

Real Time and *In Situ* Monitoring of the Crystallization of Palm Oil-based Products using Focused Beam Reflectance Measurement (FBRM)

Elina Hishamuddin

Malaysian Palm Oil Board, Malaysia

MPOB

OVERVIEW

- **Introduction**
  - Palm oil
  - Palm oil fractionation
  - Focused beam reflectance measurement (FBRM)
- **Materials and Methods**
  - Crystallization experiments
  - Compositional analyses
- **Results and Discussion**
  - Palm Oil Crystallization - Crystals population
  - Palm Oil Melting – Melting mechanisms
  - Palm Olein Crystallization - Crystal size distribution & Crystallisation mechanisms
  - Palm Olein Blend with Legume Seed Oil – effect of blending on crystallization mechanism
- **Conclusions**

PALM OIL

- **Palm oil** – largest produced vegetable oil in the world, **>59.5 million tonnes** in 2014. (Source: Oil World Annual 2014)





- Palm oil mainly consists of a multicomponent mixture of:
  - Triacylglycerols (TAGs): 94%
  - Diacylglycerols, monoacylglycerols and free fatty acids: 6%.

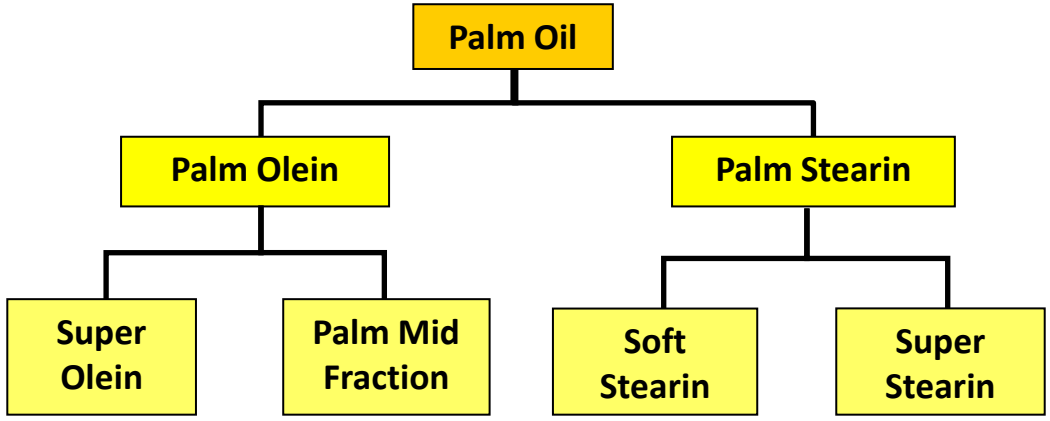
- Serves as feedstock for a variety of industries, i.e. food, oleochemicals, biofuels etc.

- Palm-based food products range from cooking oils, salad oils to margarines and shortenings and confectionery fats .



PALM OIL FRACTIONATION

- **Dry fractionation** : controlled **partial crystallisation** of oil from the melt and **filtration** of crystallized slurry
- **Crystallisation** : driven by differences in the melting points of the TAGs & operating temperature
- Quality of subproducts depend on the operating conditions during the crystallization stage and crystal characteristics such as size, numbers and shape have a profound effect on the filtration stage.



MOTIVATION

- Most crystallization studies are performed on static systems – Differential Scanning Calorimetry (DSC), polarized light microscopy (PLM), X-ray diffraction (XRD) etc.

- Static systems do not accurately reflect the actual industrial process. Dynamic systems are less studied due to fewer available monitoring devices.

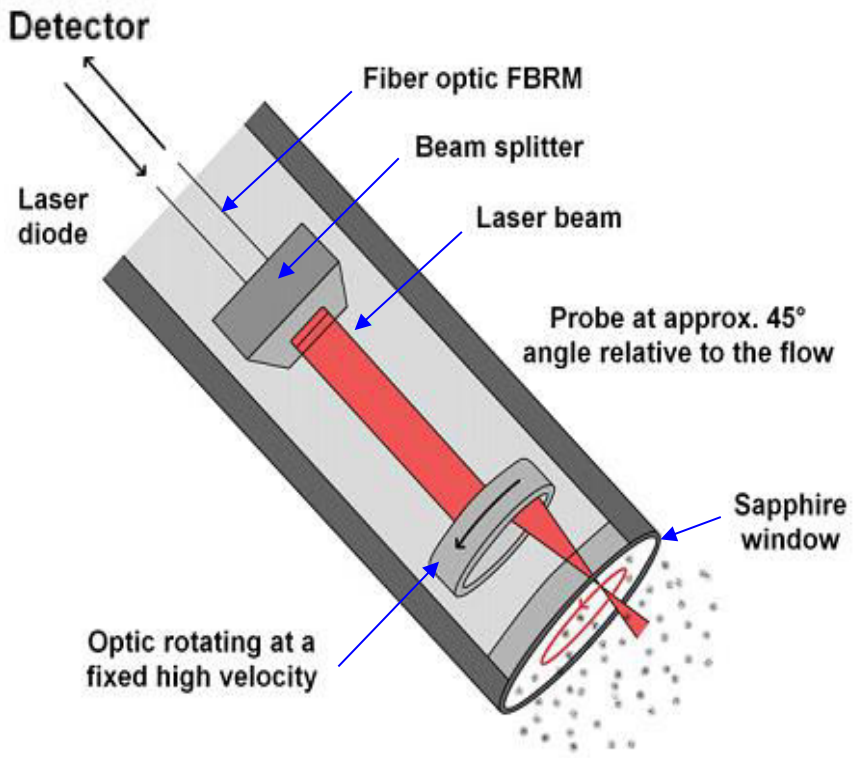
- Particle characteristic studies – image analysis, laser particle counters, particle size analyzers.

- Most require sampling the material and in some cases dilution -> changes material in study

- **Focused beam reflectance measurement (FBRM)** – non-destructive technique capable of monitoring particle characteristics on-line in real time.

FOCUSED BEAM REFLECTANCE MEASUREMENT (FBRM)

- *In-situ* measuring device
- Real time tracking of particle size, population and events.
- No sampling or dilution required
- Particle size range : 0.8 – 1000 µm
- Particle size in chord length
- Measurement depends on crystal orientation



Source : Mettler Toledo

CRYSTALLIZATION METHODS

- Material : 700 mL oil
- 1 L Mettler Toledo LabMax Automatic Lab Reactor with LASENTEC FBRM Model D600L
- Agitation speed: 50 rpm
- Isothermal Crystallization time:
  - Palm oil : 2 hours
  - Palm olein : 6 hours
  - Blended oil : 5 hours
- Morphological analysis by polarised light microscope



COMPOSITIONAL ANALYSES

Fatty acids composition

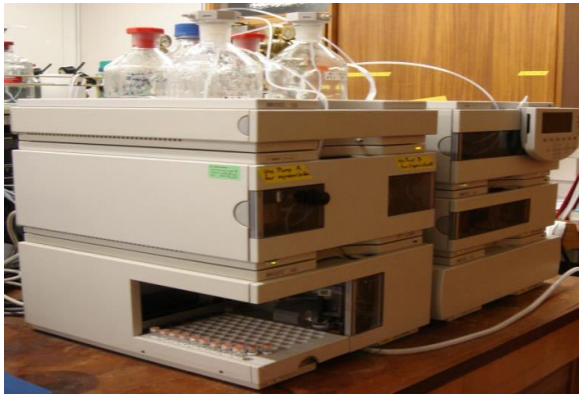
- ISO 5508:1990
- GC-FID Conditions
  - Column : DB-23, 60m L x 0.25 mm i.d., film thickness 0.25 µm
  - Oven T : 185 °C
  - Injector & detector T : 240 °C
  - Carrier gas : He @ 0.8 mL/min
  - Split ratio 1:100

Triacylglycerols composition

- AOCS Method Ce 5c-89
- HPLC-RID Conditions
  - Column : Purospher RP-18, 4.6 mm i.d. x 250 mm L, 5 µm particles, 2 connected in series
  - Column & detector T : 35 ° C
  - Acetone:Acetonitrile = 63.5:36.5
  - Flowrate : 1 mL/min

GC-FID

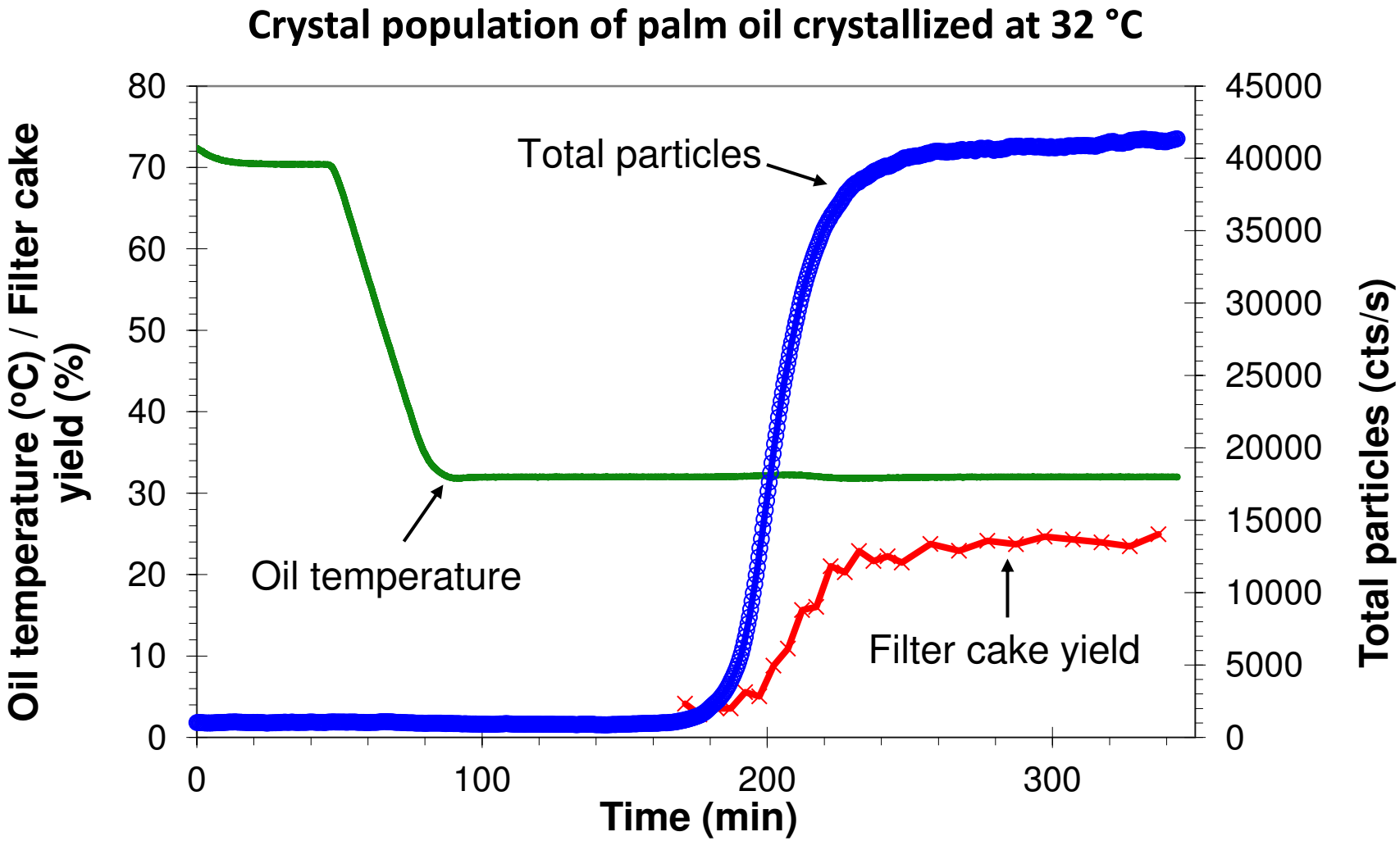




HPLC-RID

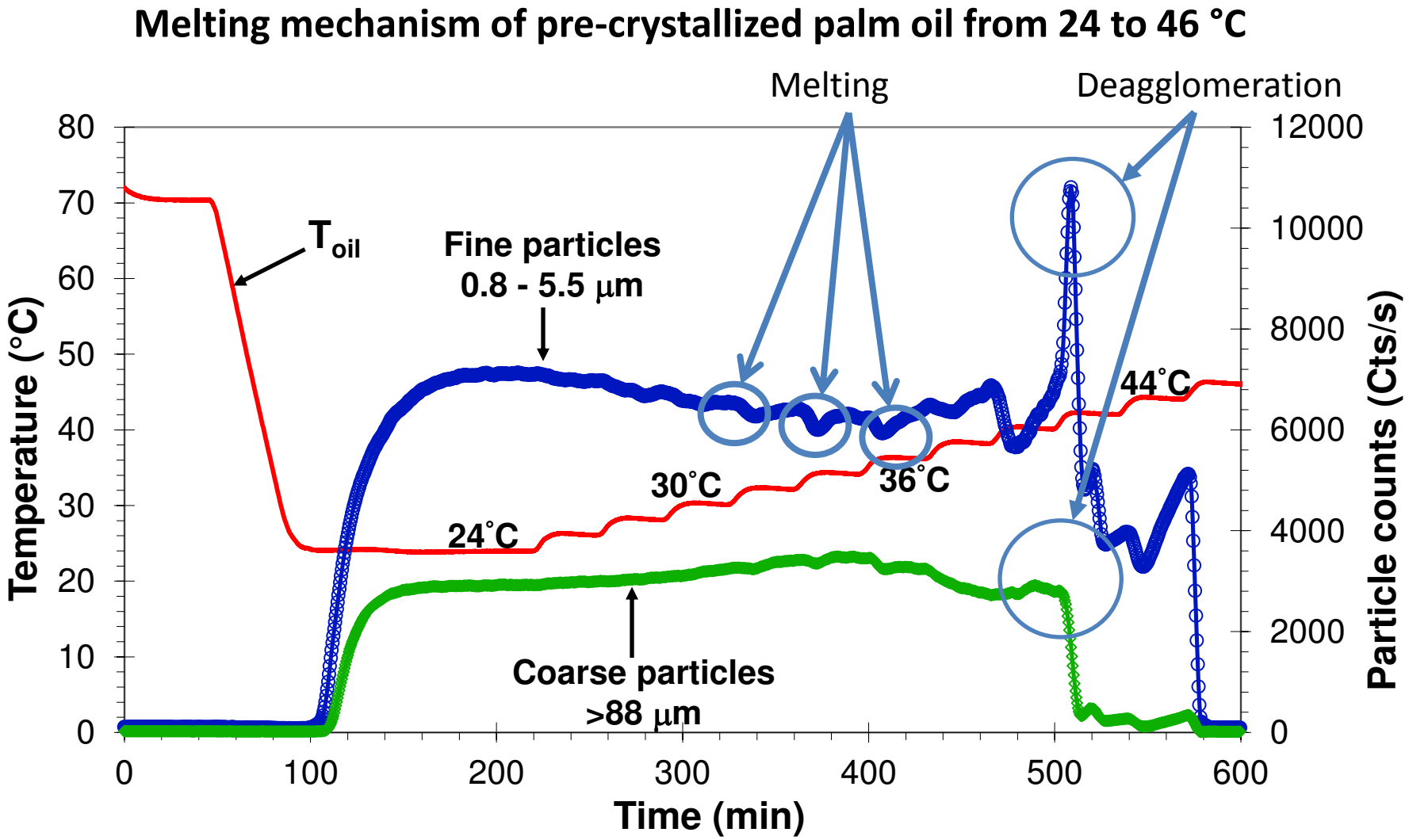
PALM OIL CRYSTALLIZATION

PALM OIL CRYSTALLIZATION



Hishamuddin *et al.* (2011), J. Cryst. Growth, 335, 172-180

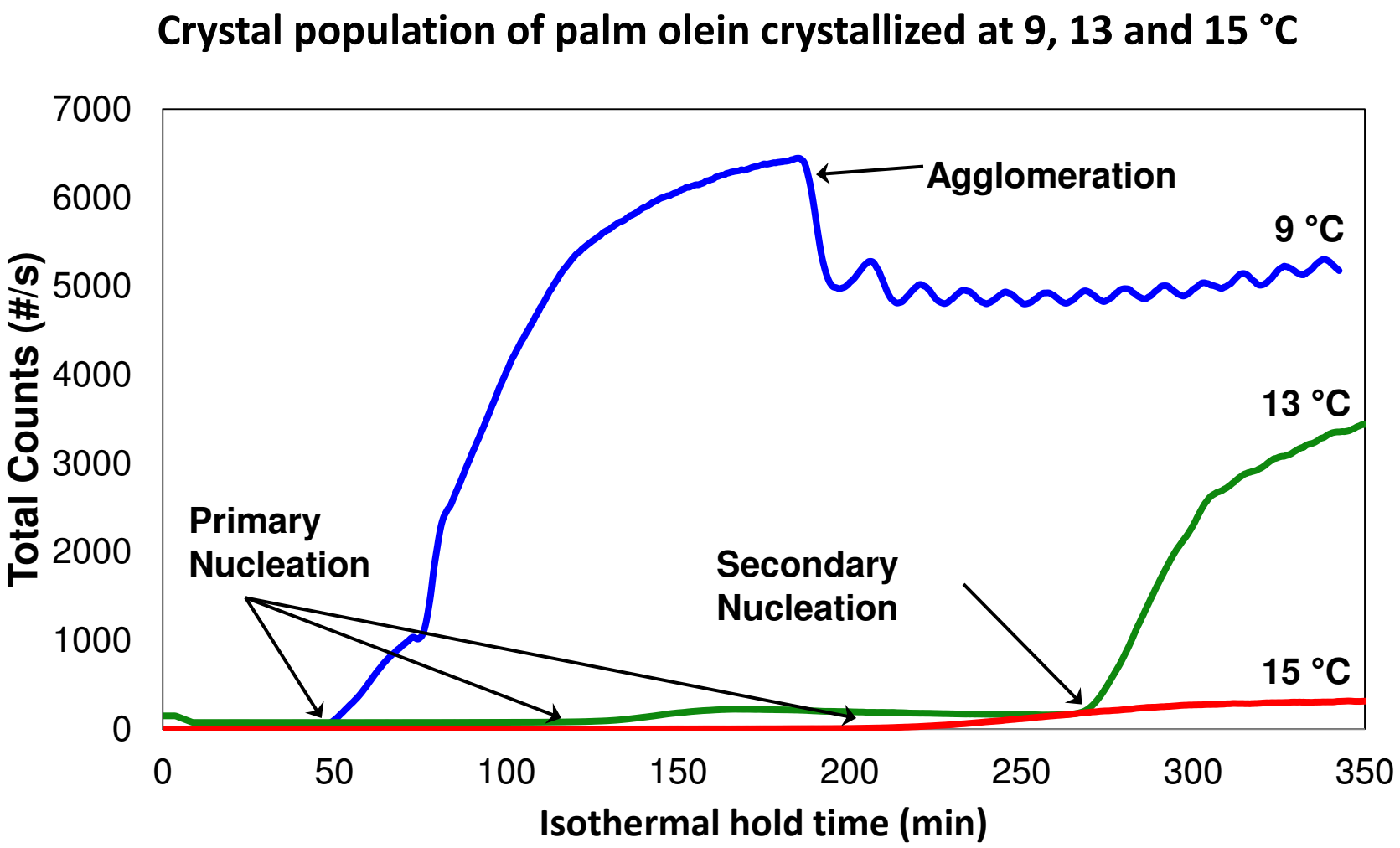
PALM OIL MELTING



Hishamuddin *et al.* (2011), J. Cryst. Growth, 335, 172-180

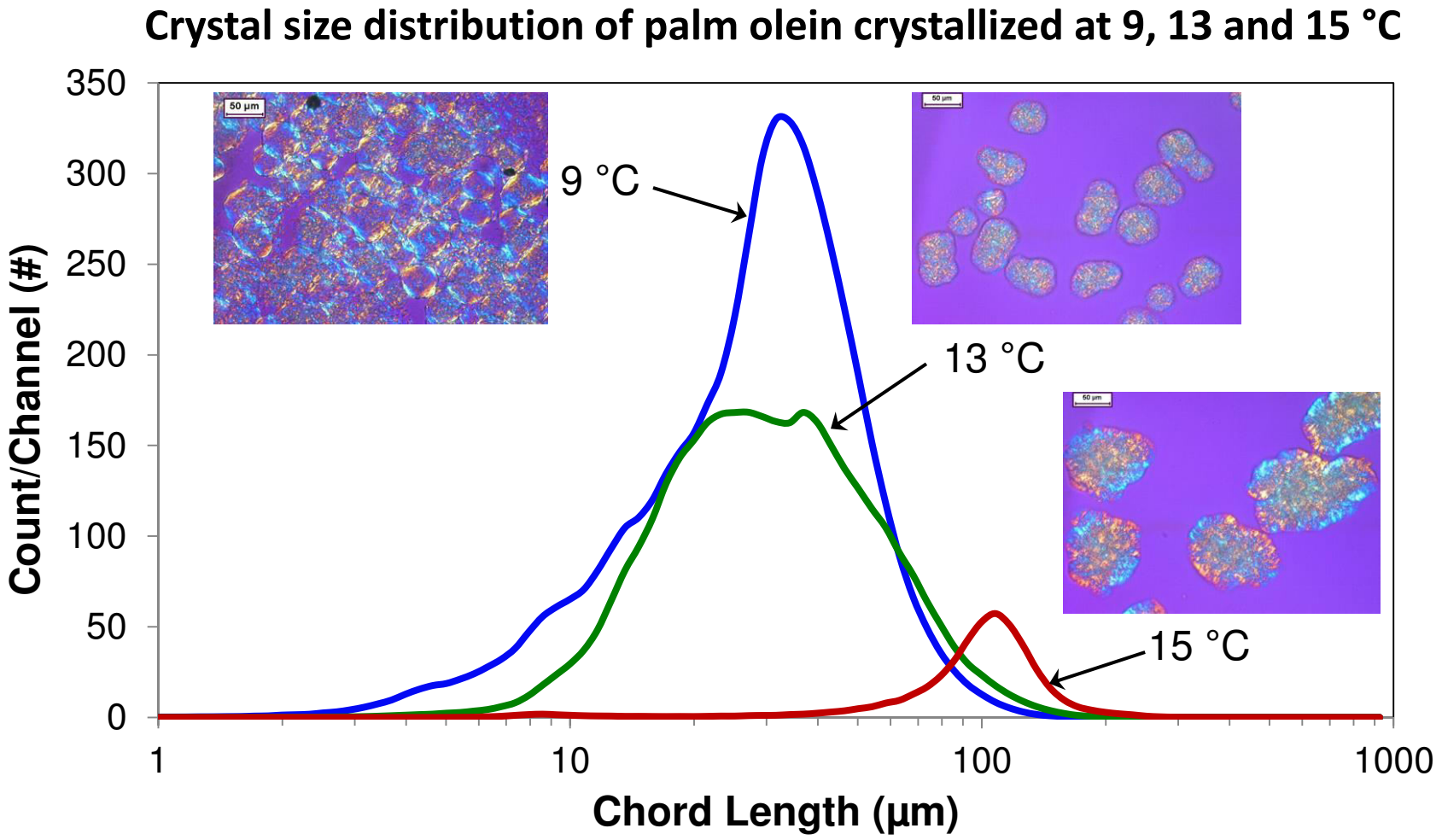
PALM OLEIN CRYSTALLIZATION

PALM OLEIN CRYSTALLIZATION



Hishamuddin *et al.* (2011*), Eur. J. Lipid Sci.,* 113(S1): 1-46

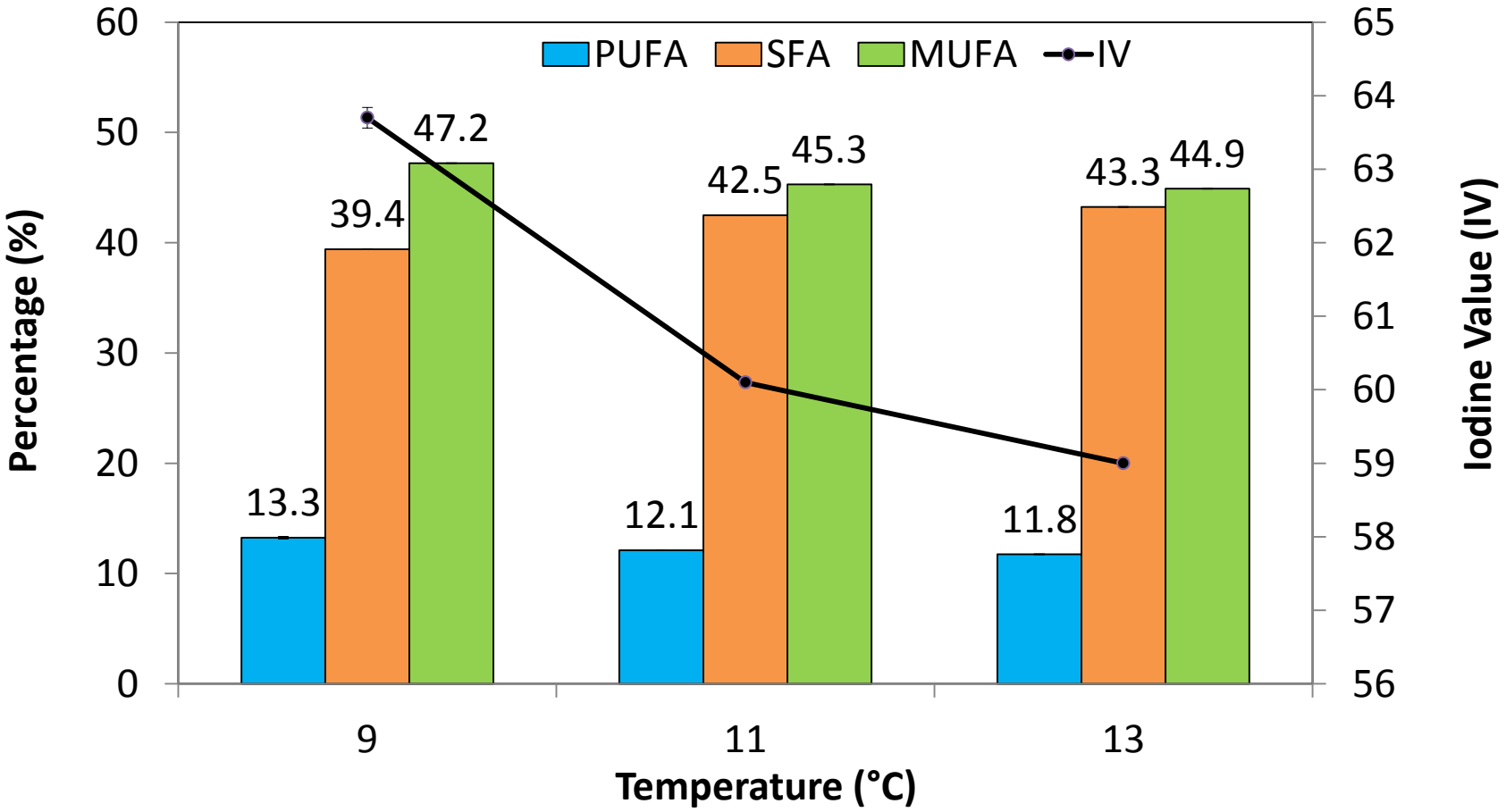
PALM OLEIN CRYSTALLIZATION



Hishamuddin (2014). *Palm Oil Dev.,* 61, 12-13 & 21-25.

PALM OLEIN CRYSTALLIZATION

Fatty acid composition of palm olein crystallized at 9, 13 and 15 °C



IV : Iodine value

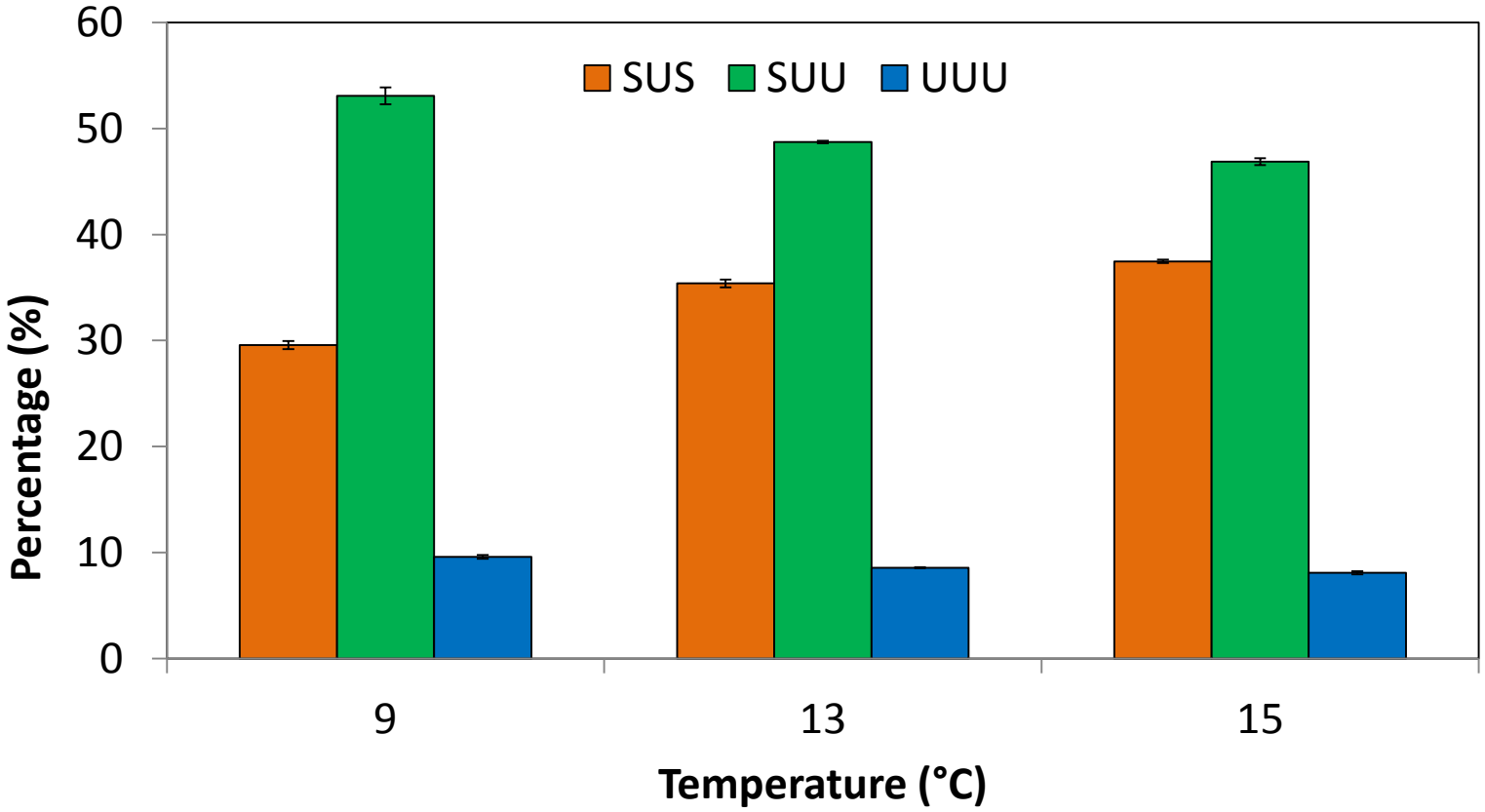
PUFA : polyunsaturated fatty acids

SFA : saturated fatty acids

MUFA : monounsaturated fatty acids

PALM OLEIN CRYSTALLIZATION

Major triacylglycerol groups of palm olein crystallized at 9, 13 and 15 °C

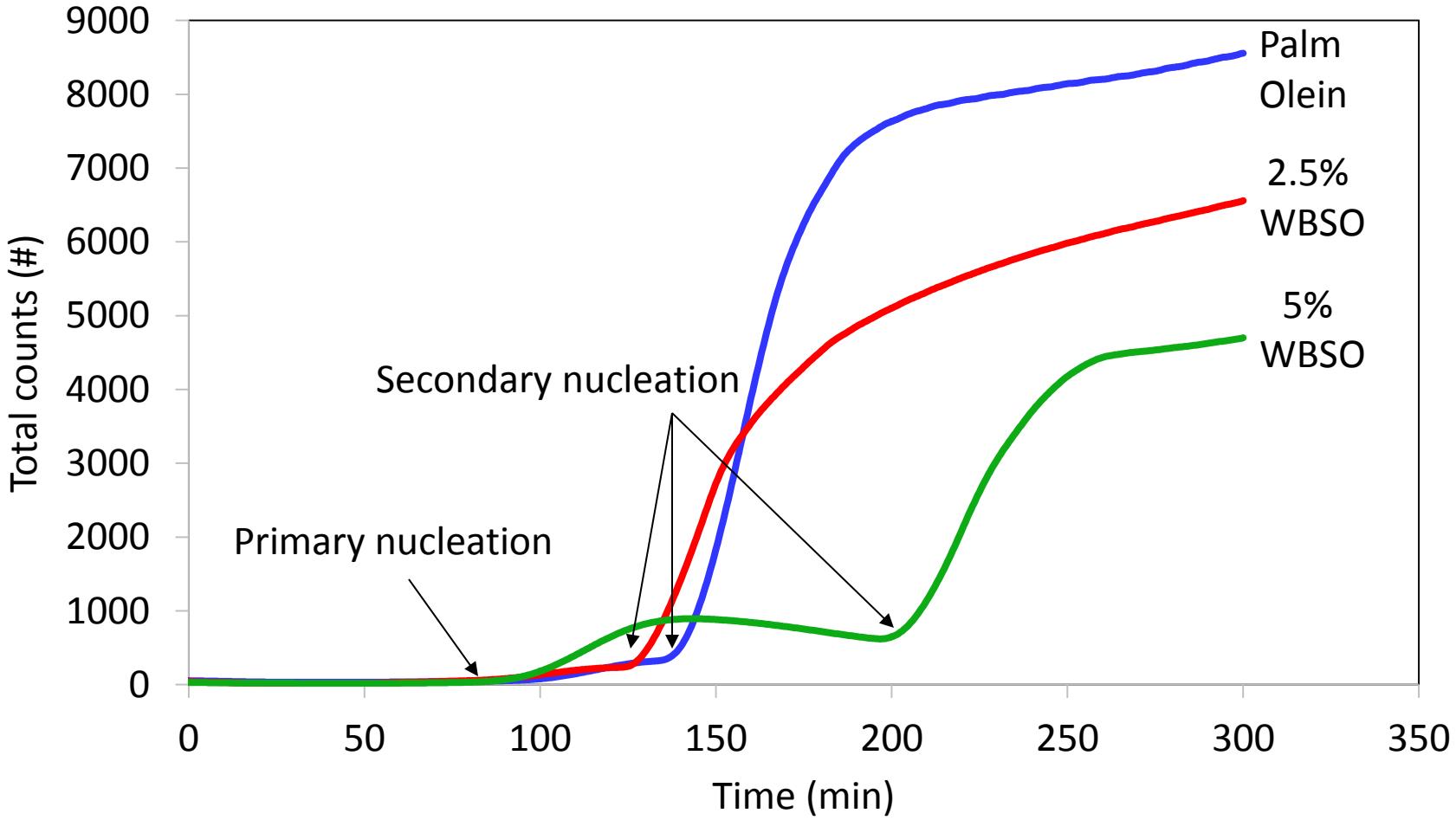


SUS : monounsaturated SUU : diunsaturated UUU : triunsaturated

OIL BLEND CRYSTALLIZATION

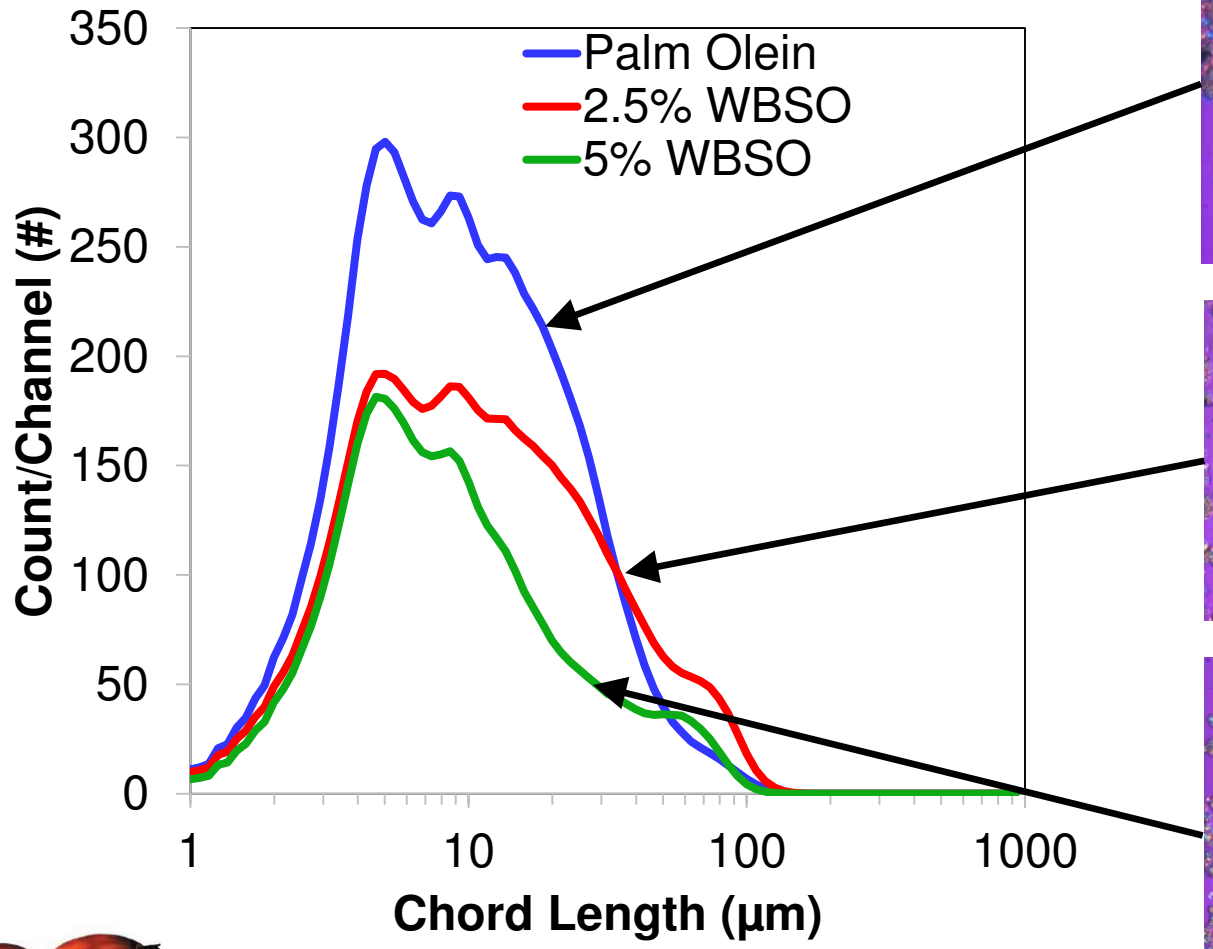
OIL BLEND CRYSTALLIZATION

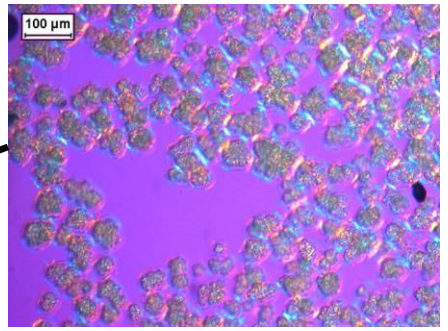
Crystals population of palm olein blended with winged bean seed oil at 13 °C

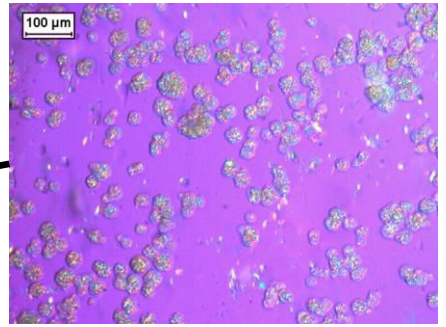


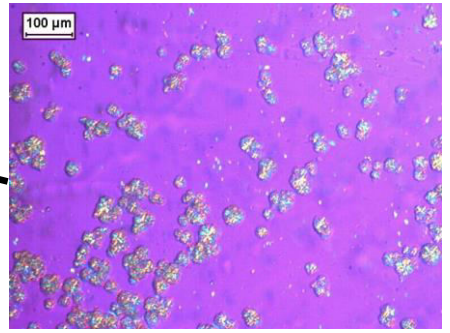
OIL BLEND CRYSTALLIZATION

Crystal size distribution of palm olein blended with winged bean seed oil at 13 °C











OIL BLEND CRYSTALLIZATION

Fatty acid composition of palm olein blended with winged bean seed oil at 13 °C

Fatty acid	Palm Olein	WBSO	2.5% WBSO in palm olein	5% WBSO in palm olein
C16:0	39.74	5.59	31.77	31.21
C18:0	4.30	3.90	3.95	3.95
C18:1	42.53	31.88	45.60	45.27
C18:2	11.21	37.13	15.74	16.27
C18:3	0.22	1.37	0.48	0.51
C20:0	0.43	1.03	0.34	0.35
C20:1	-	3.41	0.25	0.32
C22:0	-	11.90	**0.35**	**0.61**
C24:0	-	2.52	-	-
Iodine value	56.63	95.05	67.72	68.42



CONCLUSIONS

- FBRM is a useful tool to characterize crystallization of palm oil-based products and oils and fats systems, in general.
- FBRM provides valuable information such as population, size distribution and mechanisms of particle formation/disappearance.
- *In situ* and real time data – representative of actual process conditions.
- By complementing FBRM data with an imaging technique and compositional analysis, more information on crystallization behavior can be deciphered.
- Utilization of FBRM enables proper control and optimization of crystallization conditions to ensure production of optimum crystal characteristics.




MPOB
INTERNATIONAL
PIPOC 2015
PALM OIL CONGRESS
See You at
PIPOC 2015
6 - 8 October 2015
Kuala Lumpur Convention Centre,
Kuala Lumpur, Malaysia
MPOB
Malaysian Palm Oil Board (MPOB)
www.mpob.gov.my
Ministry of Plantation Industries and Commodities





Thank you



