# SYNTHESIS, CHARACTERIZATION AND CATALYTIC PROPERTIES OF CALCIUM METHOXIDE FOR TRANSESTERIFICATION OF NON-EDIBLE *JATROPHA CURCAS* AND *NANNOCHLOROPSIS OCULATA* OILS TO BIODIESEL


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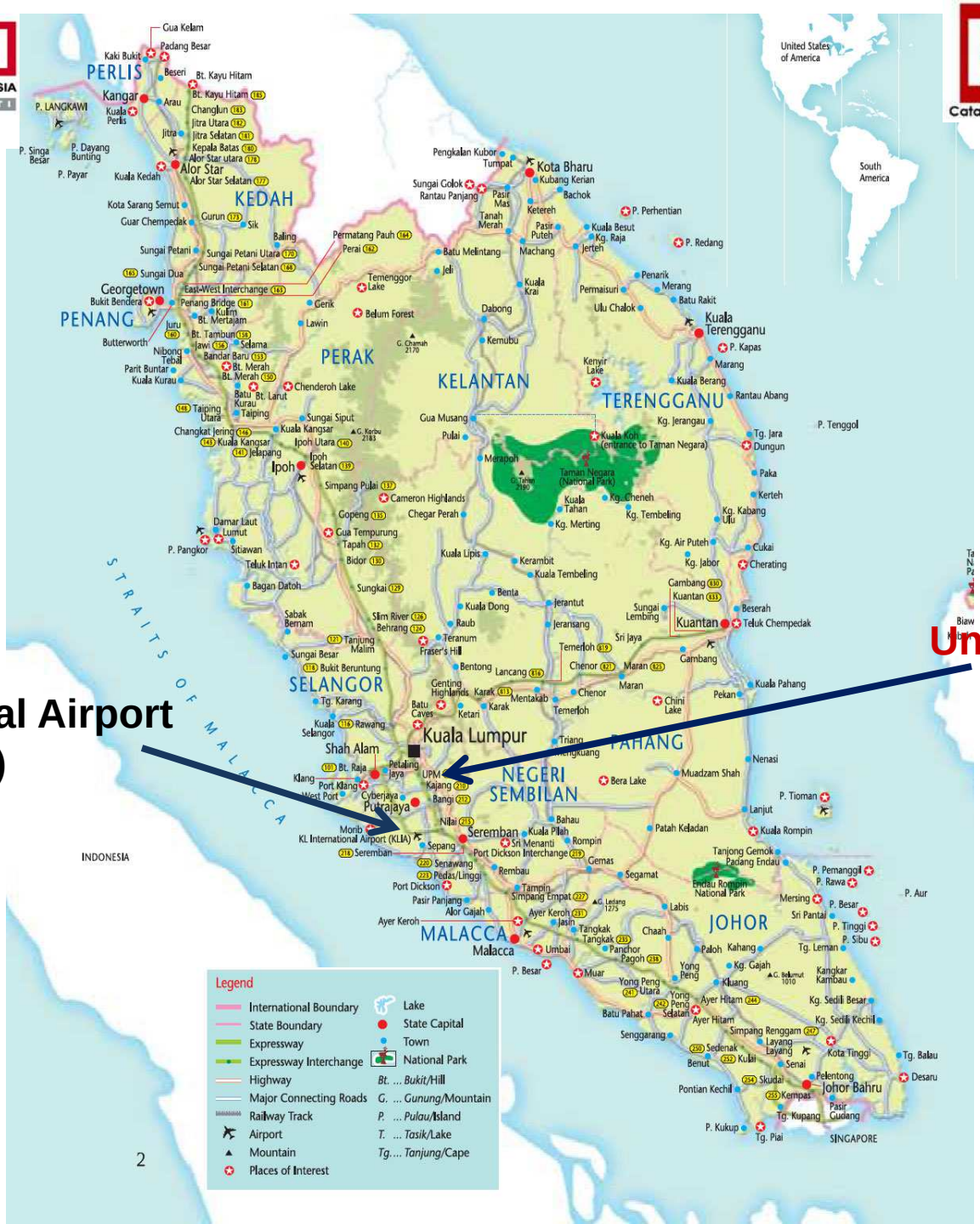


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# Selamat Datang (Welcome)
# ke (to)
# Universiti Putra Malaysia

UNIVERSITI PUTRA MALAYSIA
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Catalysis Science and Technology Research Centre

1931 – School of Agriculture
1946 - Malayan Agricultural College
1971 - Universiti Pertanian Malaysia
1997 - Universiti Putra Malaysia
(one of the first 4 Research Universities in Malaysia)

Main Campus Size - Serdang 1108.103 hectares
Branch Campus Size - Bintulu 714.717 hectares

| | | |
|---|---|---|
| Faculty | - | 16 |
| Institute | - | 9 |
| Centre | - | 16 |
| School | - | 2 |
| Academy | - | 1 |

| | | |
|---|---|---|
| Number of Undergraduates | - | 15,350 |
| Number of Postgraduates | - | 10,266 |
| Total Students | - | **25,616** |
| Number of Lecturers and Tutors | - | 1,848 |
| Number of Full Professors | - | 189 |

# Is Biofuel a good alternative to Fossil fuel?

❑ Alternative to fossil fuels produced from renewable sources.

❑ Non-toxicity, biodegradability, free sulfur & aromatic content, *i.e.* 0 % $CO_2$

❑ Similar to petroleum diesel combustible properties, *i.e*. flash point & cloud point


❑ One of the major pollutant in the world, i.e. heart & lung

❑ Is fossil fuel that released significant amount of $CO_2$ into atmosphere when burning it.




M. G.Kulkarni, A. K. Dalai, N. N. Bakhshi, (2007) *Bioresource Technology,* 98: 2027-2033.

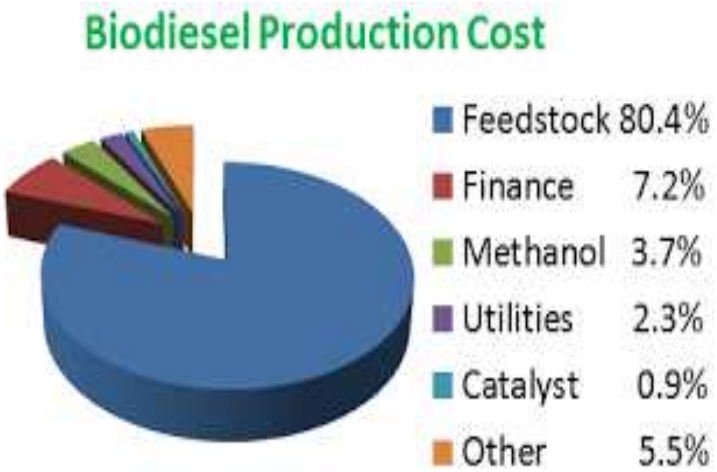
# Fuel: What is Advanced Biofuel ?



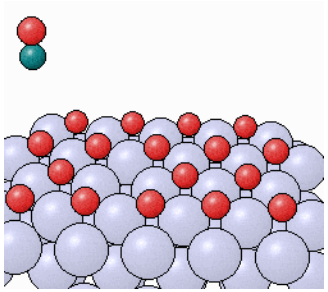

**1st generation**



**2nd generation**




Feedstock



Catalyst

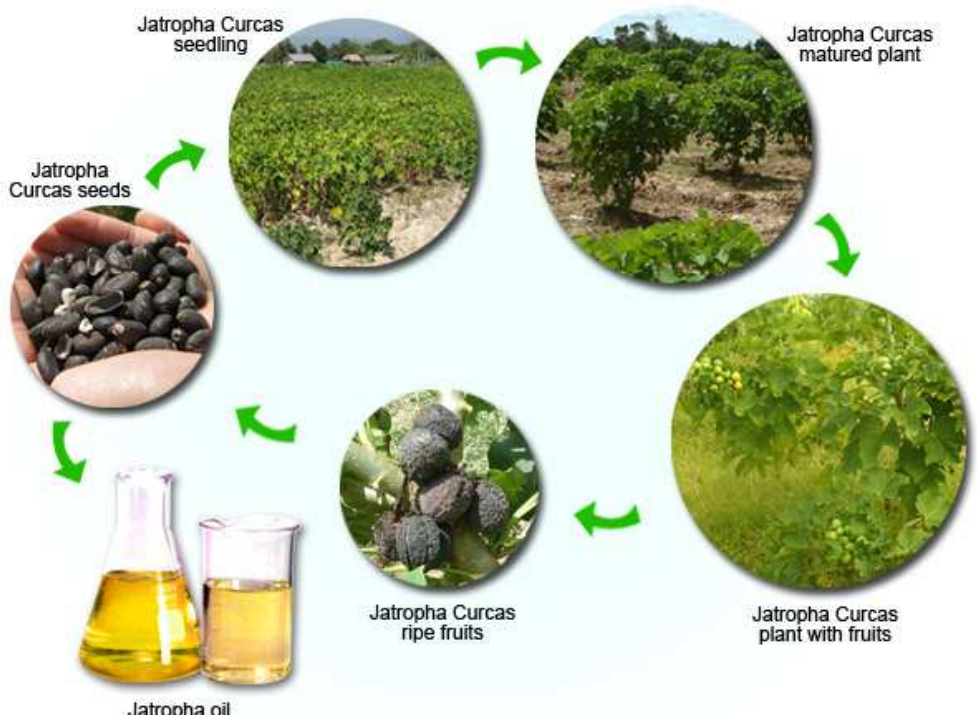
Methanol

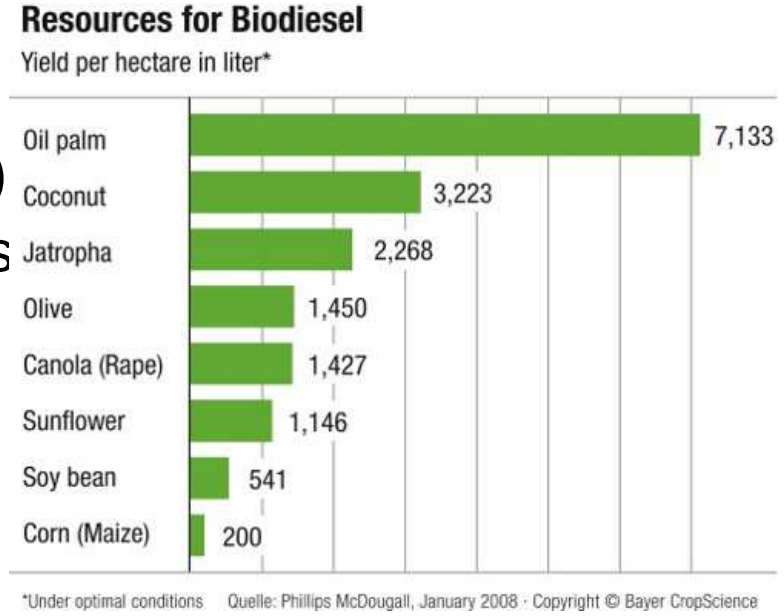

Masjuki, H. H., Mahlia, T. M. I., Choudhury, I. A. and Saidur, R. 2002. Potential $CO_2$ reduction by fuel substitution to generation electricity in Malaysia. *Energy Conversion and Management*, **43(6):** 763-770.

Lim, S. and Teong, L. K., 2010. Recent trends, opportunities and challenges of biodiesel in Malaysia: an overview. R*enewable & Sustainable Energy Reviews*, **14(3):** 938-954.

# Why Jatropha for biofuel?

- Locally known as “jarak pagar” in **Malaysia** (*Jatropha curcas*)
- **Fast-growing**, easily propagated by cuttings
- A yield of 5-10 ton/ha/year (up to 40 % oil content)
- Non-competitive to other crops
- Grow in arid and semi-arid areas
- Not browsed by animals (**non-edible**)
- Oil increase trade among all countries




# Why microalgae for biodiesel ?


BIOETHANOL

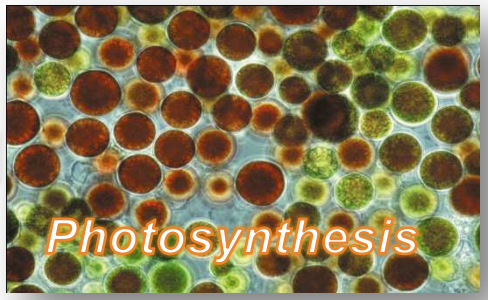
BIO-OIL

BIODIESEL

BIOMETHANE

BIOHYDROGEN

FOOD & PHARMACEUTICAL RELATED PRODUCTS





$H_2O + CO_2 + NH_3 + P_2O_5 + Photons$

↓

Biomass ($CN_xH_yO_z$) + $O_2$

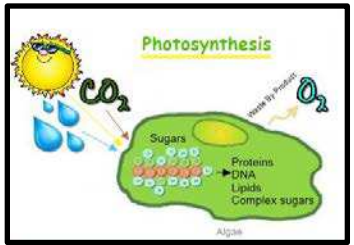
# Why microalgae for biofuel?


**Food vs fuel**: does not compete with food supply

**Adaptability & high growth rate**: > 50 times faster than land based plant & consumes very less water compare to land crops

**Lesser land requirement**: limited land resources make solved the potential biomass insufficient

**High oil yield** (15-300 time more oil than traditional crops Above 50 %, some as high as 75 %)
-Microalgae: 5000 to 15000 gallons/acre/year
-Oil Palm: 635 gallons/acre/year
-Sunflower: 102 gallons/arce/year




**High $CO_2$ capture capacity**: high photosynthesis efficiencies ($CO_2$ convert to $O_2$) – algae in carbon cycle

# Comparison of some sources of biodiesel and oil content of some microalgae

| Crop | Land Use ($m^2$/year/Kg biodiesel) | Oil Content (% oil by wt. in biomass) | Oil Yield (1 L/ha/year) | Biodiesel Productivity (Kg biodiesel/ha/year) |
|---|---|---|---|---|
| Corn | 66 | 44 | 172 | 152 |
| Soybean | 31 | 18 | 446 | 562 |
| Jatropha | 15 | 28 | 741 | 656 |
| Sunflower | 11 | 40 | 1070 | 946 |
| Canola | 12 | 41 | 1190 | 862 |
| Castor | 9 | 48 | 1307 | 1156 |
| Coconut | - | - | 2689 | - |
| Oil Palm | 2 | 36 | 5950 | 4747 |
| Microalgae[a] | 0.2 | 30 | 58700 | 51927 |
| Microalgae[b] | 0.1 | 50 | 97800 | 86515 |
| Microalgae[c] | 0.1 | 70 | 136900 | 121104 |
| -Botrycoccus braunill | | 25-75 | | |
| -Chlorella sp. | | 28-32 | | |
| -Crypthecodinium cohnii | | 20 | | |
| -Cylindrotheca sp. | | 16-37 | | |
| -Dunaliella primolecta | | 23 | | |
| -Isochrysis sp. | | 25-33 | | |
| -Monallanthus salina | | >20 | | |
| -Nannochloris sp. | | 20-35 | | |
| -Nannochloropsis sp. | | 31-68 | | |
| -Neochloris oleoabundans | | 35-54 | | |
| -Nitzschia sp. | | 45-47 | | |

[a]Microalgae produced high oil yield (30 % based on biomass weight)
[b]Microalgae produced high oil yield (50 % based on biomass weight)
[c]Microalgae produces low oil yield (70 % based on biomass weight)

Amish, P. V., Jaswant, L. V. and Subrahmanyam, N. 2010. A review on FAME production processes. *Fuel*, **89:**1-9.

# Transesterification of vegetable oil and microalgae oil using various catalysts

| Catalyst | Feedstock | Reaction temperature (°C) | Reaction time (min) | Conversion (c); yield (y) (%) | Literature |
|---|---|---|---|---|---|
| $KOH/Al_2O_3$ | Palm oil | - | - | 91.1 | Noiroj *et al.*, 2009 |
| CaO | *Jatropha curcas* oil | - | - | 93 (y) | Huaping *et al.*, 2006 |
| $K_2CO_3$/MgAl | *Jatropha curcas* oil | - | - | >96 (y) | Teng *et al.*, 2010 |
| $H_2SO_4$ | ***Chlorella prototheco**ides* | 30 | 240 | > 80 (y) | Xu *et al.*, 2006 |
| Candidia sp. 99-125 (Immobilized lipase) | *Chlorella prototheco**ides* | 38 | 720 | 98.2 (c) | Xiong *et al.*, 2008 |
| Zeolite (h-Beta) | *Nannochloropsis gaditana* | 45 | 115 | 25 (y) | Carrero *et al.*, 2010 |
| *Burkholderia* sp./alkyl-grafted $Fe_2O_3$-$SiO_2$ (Immobilized lipase) | *Chlorella vulgaris* | 40 | 2880 | 97.3 (c) | Tran *et al.*, 2012 |
| $CaO/Al_2O_3$ | *Nanochloropsis oculata* | 50 | 240 | 97.5 (y) | Umdu *et al.*, 2009 |
| $MgO/Al_2O_3$ | *Nanochloropsis oculata* | 50 | 240 | 50 (y) | Umdu *et al.*, 2009 |
| SrO | *Nannochloropsis* sp. | - | - | 37.1 (y) | Koberg *et al.*, 2011 |
| Mg-Zr | *Nannochloropsis* sp. | 65 | 240 | 22.2 (y) | Li *et al.*, 2011 |

# Objectives

- To synthesis bulk **calcium methoxide solid catalyst** with different synthesis reaction time.
- To study the **physico-chemicals properties** of catalysts.
- To test the **catalytic activity** of calcium methoxide soild catalyst on crude *Jatropha curcas* and microalgae oils (*Nannochloropsis oculata*).
- To investigate the effect of **variables** transesterification parameters on biodiesel production (reaction time, catalyst concentration in weight % and molar ratio of methanol to oil).

# Methodology

## (1) Microalgae propagation & extraction

Light  Water  $CO_2$  Nutrient

↓

**Cultivated of microalgae species**

↓

**Harvesting of microalgae**

↓

**Microalgae biomass processing**

**(dewatering, filtering, drying, sedimentation, centrifuging, grinding)**

↓

**Dry microalgae biomass**

↓

**Oil extraction**

**(cell disruption and oil being extract)**

↓

**Catalysis transesterification**

**(Biodiesel)**

# Methodology

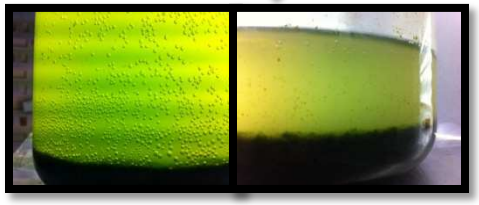
## Cultivation, Extraction & Catalytic test: crude microalgae derived oil (MDO)

Algae Seeds

1 week cultured

2 week cultured

Large-scale microalgae production



$FeCl_3$ flocculation

Dewatering

Dry microalgae

Solvent extraction

FAME

Methanol

+

Activated catalyst

Transesterification

Biodiesel

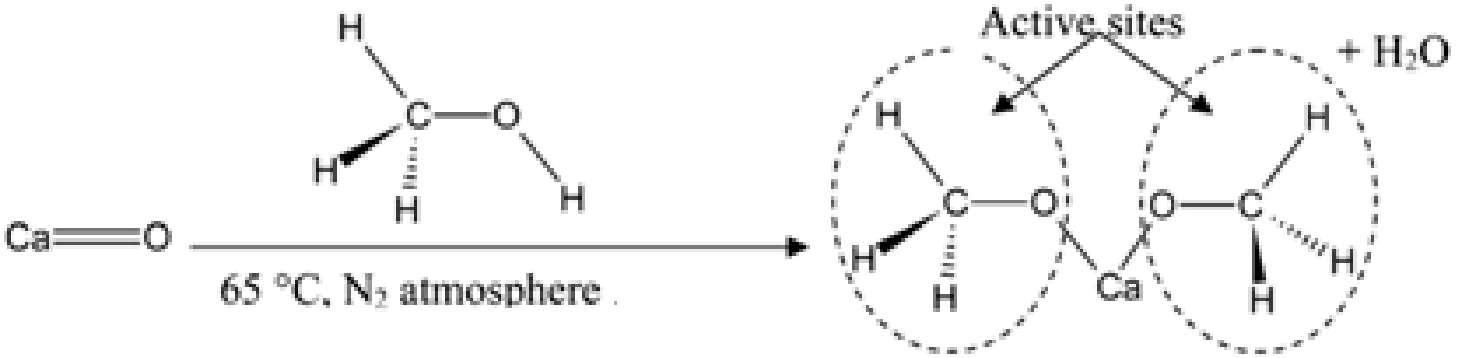
+

Glycerol

Gas chromatographic-mass spectrometer analysis

# Methodology

## (2) Synthesis of calcium methoxide catalyst




Reflux & stir at 65 °C for 2 – 12 hours

50 ml $min^{-1}$ $N_2$ flow

**Formation of white slurry solid base**

**Dry in the Oven at 100°C for 1h**

**CM2, CM4, CM6, CM8, CM10, CM12**

**Physico-chemical characteristics evaluation**

# Methodology

## (3a) Catalytic test (phase I): crude plant derived oil (PDO)

Jatropha oil

\+

Methanol

Activated catalyst

Transesterification/
FAME conversion

Biodiesel
+
Glycerol

Gas chromatographic analysis

## (3b) Catalytic test: crude microalgae derived oil (MDO)

Dry microalgae biomass

Solvent extraction

Crude MDO

Methanol

\+

Activated catalyst

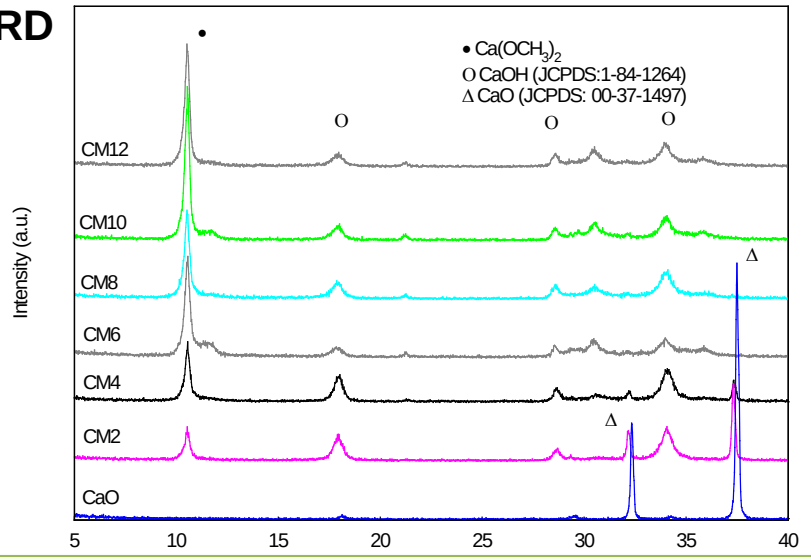
Transesterification

Biodiesel
+
Glycerol

Gas chromatographic analysis

# Physico-chemical properties of $Ca(OCH_3)_2$

**XRD**




| Catalyst | [a] Crystallite size (nm) CaO | [a] Crystallite size (nm) $Ca(OCH_3)_2$ | [b] $S_{BET}$ |
|---|---|---|---|
| Pure CaO | 66.3 | - | |
| CM2 | - | 32.0 | 16.2 |
| CM4 | - | 31.1 | 18.3 |
| CM6 | - | 30.6 | 17.5 |
| **CM8** | **-** | **27.5** | **30.5** |
| CM10 | - | 29.5 | 28.8 |
| CM12 | - | 31.4 | 26.0 |

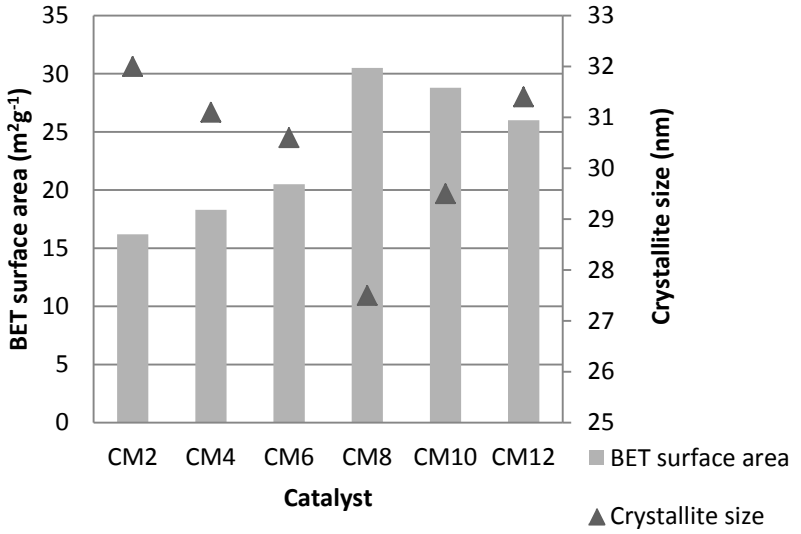
[a]Determinded using Sherrer's equation from the XRD pattern.
[b]Dertermined using BET surface area.

Obvious broad peaks of **cubic CaO** (32.1°, 37.2° ) –pure calcium oxide.

Calcium oxide was transformed into **calcium methoxide** as evident from the characteristic peak at $2\theta$ value of 10.8° after (2-12 h) react with methanol under reflux.

$$CaO + 2CH_3OH \xrightarrow{65\ ^oC} Ca(OCH_3)_2 + H_2O$$

calcium oxide — calcium methoxide

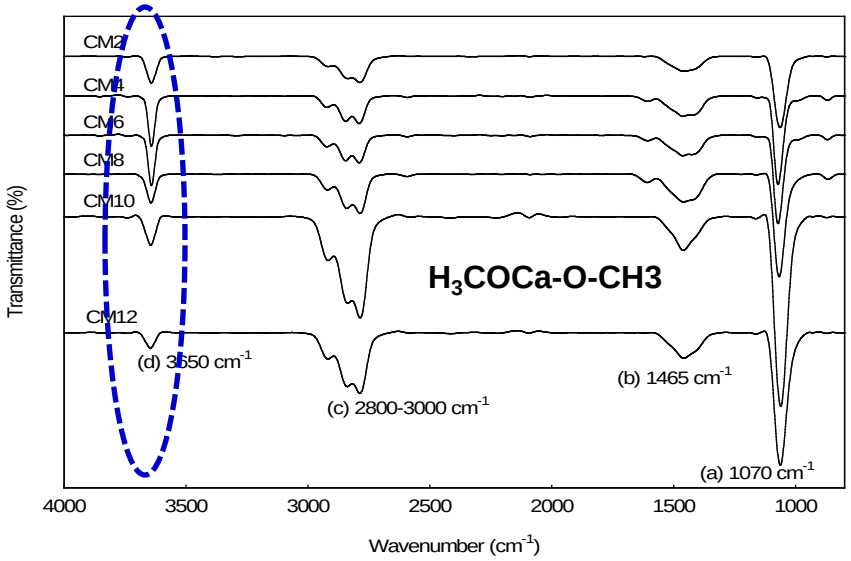



No indication regarding the presence of unreacted CaO in the final product (CM6-12).

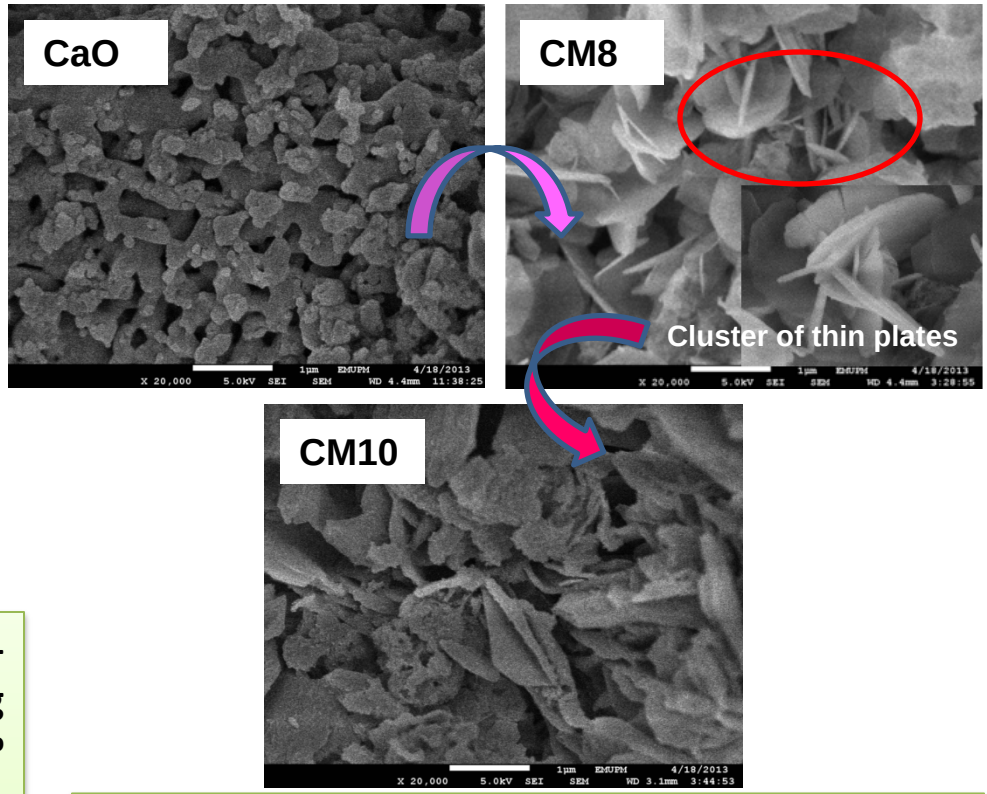
M. Hassan, Y. Robiah, S. Y. Thomas Chong, R. Umer and Y. H. Taufiq Yap, Synthesis and cahracterization of calcium methoxide as heterogeneous catalyst for trimethylolpropane esters conversion reaction. (2012) *Applied Catalysis A: General,* 425-426: 184-190.

# Physico-chemical properties of $Ca(OCH_3)_2$

## FTIR




## SEM




**(a) -C-O stretching vibration of 1º alcohol; (b) -C-H (alkane) bending; (c) -$CH_3$ streching vibration; (d) -OH streching vibration of 1º alcohol.**

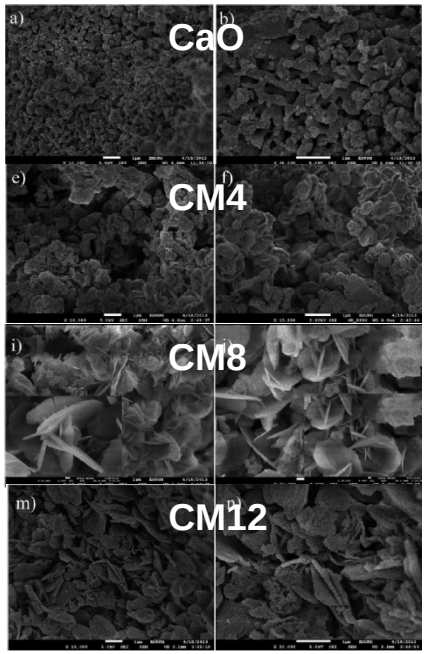
**Water was produced as a by product in the catalyst synthesis reaction – these isolated –OH groups might have produced from water to facilitate the strong basic property of $Ca(OMe)_2$.**

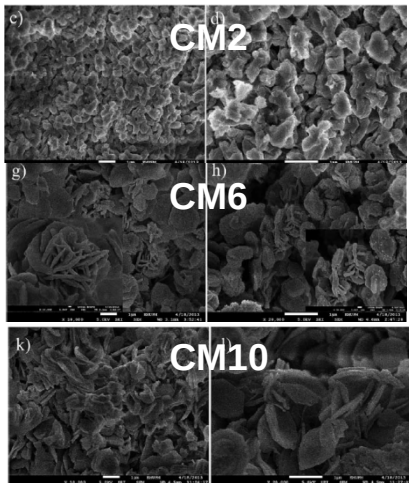
The image of CaO catalyst showed the mophology of CaO with irregular shape.

The surface of $Ca(OMe)_2$ appears to be formed the **cluster of thin plates** which are visible on the surface of catalyst.

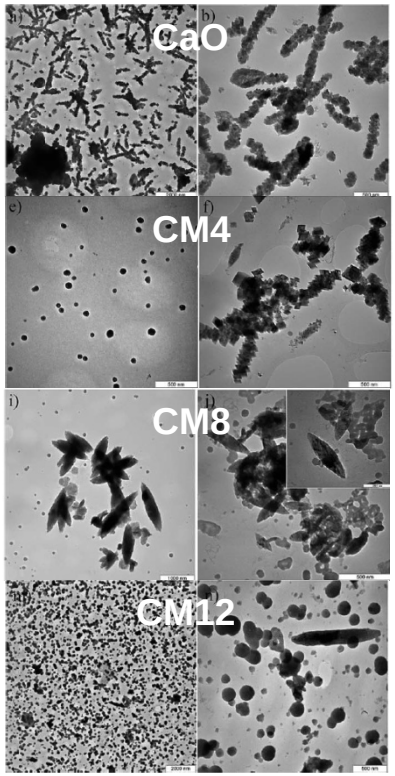
The particle start to **agglomerate** with prolong the synthesis duration.

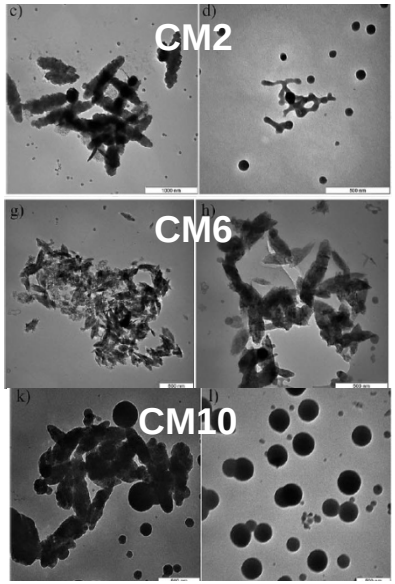
# SEM & TEM




SEM




TEM

| Catalyst | 2$\theta$ (°) | FWHM | Crystallite size[a] (nm) | $S_{BET}$[b] ($m^2$ $g^{-1}$) | Shape[c] | Particle size[d] (nm) |
|---|---|---|---|---|---|---|
| CaO | 37.4701 | 0.1510 | 66.3 | 9.2 | Cubic crystal | 137.02 ± 11.30 |
| CM2 | 10.8448 | 0.2784 | 29.0 | 16.2 | Cubic crystal & irregular bulky round | 170.71 ± 25.26[e] & 68.06 ± 19.29 |
| CM4 | 10.8348 | 0.2597 | 31.1 | 18.3 | Cubic crystal & irregular bulky round | 120.07 ± 23.32[e] & 34.74 ± 3.26 |
| CM6 | 10.8248 | 0.3074 | 30.6 | 20.5 | Plate | 267.10 ± 35.10 (D) 72.46 ± 22.34 (T) |
| **CM8** | **10.8105** | **0.2941** | **27.5** | **30.5** | **Plate** | **391.77 ± 65.34 (D) 111.38 ± 13.12 (T)** |
| CM10 | 10.8213 | 0.2721 | 29.7 | 28.8 | Plate & round | 530.41 ± 21.69 (D) 157.38 ± 5.69 (T) & 302.30 ± 46.93 |
| CM12 | 10.8223 | 0.2847 | 31.4 | 26.0 | Irregular bulky round | 366.52 ± 24.82 |

[a]Determined from XRD patterns using the Scherrer equation. [b]BET surface area. [c]Observed by SEM analysis. [d]Measured by TEM technique. [e]Unreacted CaO. D: diameter and T: thickness.

# Fatty acid composition of *N. oculata*

| Subtituent | Name of the fatty acid | Structure (*xx:y*) | *Lit | Composition % | | | |
|---|---|---|---|---|---|---|---|
| | | | | Hexane | $CHC_l$ 3 | $CHCl_3$: MeOH (1:2 v/v) | MeOH |
| Saturated lipid | Lauric acid | 12:0 | | - | - | 0.21 | 0.34 |
| | Myristic acid | 14:0 | 4.9 | 0.42 | 3.08 | 3.90 | 6.73 |
| | Pentadecanoic acid | 15:0 | | - | - | 0.26 | 0.57 |
| | Palmitic acid | 16:0 | 25.2 | 3.65 | 15.49 | 27.45 | 24.67 |
| | Margaric acid | 17:0 | | - | - | 0.25 | 0.67 |
| | Stearic acid | 18:0 | 1.1 | - | - | 0.68 | 0.69 |
| | Arachidic acid | 20:0 | | - | - | - | 0.13 |
| Monosaturated lipid | Palmitoleic acid | 16:1 | 29.3 | 1.96 | 9.16 | 20.10 | 22.43 |
| | Oleic acid | 18:1 | 13.6 | 0.73 | 2.60 | 5.20 | 7.68 |
| Polysaturated lipid | Hexadecadienoic acid | 16:2 | | - | - | - | 0.42 |
| | Hexadecatrienoic acid | 16:3 | | - | - | - | 0.16 |
| | Linoleic acid | 18:2 | 1.1 | 0.32 | 2.33 | 4.60 | 5.53 |
| | Linolenic acid | 18:3 | | - | - | - | 0.60 |
| | Eicosadienoic acid | 20:2 | | - | - | - | 0.24 |
| | Eicosatrienoic acid | 20:3 | | 0.36 | - | - | 0.12 |
| | Arachidocic acid (AA) | 20:4 | | - | 1.77 | 2.52 | 3.18 |
| | 3,7,11,15 methylhexadecanoic acid | 20:4 | | - | - | 0.50 | - |
| | Timnodonic acid | 20:5 | 22.6 | 0.97 | 12.66 | 16.98 | 14.63 |
| Others | | | 1.2 | 91.59 | 52.91 | 17.35 | 11.13 |
| [a]Unsaturated/ saturated ratio | | | | 1.07 | 1.54 | 1.52 | 1.63 |

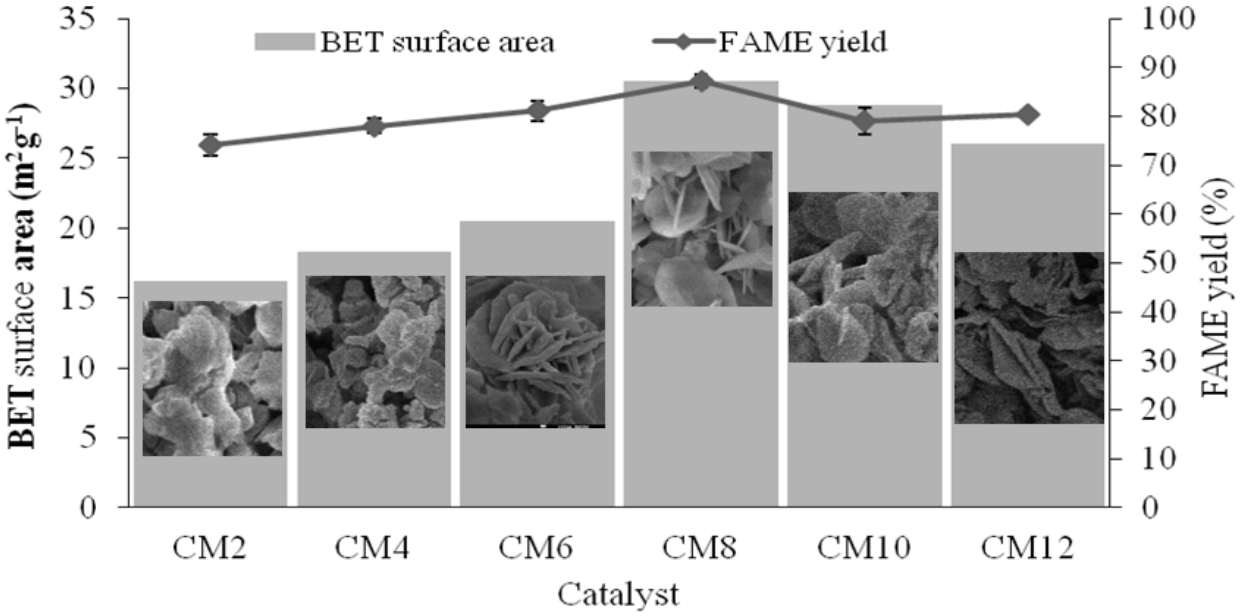
[a]Percentages may not add to 100 % due to other constuitents not included.

* Lit. Li. Y. S. *et al.*, 2011

# Catalytic activity

**Methanolysis reaction on crude *Jatropha curcas* oil: correlation between biodiesel yield and synthesis time, surface area and morphology of catalyst**

| MeOH molar ratio | FAME yield (%) |
|---|---|
| CM2 | 74 |
| CM4 | 78 |
| CM6 | 81 |
| CM8 | **87** |
| CM10 | 79 |
| CM12 | 80 |




Influence of **different synthesis times** on transesterification of crude *Jatropha curcas* oil*:* catalyst dosage 2 %, reaction time 2 h, reaction temperature 65 °C.

It is noteworthy that the catalytic activity of synthesized $Ca(OCH_3)_2$ catalysts showed high transesterification activity in the range of 74–87%.

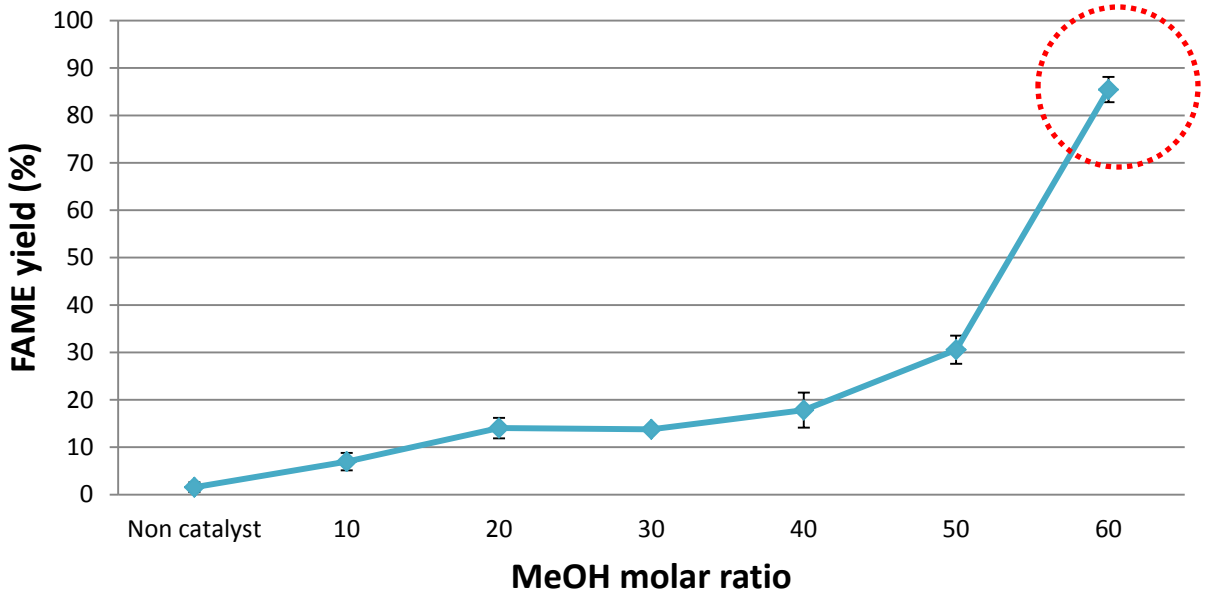
The FAME yield was increased from CM2 to CM8, while the further increment of synthesis time from CM10 to CM12 resulted in small decreases in catalytic performance.

$Ca(OCH_3)_2$

# Catalytic activity

Influence of **methanol ratio** on transesterification of extracted *N. oculata* lipid from dried microalgae powder*:* catalyst dosage 3 %, reaction time 3 h, reaction temperature 60 $^{o}$C.

| MeOH molar ratio | FAME yield (%) |
|---|---|
| Non catalyst | 1.57 |
| 10 | 6.95 |
| 20 | 14.04 |
| 30 | 13.79 |
| 40 | 17.83 |
| 50 | 30.57 |
| 60 | 85.44 |




The figure show the transesterification of lipid with various amount of MeOH ratio (10 – 60 %) catalyzed by $Ca(OMe)_2$ catalyst.

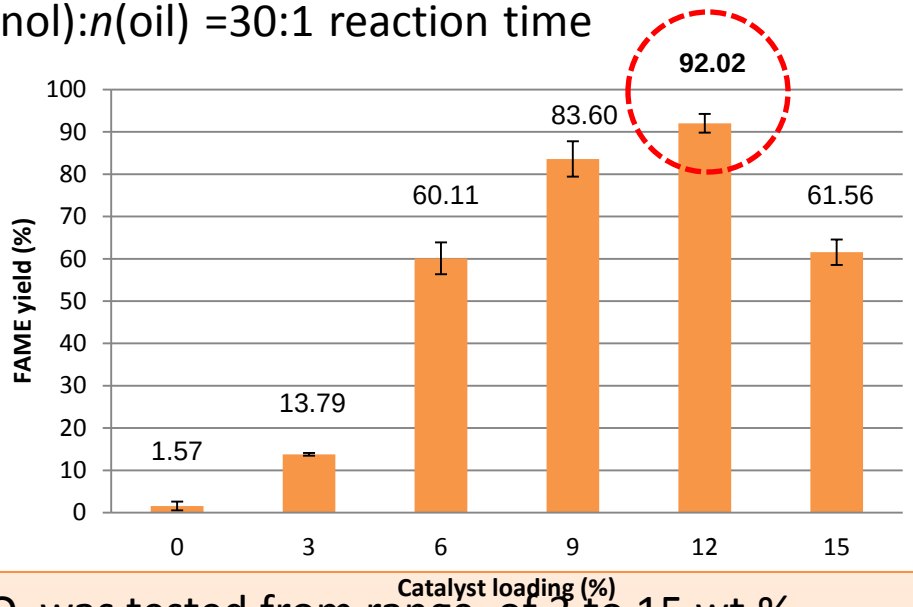
The yield of methyl ester increased with increasing molar ratio MeOH to lipid and reached maximum of 85 **%** at the MeOH ratio of 60 : 1.

**The increasing the amount of MeOH was required to accelerate the formation of the product due to this reaction was reversible.**

# Catalytic activity

Influence of **catalyst dosage** on transesterification of extracted *N. oculata* lipid from dried microalgae powder *:* (*n*(methanol):*n*(oil) =30:1 reaction time 3 h, reaction temperature 60 $^{o}$C.

| Catalyst loading (wt.%) | FAME yield (%) |
|---|---|
| Non catalyst | 1.57 |
| 3 | 13.79 |
| 5 | 60.11 |
| 7 | 83.60 |
| 10 | 92.02 |
| 15 | 61.56 |




The effect of catalyst concentration on the MDO was tested from range of 3 to 15 wt.%.

**The yield of methyl ester increased with increasing amount of catalyst and reached a higher value of 92 % (10 wt.% catalyst). The yield of FAME decreased when the catalyst amount increased to 15 wt.%.**

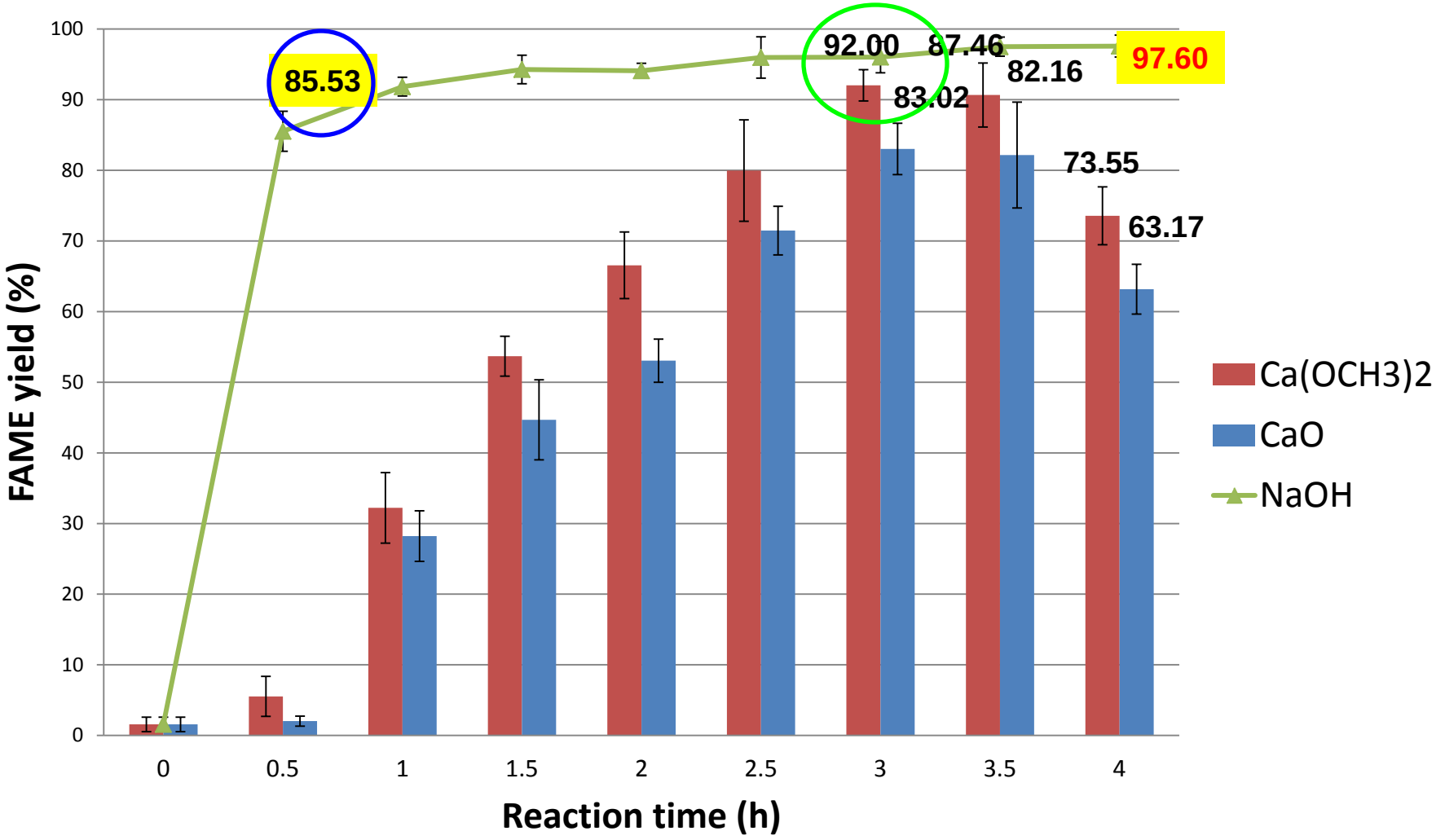
Reason:

(i) The high concentration of **$RO^-$ species** formed from the dissociation of methanol on the basic site and the catalyst as well.

(ii) Too high amount of catalyst in the system cause **a multiphase mash formed** will increase the resistance to stirring and decrease the dispersion of catalyst, led to the low yield of methyl ester.

# Catalytic activity

Influence of **reaction time** on transesterification of extracted *N. oculata* lipid by **$Ca(OCH_3)_2$** (a)**, CaO** (b) **and** (c) **NaOH** catalyst : catalyst dosage = 12 wt. % (a & b) and 1 mol (c), *n*(methanol):*n*(oil) = 30:1, reaction temperature = 60 °C




**$Ca(OCH_3)_2$ (CM8)**

The comparison of FAME yield using two solid catalysts ($Ca(OCH_3)_2$ and CaO) and one homogeneous catalyst (NaOH).

**The NaOH catalyst reacts significantly faster compared to the heterogeneous catalysts (completely converted to FAME within 20-30 min).**

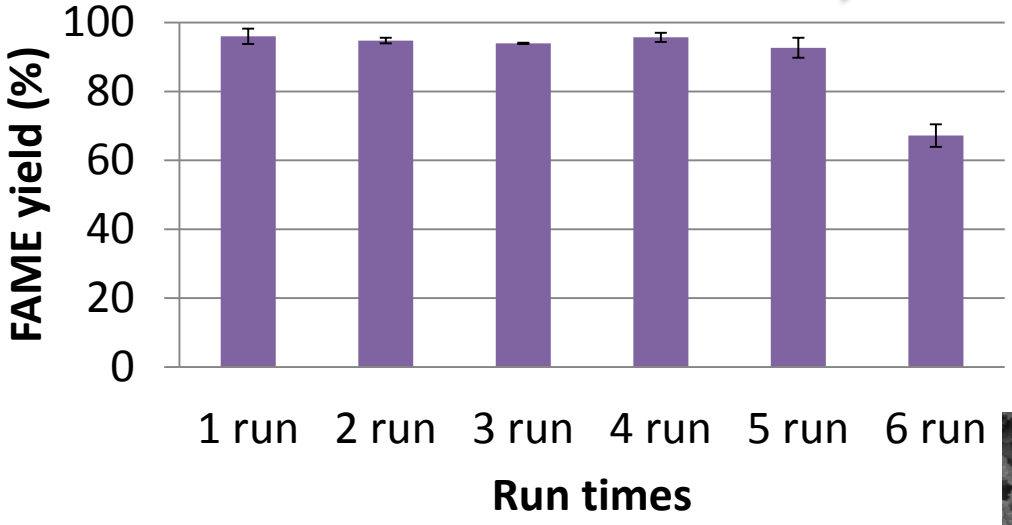
**For the heterogeneous catalyst system, the results revealed that the reaction rate is very slow and maximum FAME yield of 80 % is achieved after 3 h which is due to the heterogeneous mass transfer systems between the solid catalyst and the liquid reactant.**

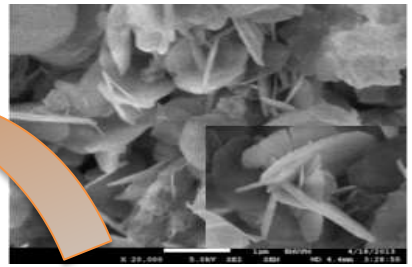
The catalyst showed higher conversion rate and achieved 92 % FAME yield compared to 80 % of CaO over the same duration.

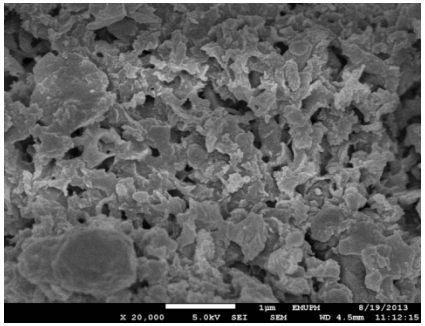
Drawback of NaOH:
(i) Residual catalyst was found in the FAME. An extra procedure was conducted, *i.e.* water washing.
(ii) When water droplets were added to the FAME layer, the unreacted TG and other intermediates were hydrolyzed and saponified.

# Reusability

FAME yield **> 90 %**



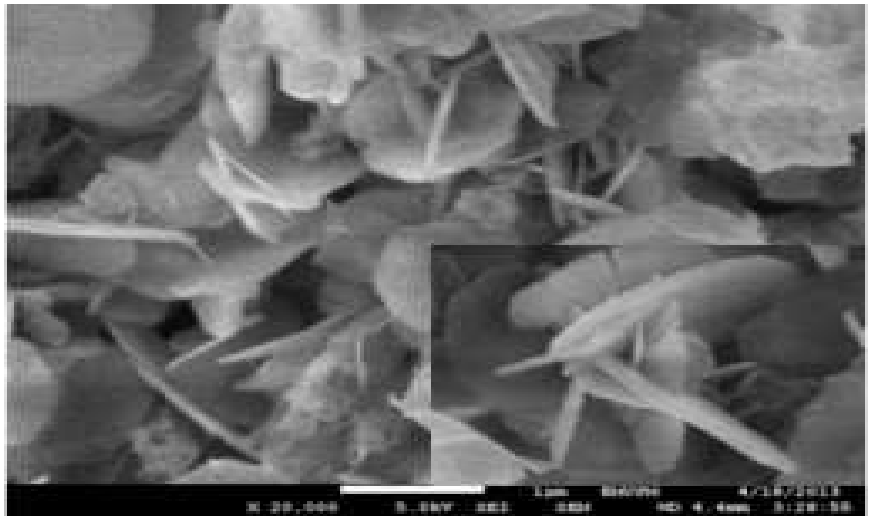





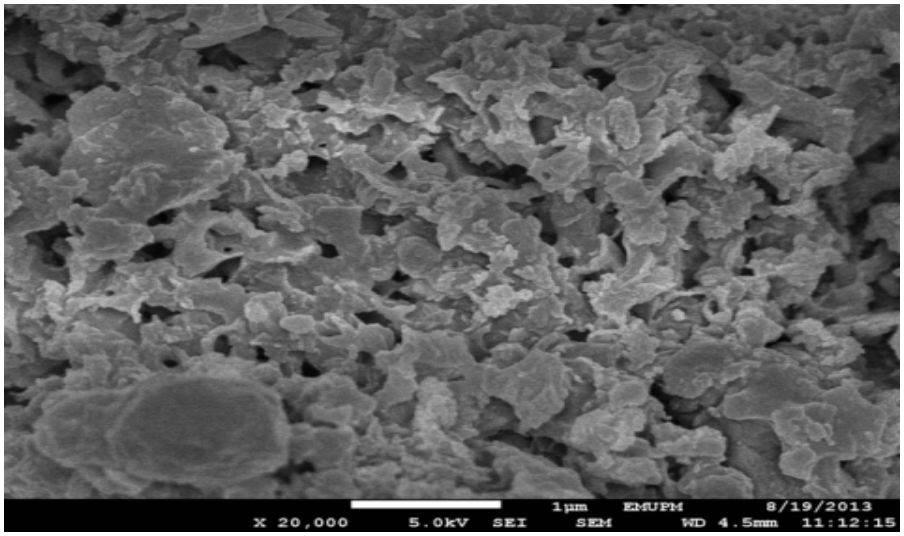
The results showed that the catalyst can be **reused up to five times**.

The deactivation of the catalyst is due to the impurities that come from microalgae lipid, *i.e.* phospholipids and moisture, which changed the active phase and morphology of catalyst as shown.

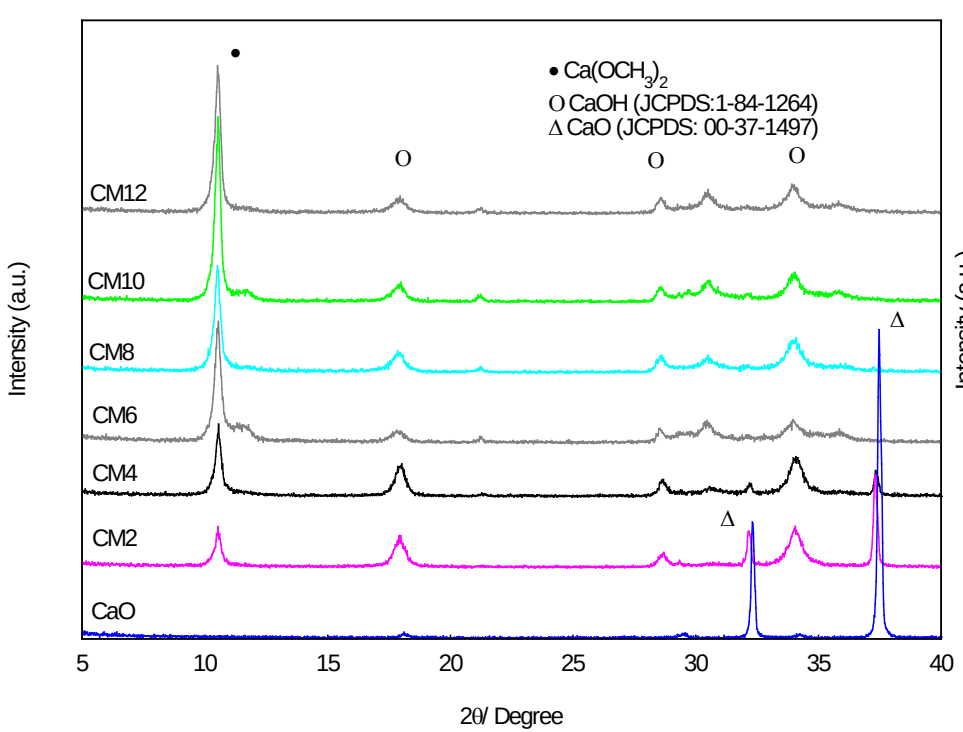
## Fresh

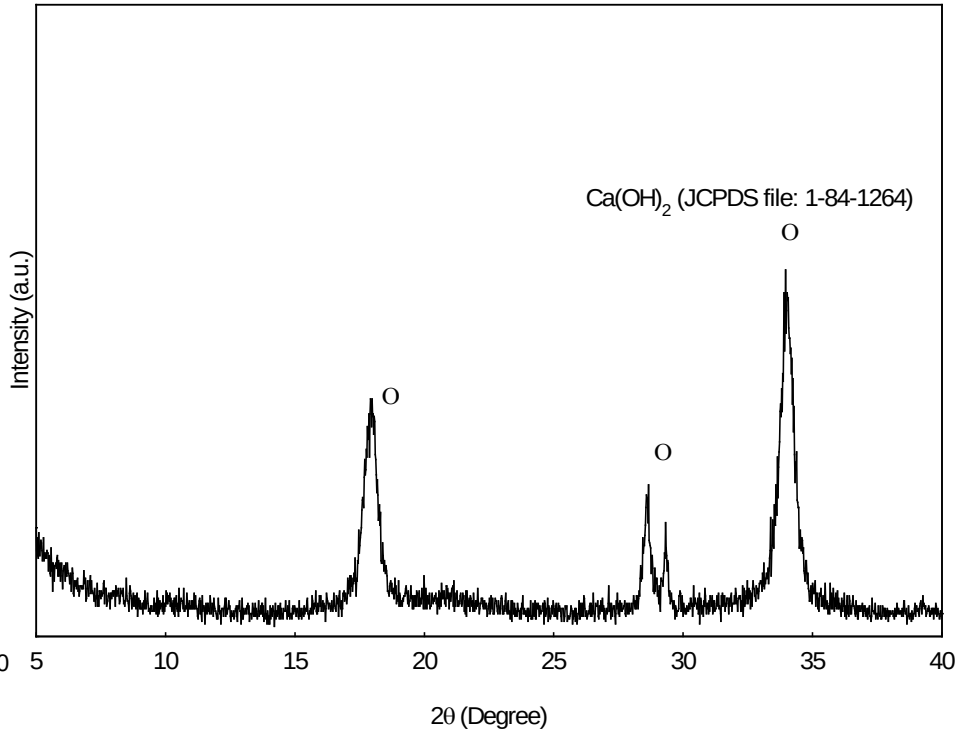


## Used



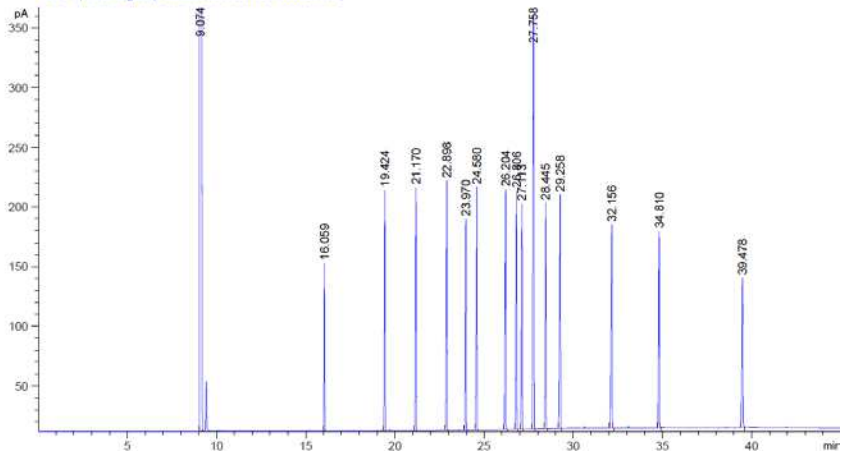

# XRD patterns and SEM micrographs of fresh and deactivated $Ca(OCH_3)_2$ catalyst



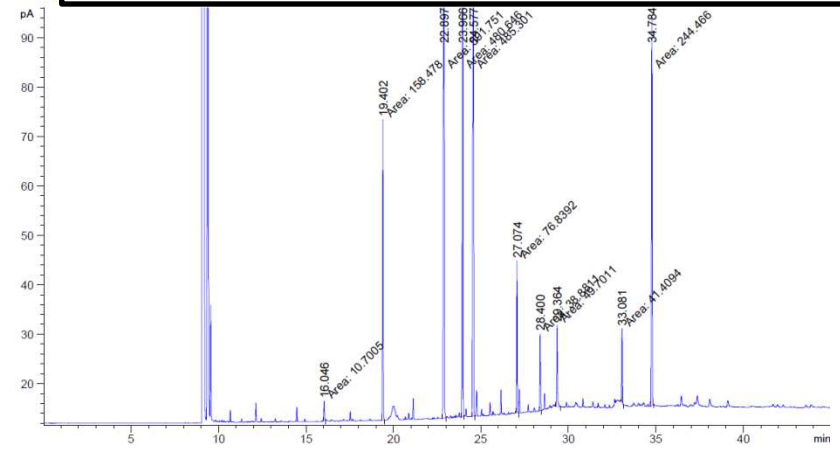



# GC chromatogram

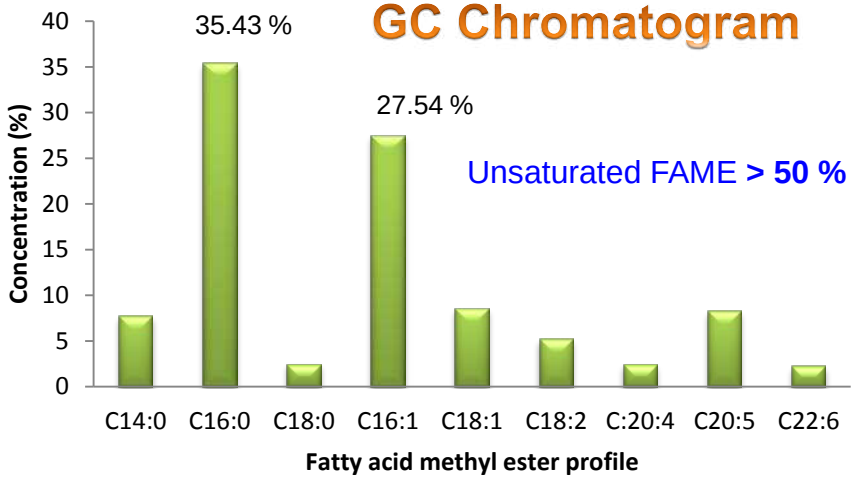
Reference standards

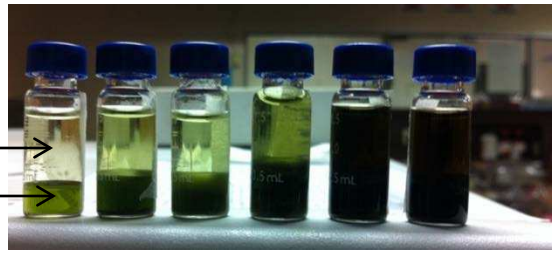



Fatty acid methyl esters derived from microalgae lipids




| Methyl ester | Carbon |
|---|---|
| Methyl laurate | C 12:0 |
| Methyl myristate | C 14:1 |
| **Methyl palmitate** | C 16:0 |
| Methyl palmitoleate | C 16:1 |
| Methyl stearate | C 18:0 |
| Methyl oleate | C 18:1 |
| Methyl linoleate | C 18:2 |
| Methyl arachidate | C 20:0 |
| Methyl docosanoate | C 22:0 |
| All cis 5,8,11,14,17-methyl eicosapentenoate | C 20:5(n3) |



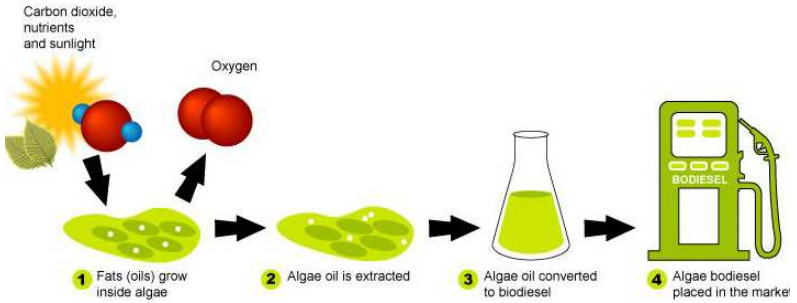



# Summary

- Solid base calcium methoxide catalyst **successfully** synthesized for methanolysis of crude *Jatropha curcas* and crude microalgae oils to produce biodiesel.

- The bulk calcium methoxide (CM8) showed **superior effect** & **fast conversion** in catalytic activity due to the **high surface area tend to promote highly basic property**.

- At optimization condition, CM8 show **high methyl ester production** (92%) from microalgae derived oil at the condition of (*n*(methanol):*n*(lipid) =30:1, 12 % of catalyst , reaction time of 3 h at 60 $^{o}$C.



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- Dr. Teo Siow Hwa
- Dr. Sivasangar Seenivasagam

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11. Dr. Teo Siow Hwa
12. Dr. Theam Kok Leong
13. Dr. Mohd. Hasbi Abdul Rahim (*UM Pahang*)
14. Mr. Saw Chaing Sen (*INTEL Malaysia*)
15. Mr. Ahmad Raslan Mat Hussin (*Bernas*)
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28. Rabiah Nizah
29. Lai Fook How


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4th
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Toward New Developments
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Venue:Hitotsubashi Hall,
Hitotsubashi University(Tokyo)
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# THANK YOU

# TERIMA KASIH

ありがとうございました

감사

شكرا لكم

謝謝/谢谢


Thank
you!