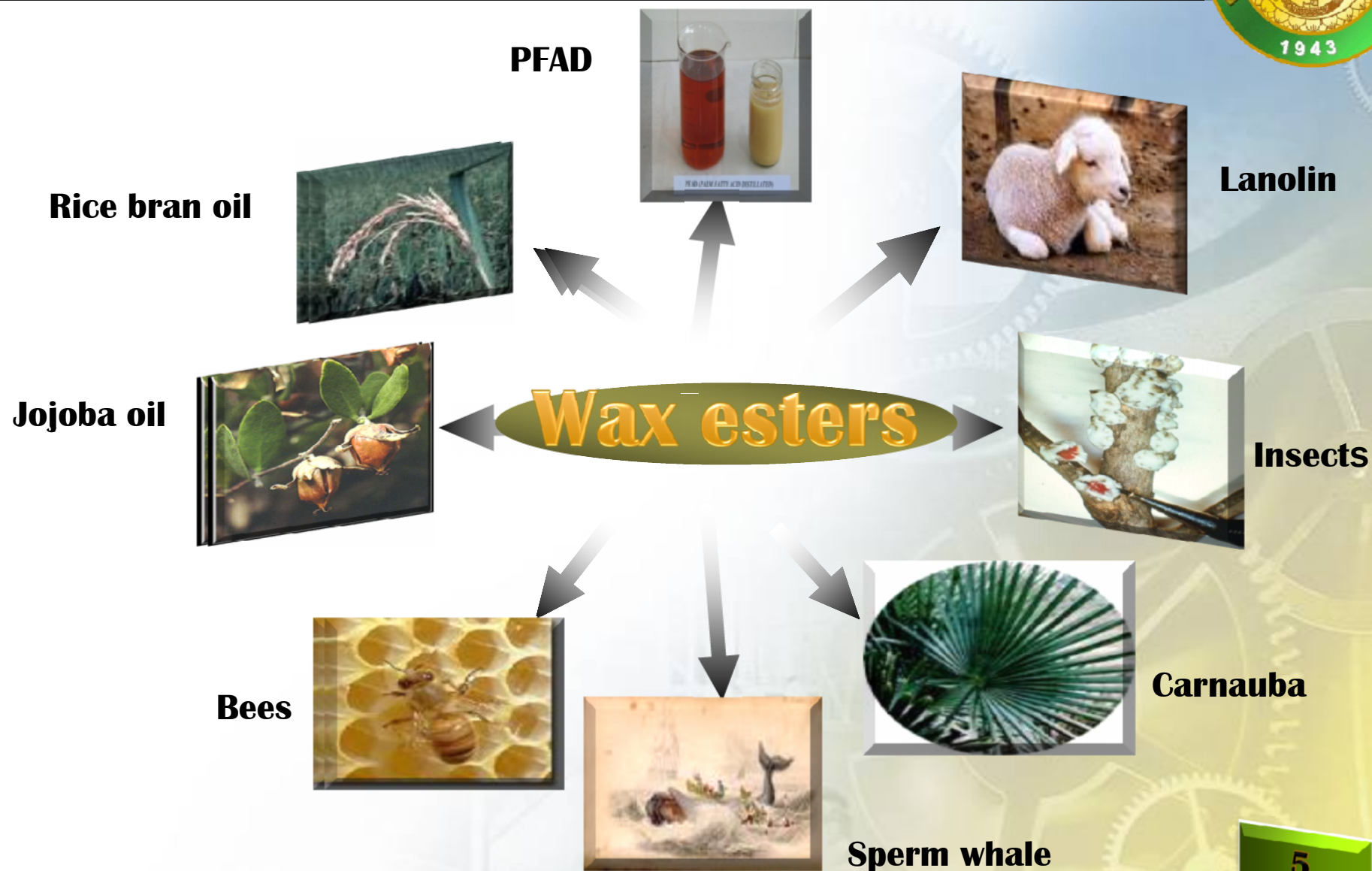
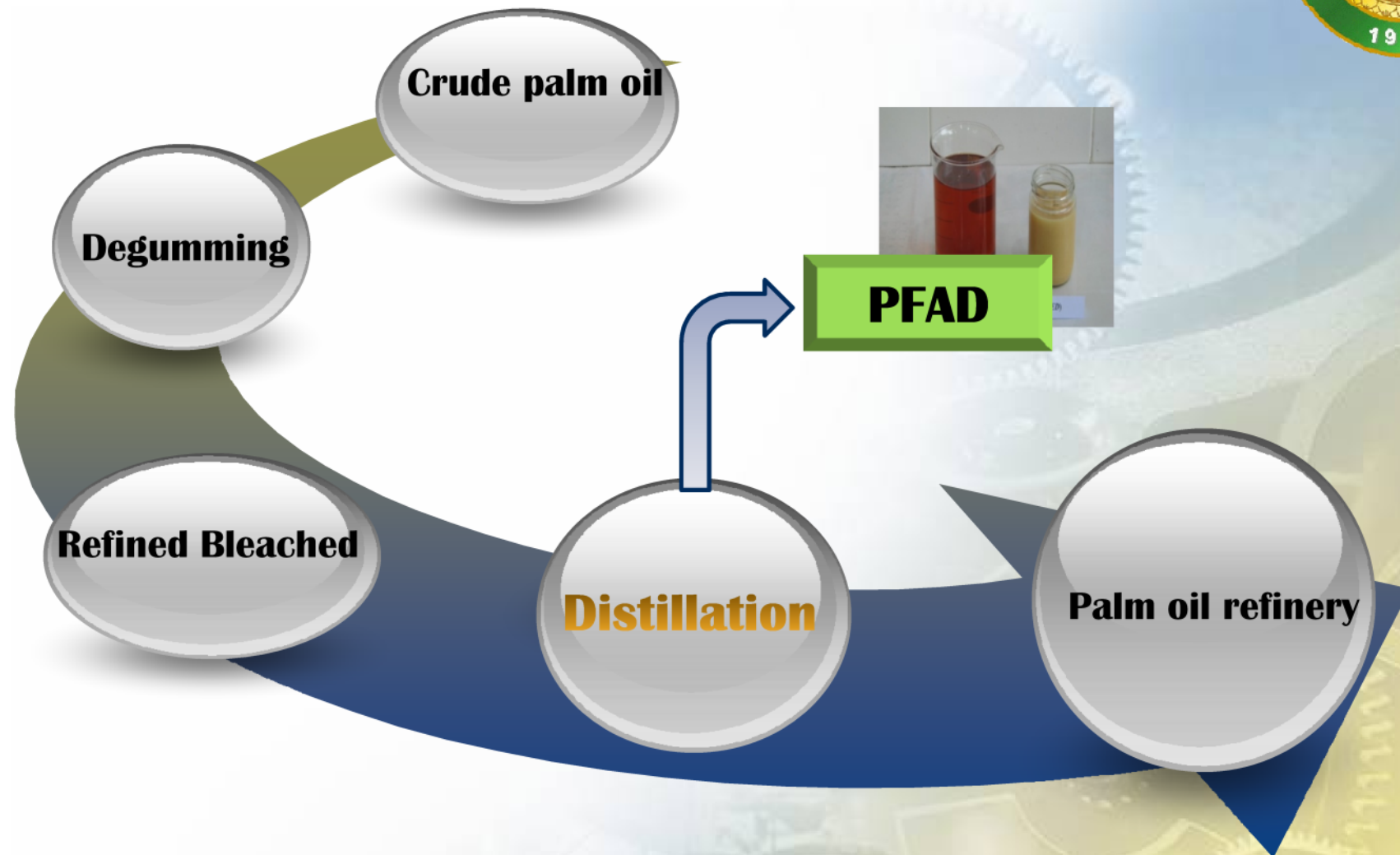


Fig1. Structure of wax ester

Sources of wax esters



Palm Fatty Acid Distillate (PFAD)



Esterification reaction

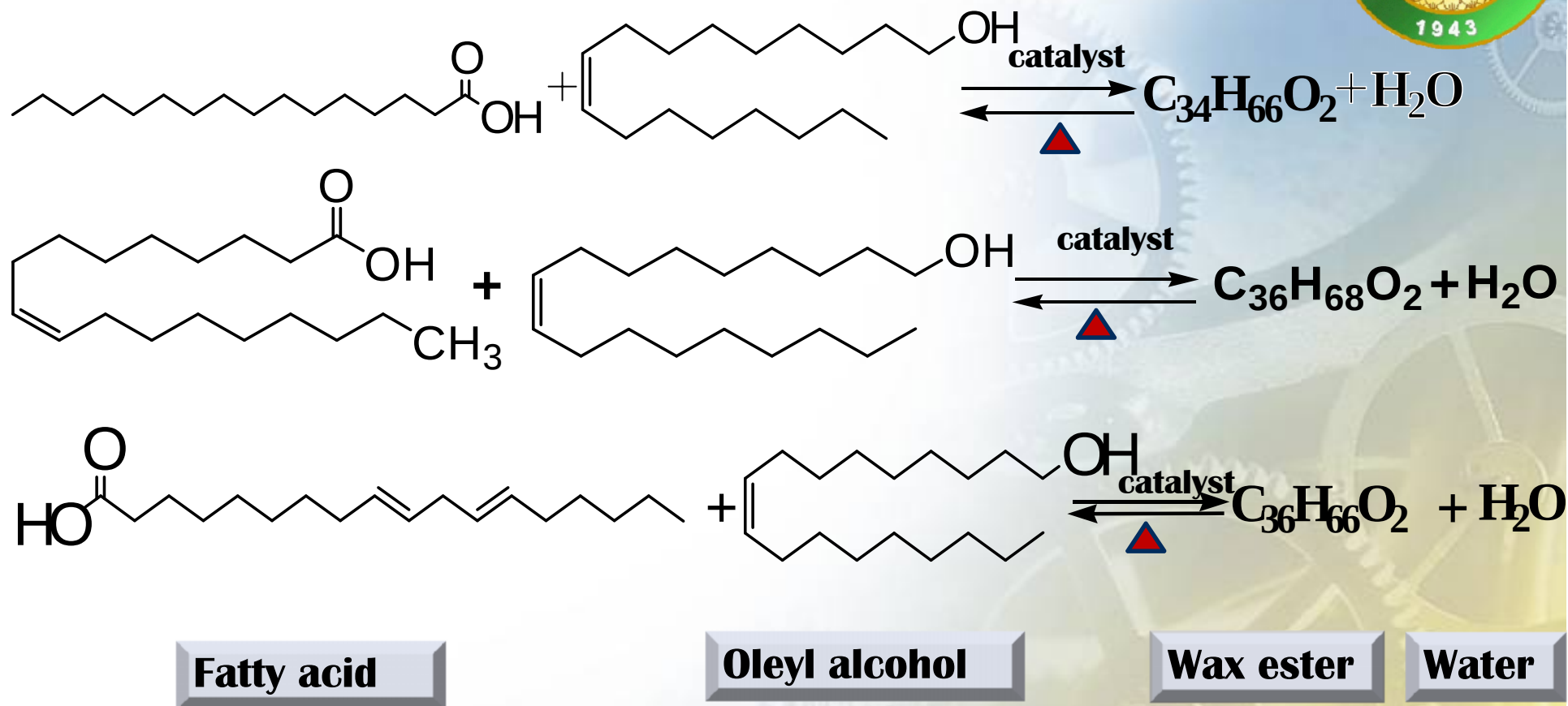


Fig 2. Esterification reactions of PFAD to produce wax esters

Application



Fruit Coating



Cosmetics



**Plasticizers
and polishes**



Lubricants



Pharmaceuticals



Objective



To study the optimum condition for synthesis liquid wax esters from Palm Fatty Acid Distillate and Oleoalcohol

- Effect of reaction time
- Effect of reaction temperature
- Effect of Molar Ratio of oleyl alcohol to PFAD
- Effect of amount of Amberlyst15
- Effect of agitation speed

To study the physical properties of wax esters

- Refractive index
- Saponification value
- Iodine value
- Acid value
- Density



EXPERIMENT

Materials



1 Palm Fatty Acid Distillate(PFAD)

2 Oleyl alcohol

3 Amberlyst15

5 Standard esters

Oleyl oleate, Oleyl linoleate, Oleyl palmitate

Methyl laurate for internal standard

Methods



Reaction mixture

Separate of wax esters and water

Analyst; %FFA, Oleyl esters, FT- IR, Physical properties

- **Temp= 50-90 °C**
- **Time = 0.5-9 h**
- **Speed =125-1250 rpm**
- **Ratio PFAD to alcohol = 1:1-1:3**
- **Catalyst = 10-60%**

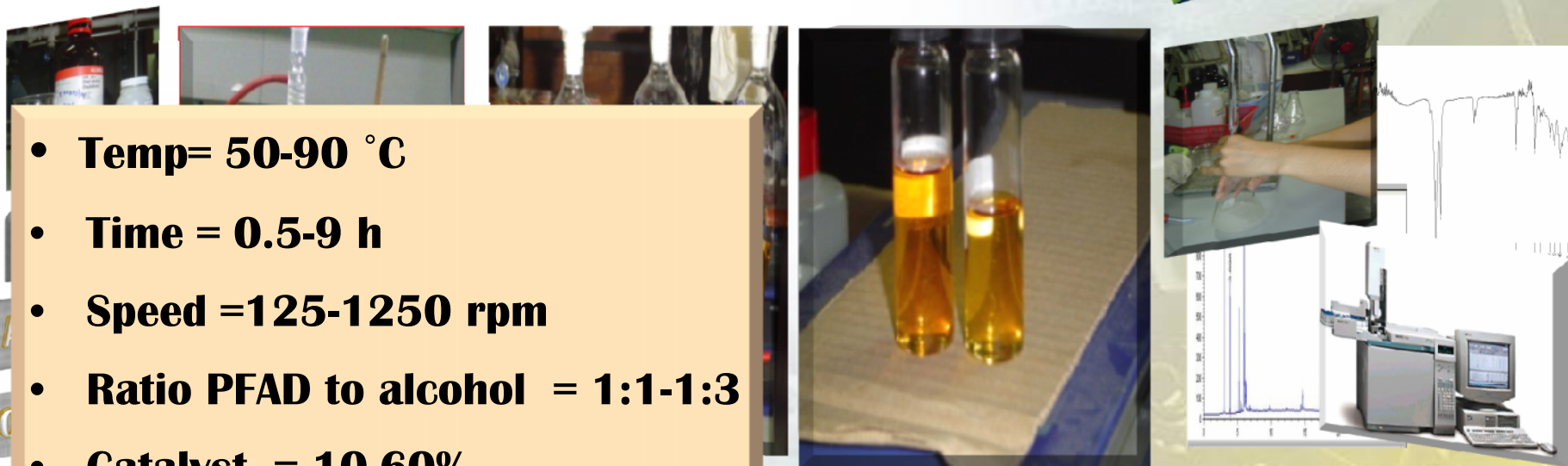


Fig 3. Process for synthesis wax esters

Analysis product wax esters



Wax esters

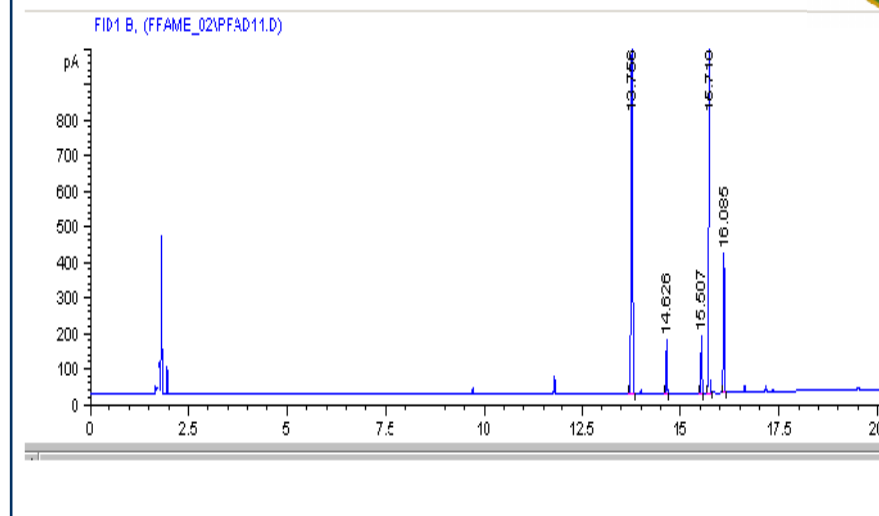
Water

- ☐ The composition of wax esters by GC
- ☐ %Conversion of FFA by titration
- ☐ The confirm wax esters by FT-IR
- ☐ The physical properties of wax esters



RESULTS AND DISCUSSION

Table 1. The fatty acid composition of Palm fatty acid distillate (PFAD)



Fatty acid	(%)
Myristic acid(C ₁₄)	0.86
Palmitic acid(C ₁₆)	42.49
Stearic acid(C ₁₈)	3.35
Oleic acid(C _{18:1})	34.77
Linoleic acid(C _{18:1})	8.25

The optimum condition for synthesis wax esters



Effects of reaction time

Fig 4. Effect of reaction time on % onversion

Effect of amount of catalyst

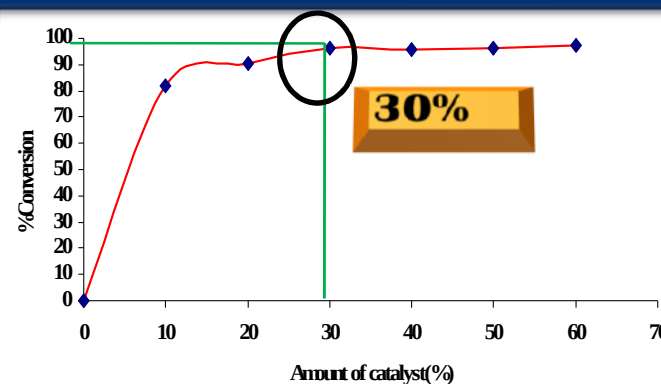


Fig 5. Effect of amount catalyst on % conversion

Effect of reaction temperature

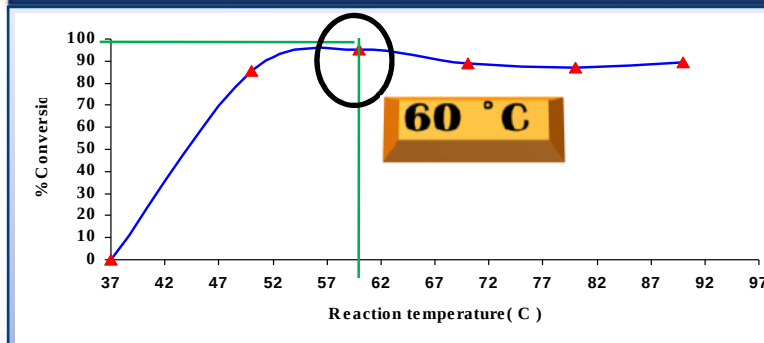


Fig 6. Effect of reaction temperature on % conversion

The optimum condition for synthesis wax esters



Effect of Molar Ratio of oleyl alcohol to PFAD

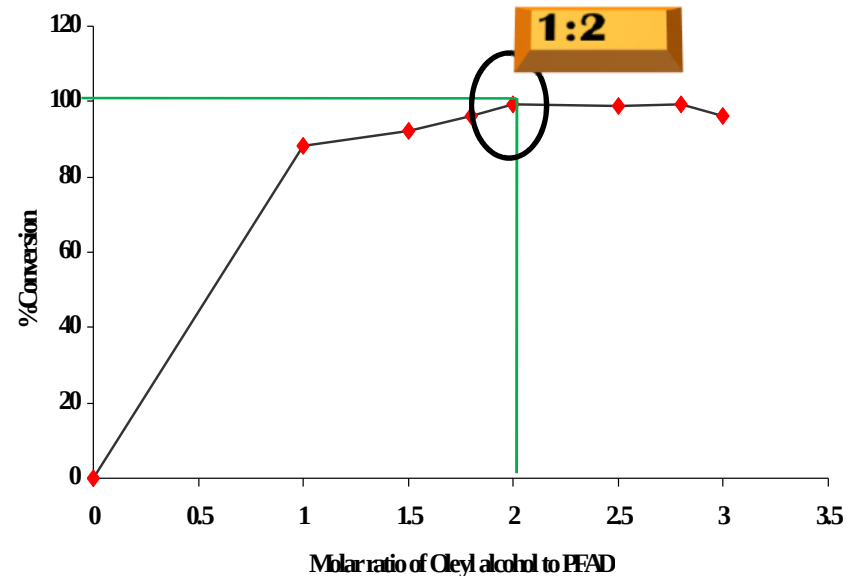


Fig 7. Effect of Molar ratio of oleyl alcohol to PFAD on % Conversion

Effect of agitation speed

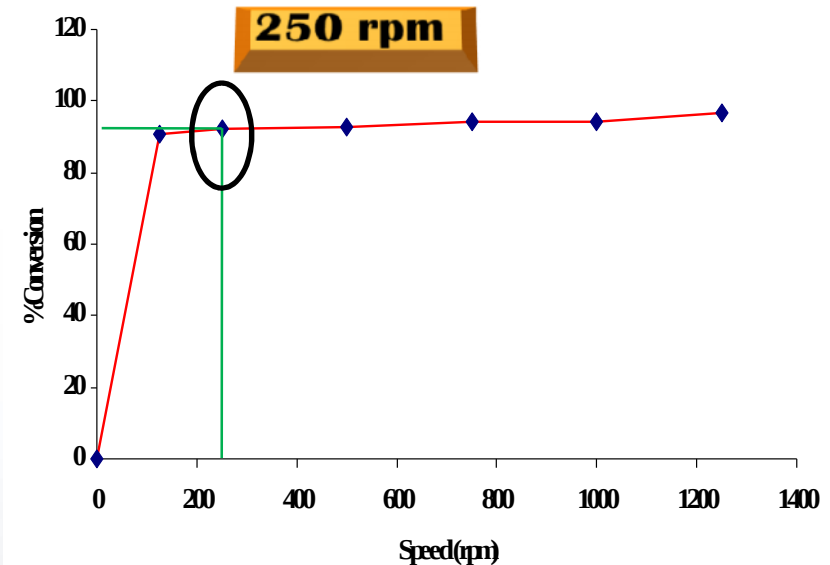


Fig 8. Effect of agitation speed on % Conversion

Confirmation wax esters by GC

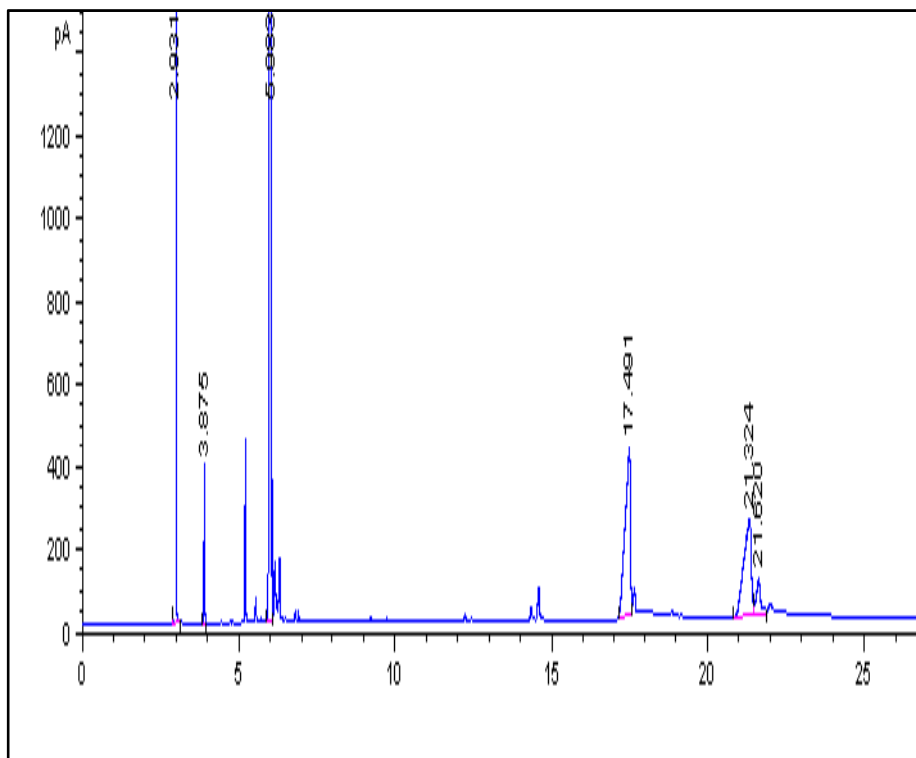


Table 2. Retention time of Oleyl esters

Retention time (min)	Compound
3.920	Methyl laurate
17.401	Oleyl palmitate
21.320	Oleyl oleate
21.520	Oleyl linoleate

Fig 9. Chromatogram of wax ester by GC

Confirmation Wax esters and PFAD by FT-IR

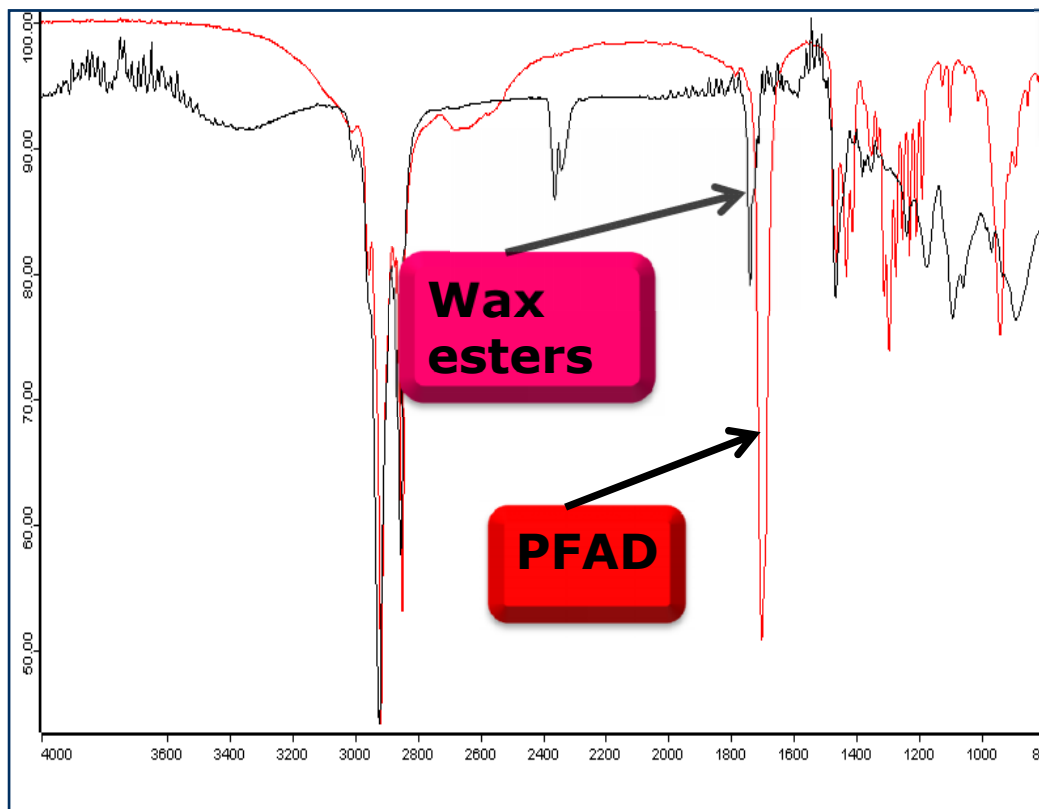


Table 3. Functional group Oleyl esters

Frequency (cm ⁻¹)	Functional group
1699	C=O carboxylic group of PFAD
1739	C=O ester group of wax esters

Fig10. spectrum of wax esters and PFAD by FT-IR

Table 4. Physical properties of PFAD and wax esters



Characteristics	PFAD	Wax esters ¹	Wax esters ²
Refractive index	-	1.460	1.456
Density(g/cm ³)	-	0.856	0.839
Saponification value (mg KOH/g sample)	200.08	69.36	93.10
Iodine value(gI ₂ /100g)	57.57	83.18	69.70
Acid value(mg KOH/g)	170	0.5	0.6
% Water	0.05	0.05	-

1; My research

2; Keng *et al.* (2008)



CONCLUSIONS

The optimum condition to produce wax esters



- 1 Reaction temperature 60 °C
- 2 Reaction time; 60 min
- 3 Amount of Amberlyst15; 30% wt
- 4 Molar ratio of oleyl alcohol to PFAD; 2:1
- 5 Agitation speed 250 rpm
- 6 Analysis of yields of esters show that >80%
- 6 FT-IR confirmed the occurred esters peak in wax esters

ACKNOWLEDGEMENTS



- **Department of Chemistry, Faculty of Science, Kasetsart University**
- **Thai Lube Base (Public Company)., Ltd.**
- **Center of Excellence-Oil Palm of Kasetsart University.**
- **Thailand Research Fund and Faculty of Science, Kasetsart University**
- **The donation of PFAD from Patum oil co., Ltd.**



Thank You !

Amberlyst-15



Amberlyst-15 is a strongly acidic sulfonic acid, macroreticular polymeric resin based on crosslinked styrene divinylbenzene copolymers. It has the optimal balance of surface area, acid capacity and a Pore diameter to make it the catalyst of choice for esterification reaction

ผลการวิเคราะห์พื้นที่ผิวสัมผัสของ **Amberlyst-15** ด้วยเครื่อง **BET** เท่า **48.02 m²/g**,

Pore volume เท่า **0.30 cc/g**, **Particle Size** เท่า **0.3-1.2 mm**

ผลการวิเคราะห์องค์ประกอบของธาตุ ใน **Amberlyst-15** ด้วยเครื่อง **Energy**

Dispersive x-rays Spectrometer (EDS) ผลดัง ตาราง

องค์ประกอบของธาตุ	%
C	43.50
O	22.60
Mg	0.43
Al	5.01
S	26.8
Ca	2.17
Cr	0.91
Cu	6

<u>Media Grade - Porosity</u> (um)	<u>Functional Group</u>	<u>Matrix</u>	<u>Temperature Limit,</u> (C.)	<u>Fine Contents</u> (mm)	<u>Total Exchange Capacity</u> (ek/g)	<u>Ionic Form</u>	<u>Process</u>
250	S03H	Macrorreticular	120	< 0.300	4.7	Hydrogen	Alkylation, esterification, etherification

Table 1 The properties of Amberlyst 15 (solid acid catalyst)

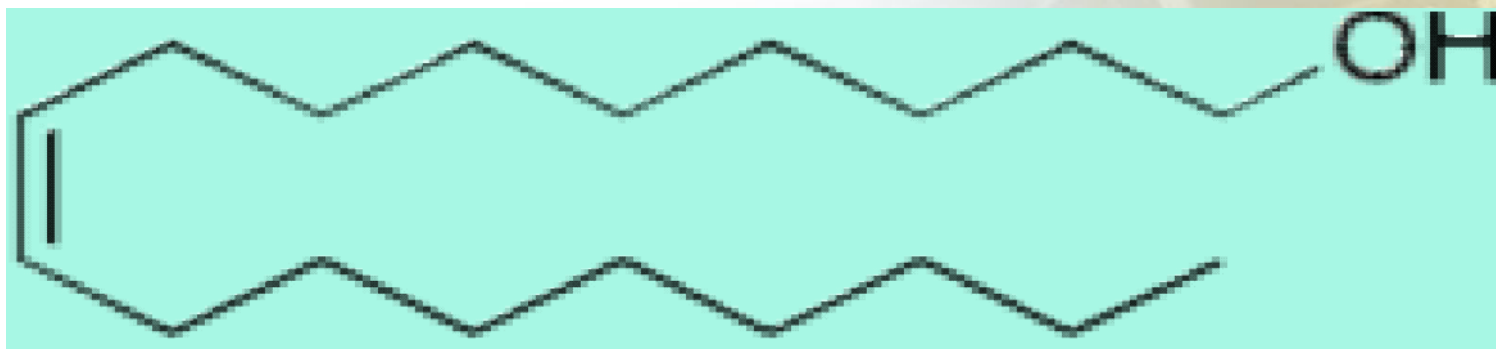
Matrix	Styrene– divinylbenzene
Form	H⁺
Crosslinking level	Hydrogen bonding
Exchange capacity (meq/g)	4.7
Average pore diameter (nm)	30
Particle diameter (mm)	0.74
Surface area(m²/g)	53
Moisture	<2%
Temperature_{max} (° C)	120
Porosity (%)	33

Oleyl alcohol

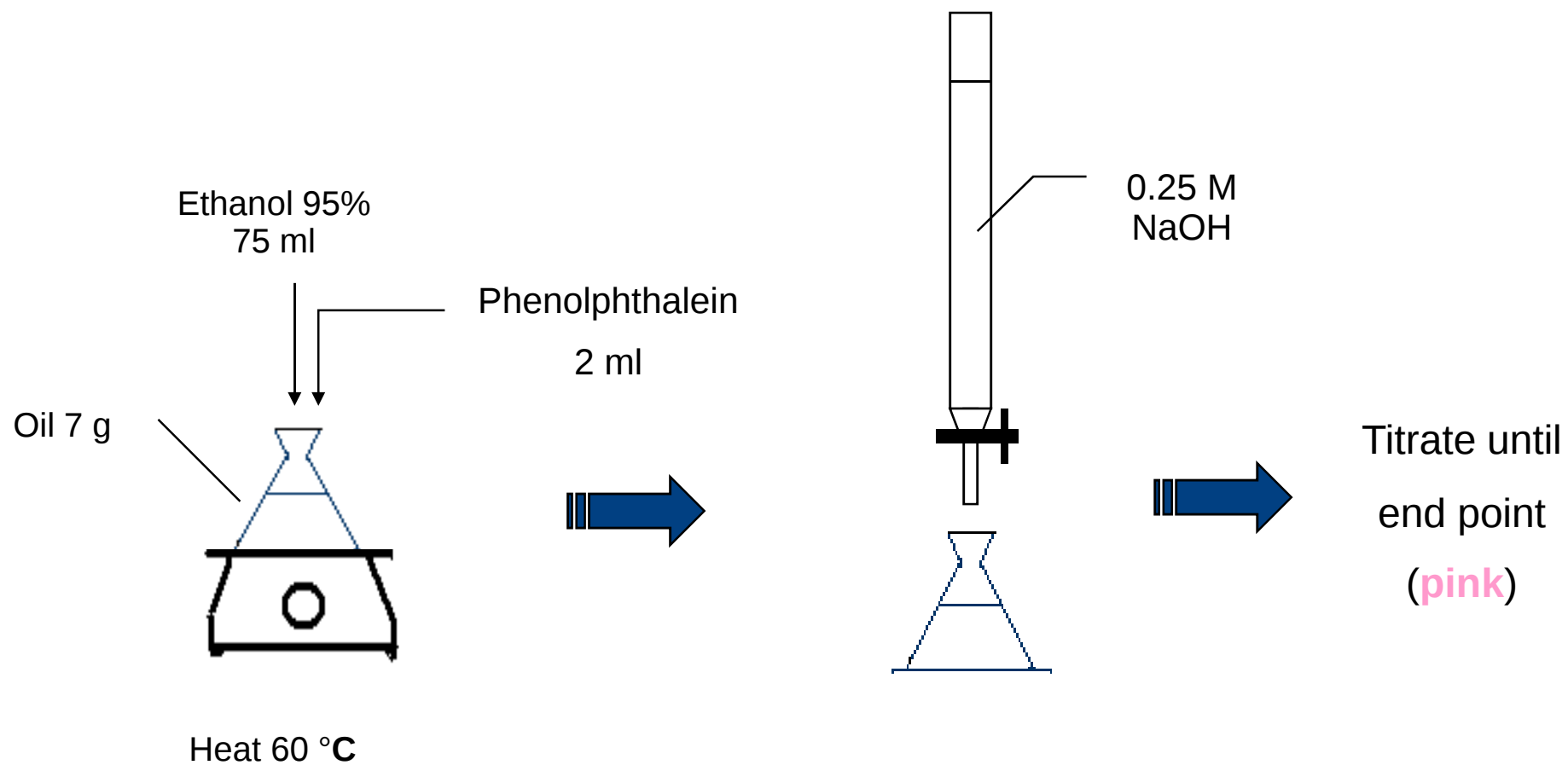


Non-ionic, Unsaturated fatty alcohol

It has uses as a nonionic surfactant, emulsifier, emollient and thickener in skin cream, lotion, cosmetic



Determined Free Fatty Acid





The concentrations of palm wax ester

$$C_x = (A_x/A_{IS}) \times (C_{IS} \times D_{Rf}^{IS}/D_{Rf}^X)$$

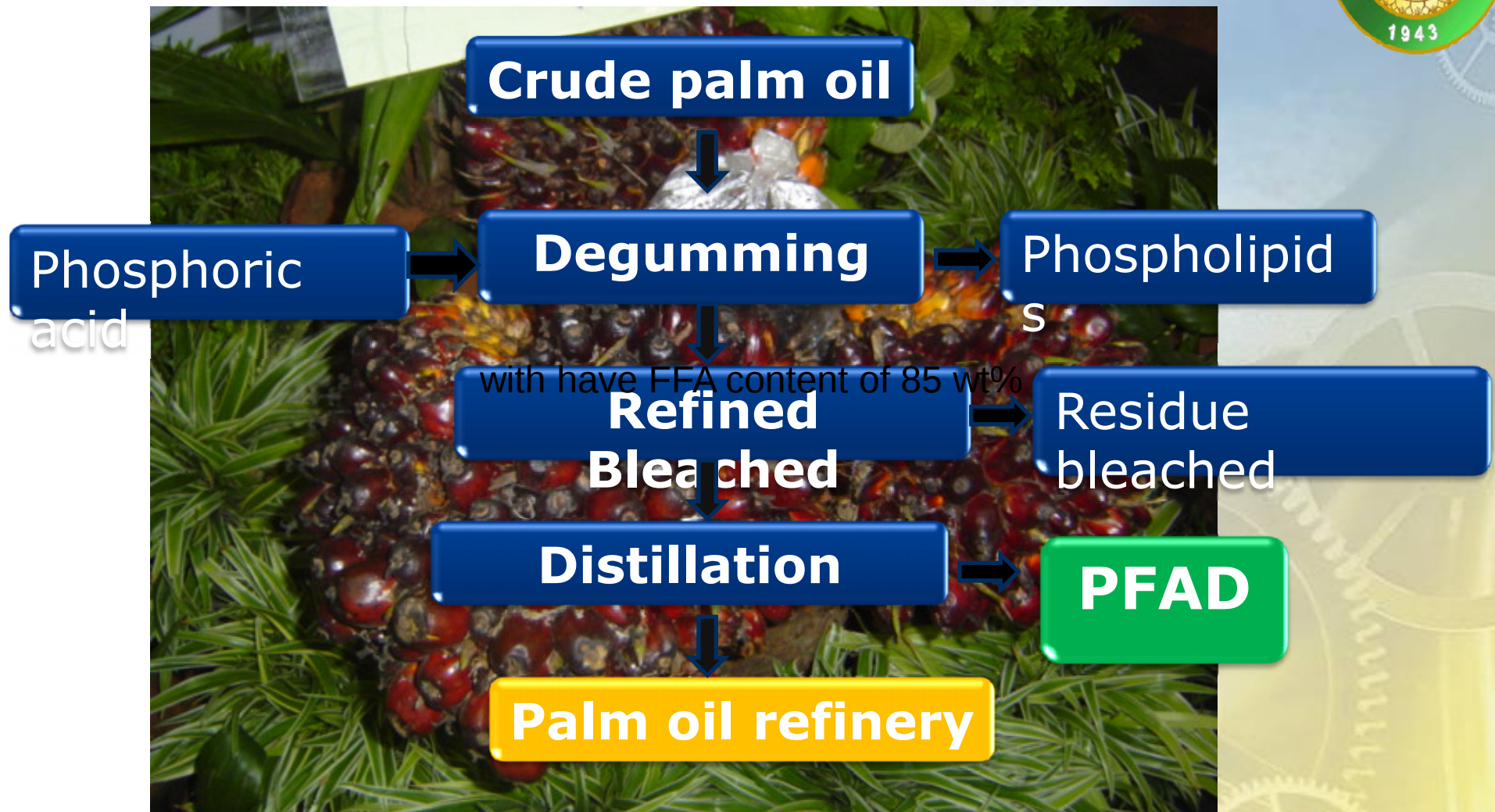
C = Amount of component x ,

A = Area of component x or internal standard ,

D_{Rf}^{IS} = Detector response factor for component x or
internal standard

$$D_{Rf}^X = A_x/C_x \quad , \quad D_{Rf}^{IS} = A_{IS}/C_{IS}$$

Palm Fatty Acid Distillate



Ethanol 95%
75 ml

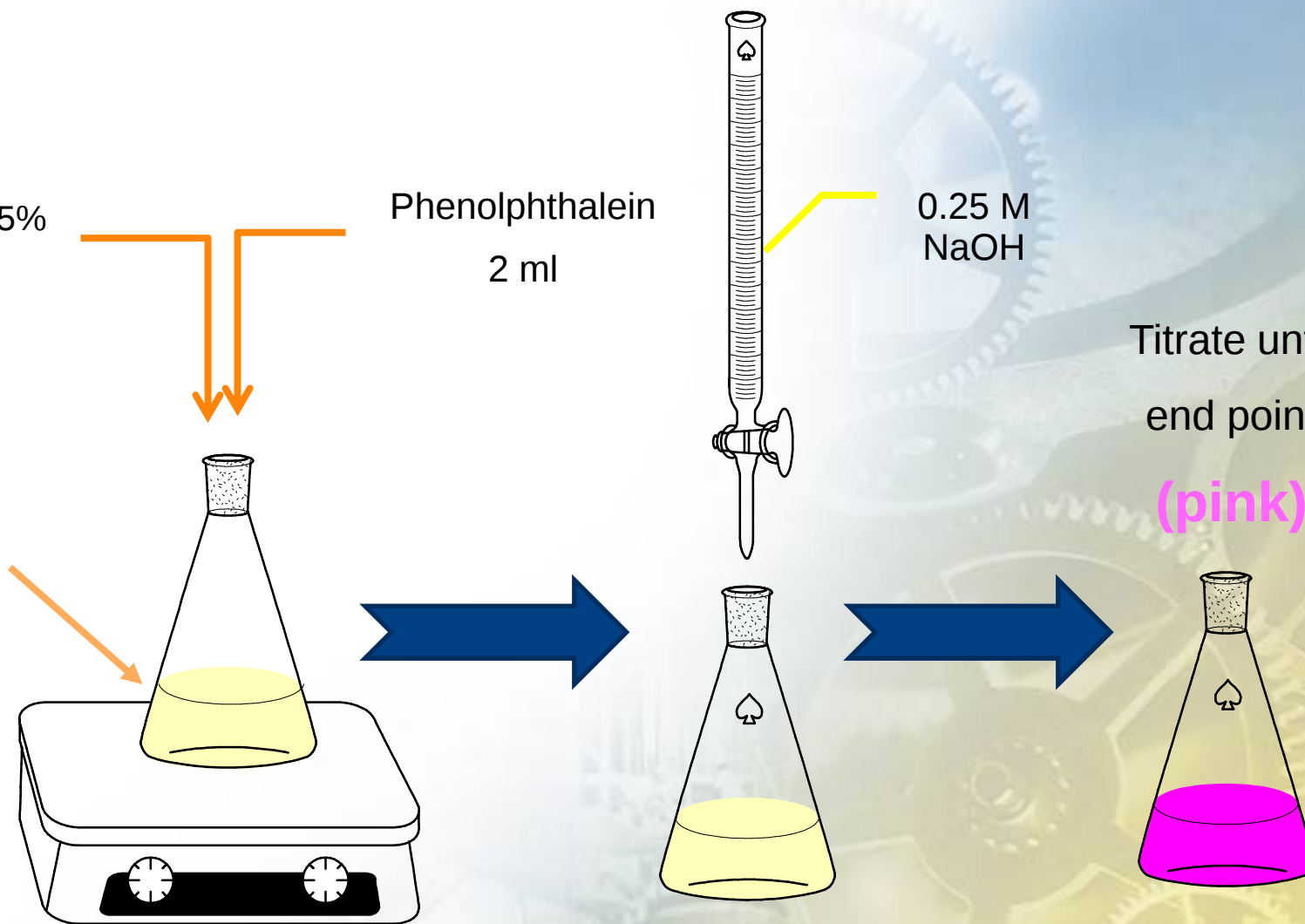
Phenolphthalein
2 ml

0.25 M
NaOH

Oil 7 g

Titrate until
end point
(pink)

Heat 60 °C



Esterification reaction

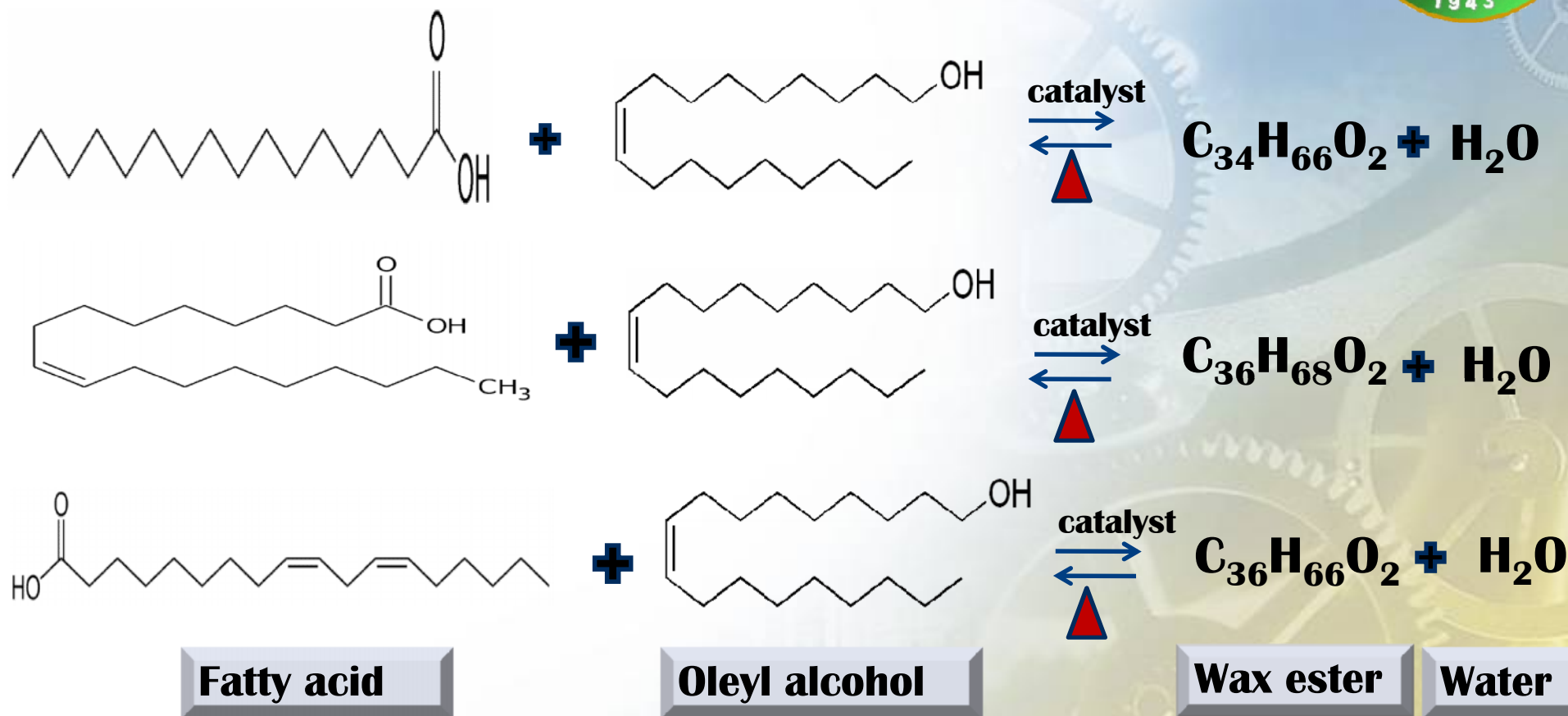


Fig 2. Esterification reactions of PFAD to produce wax esters