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Where we are with the mitigation of 2- and 3-MCPD Esters and Glycidyl Esters?

Dr. Frank Pudel

Pilot Pflanzenöltechnologie Magdeburg e.V.

Outline

Oil processing, ester formation and mitigation strategies

Avoiding (removing) precursors

Changing refining parameters

Post refining

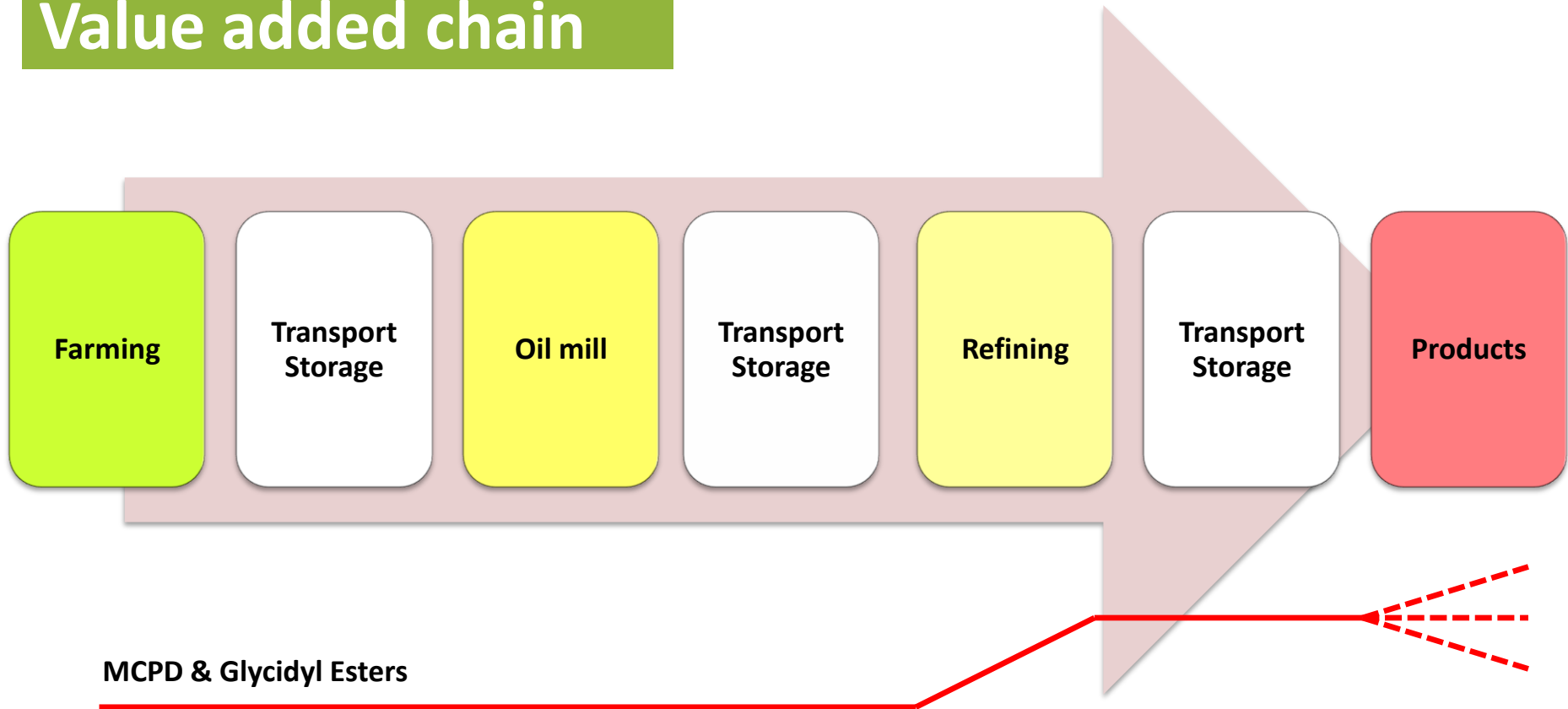
Ionic liquid treatment (a new refining process by Evonik)



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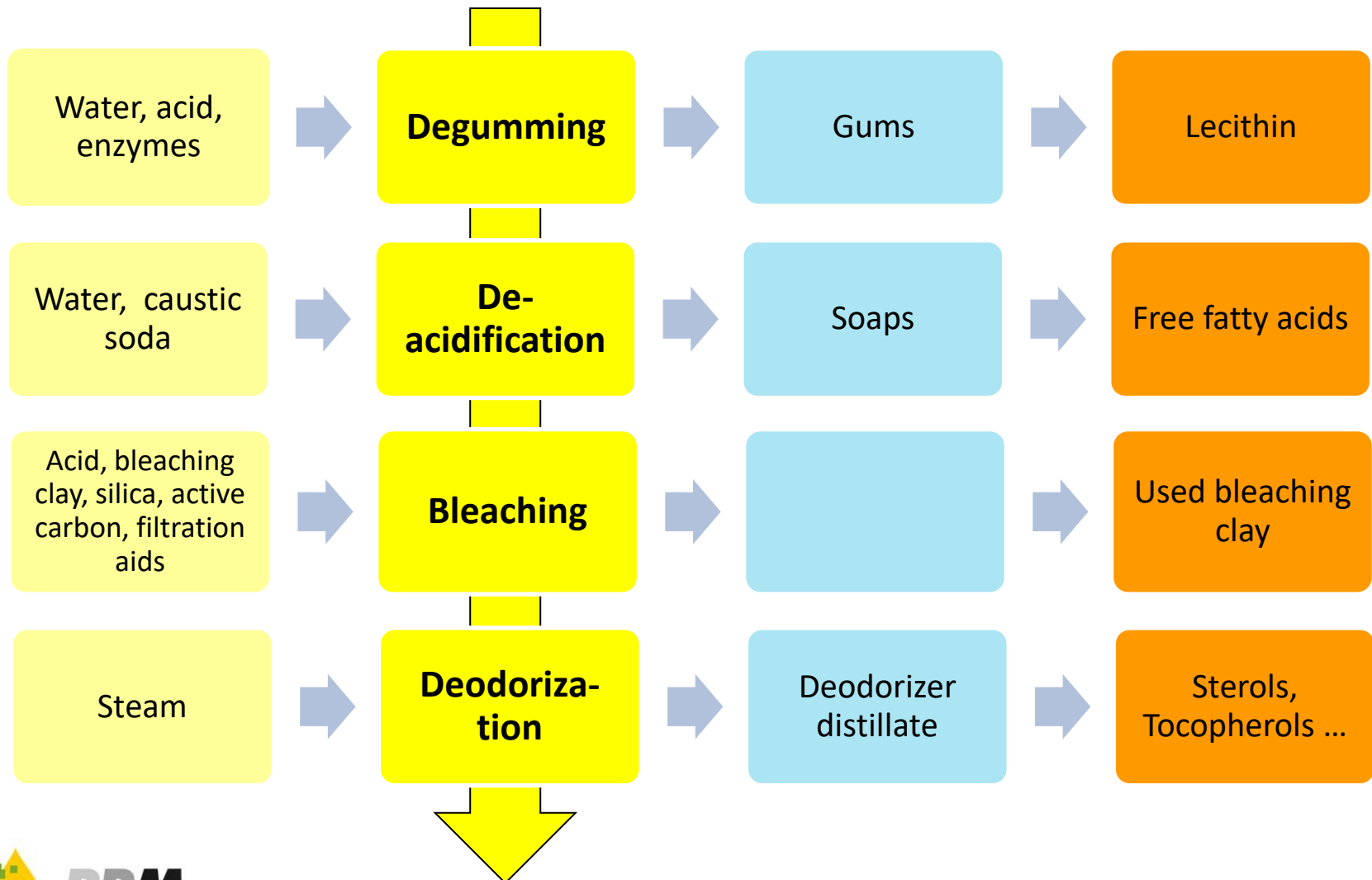
Value added chain



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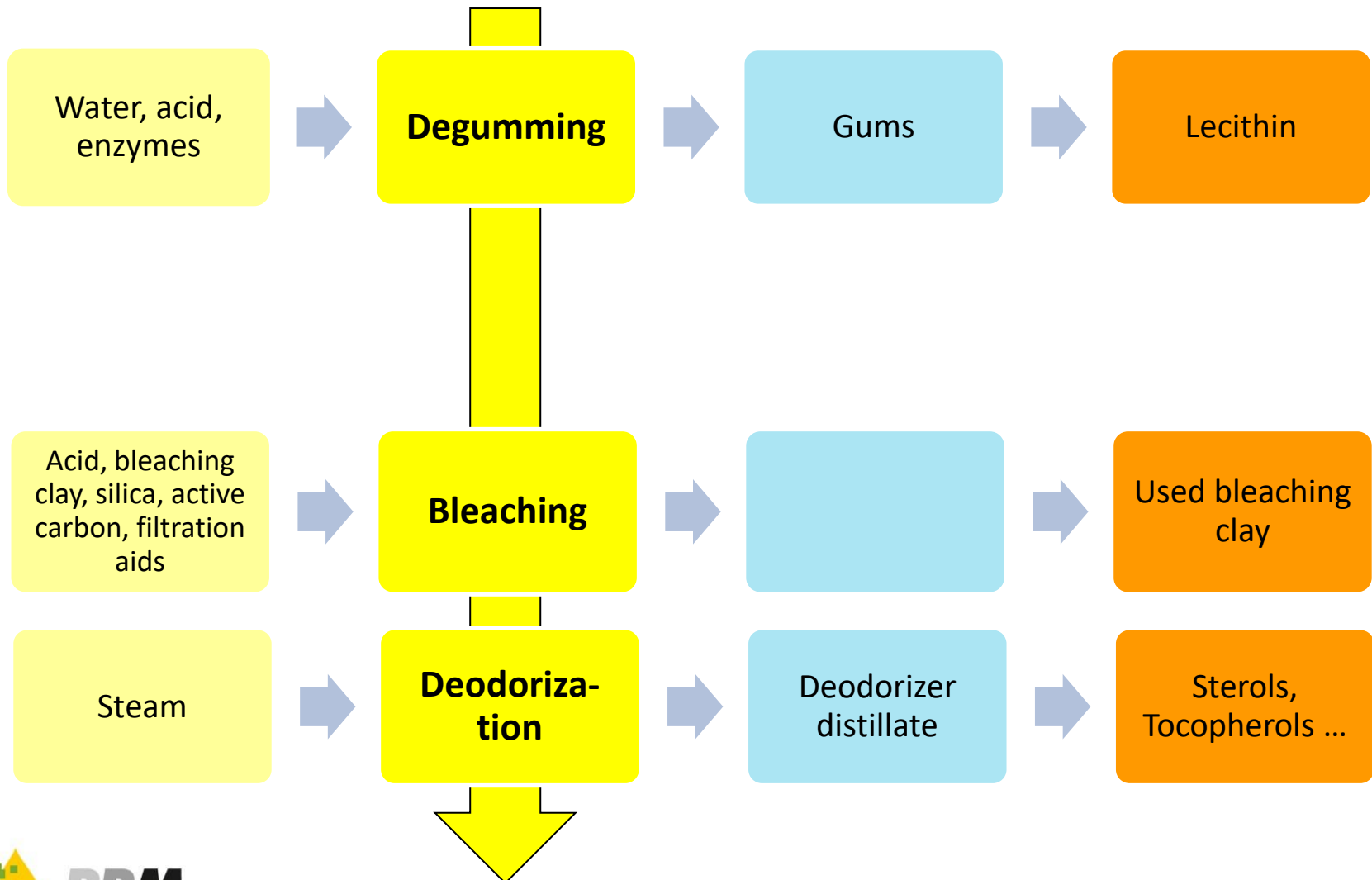
Chemical refining



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Physical refining



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Factors for ester forming

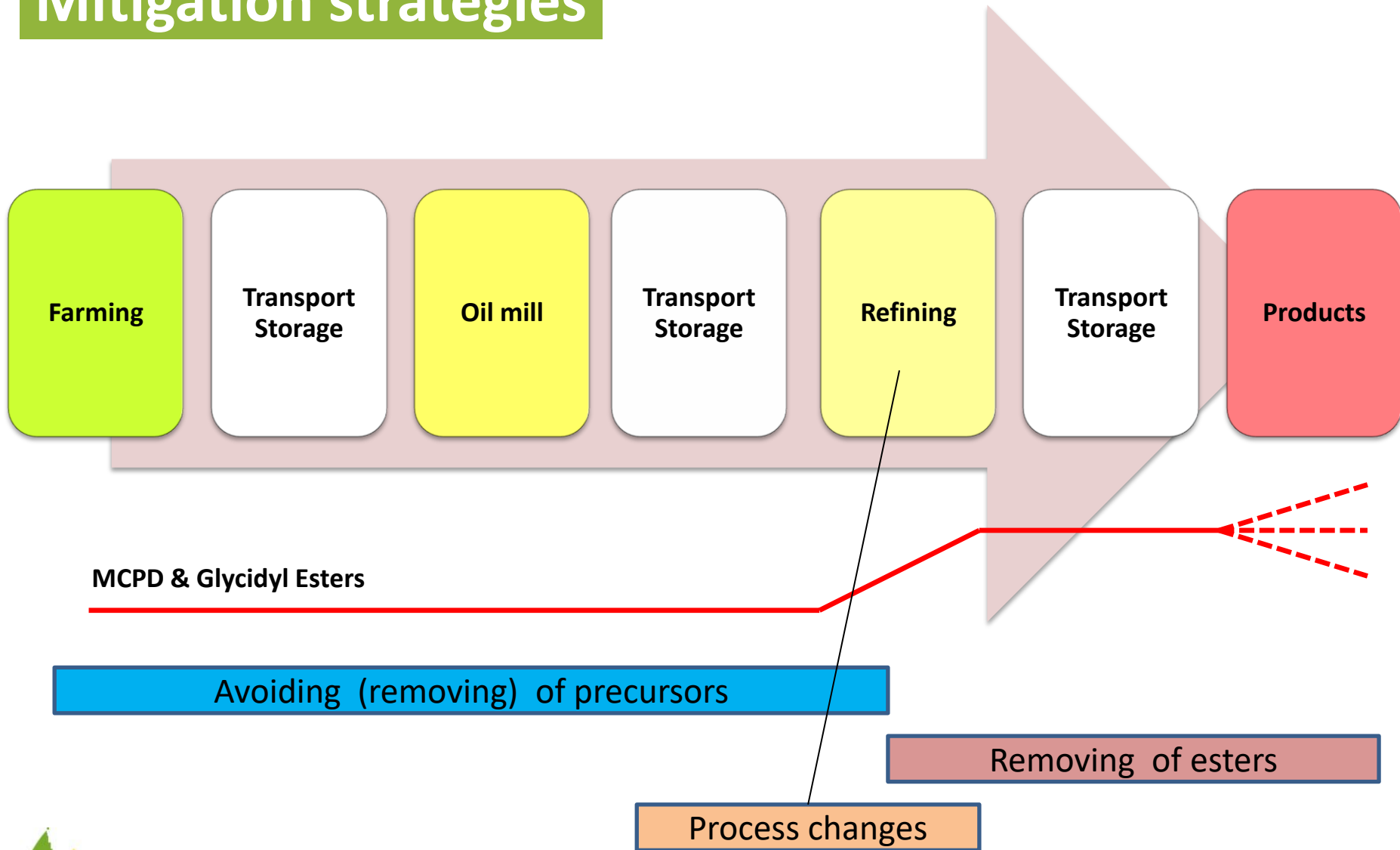
	MCPD Esters	Glycidyl Esters
Precursors	Cl (organic & inorganic) Lipids (mono-, di-, triglycerides, phospholipids)	Diglycerides
Process conditions	Temperature (> 150°C) Time pH	Temperature (>230°C)



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Mitigation strategies



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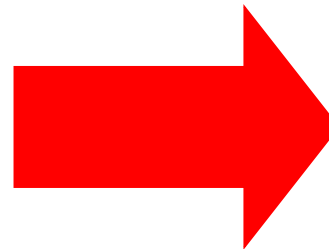
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Importance of raw material



Palm fruits are

- not suitable for storage
- metabolic processes begin already on the tree (at full ripeness) or directly after harvest
- very sensitive against pressure and injuries



Formation of

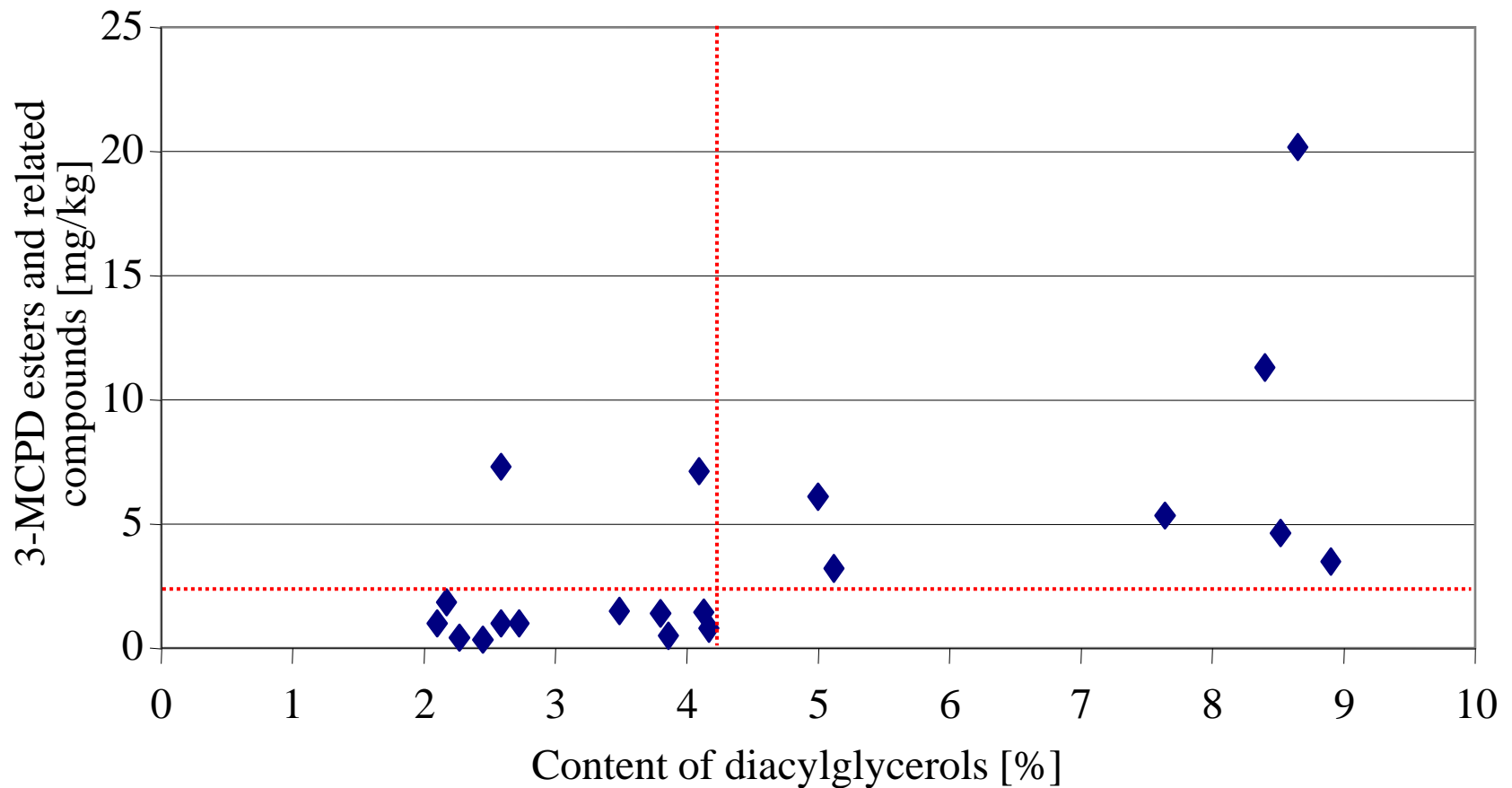
- Free fatty acids
- Diglycerides



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Influence of DAGs



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How to get low DAGs

- Harvest at optimal ripeness
- Reject damaged material
- Do not use loose fruits
- Process (sterilization) as quick as possible (3-6 hours)

Zieverink et al., 8th EFL Congress, Munich, 2010

- Choice / Breeding of varieties poor in lipase activity (?)

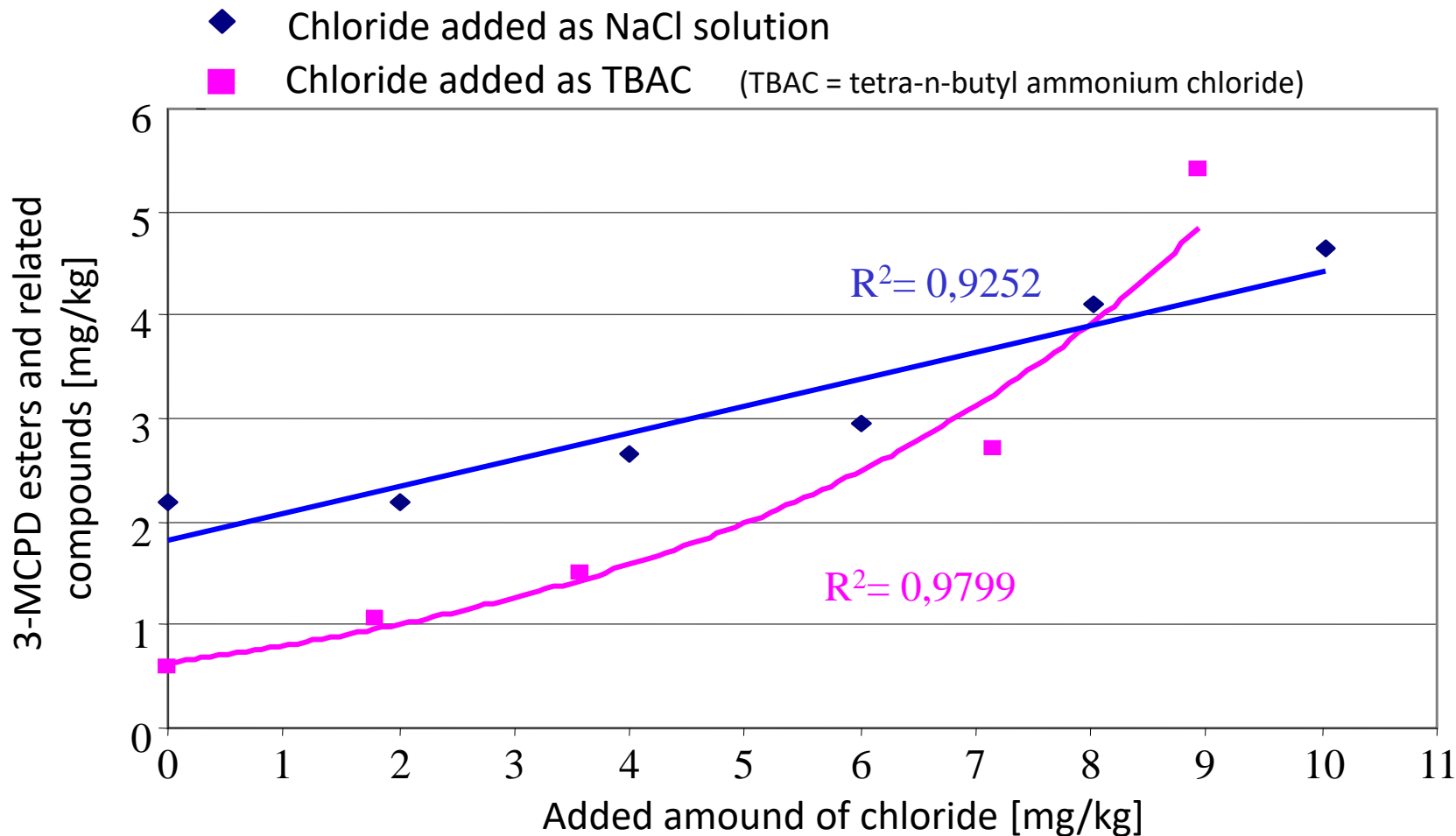
Ebongue et al., EJLST 110 (2008) 505-509



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Influence of chloride



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How to get low Cl

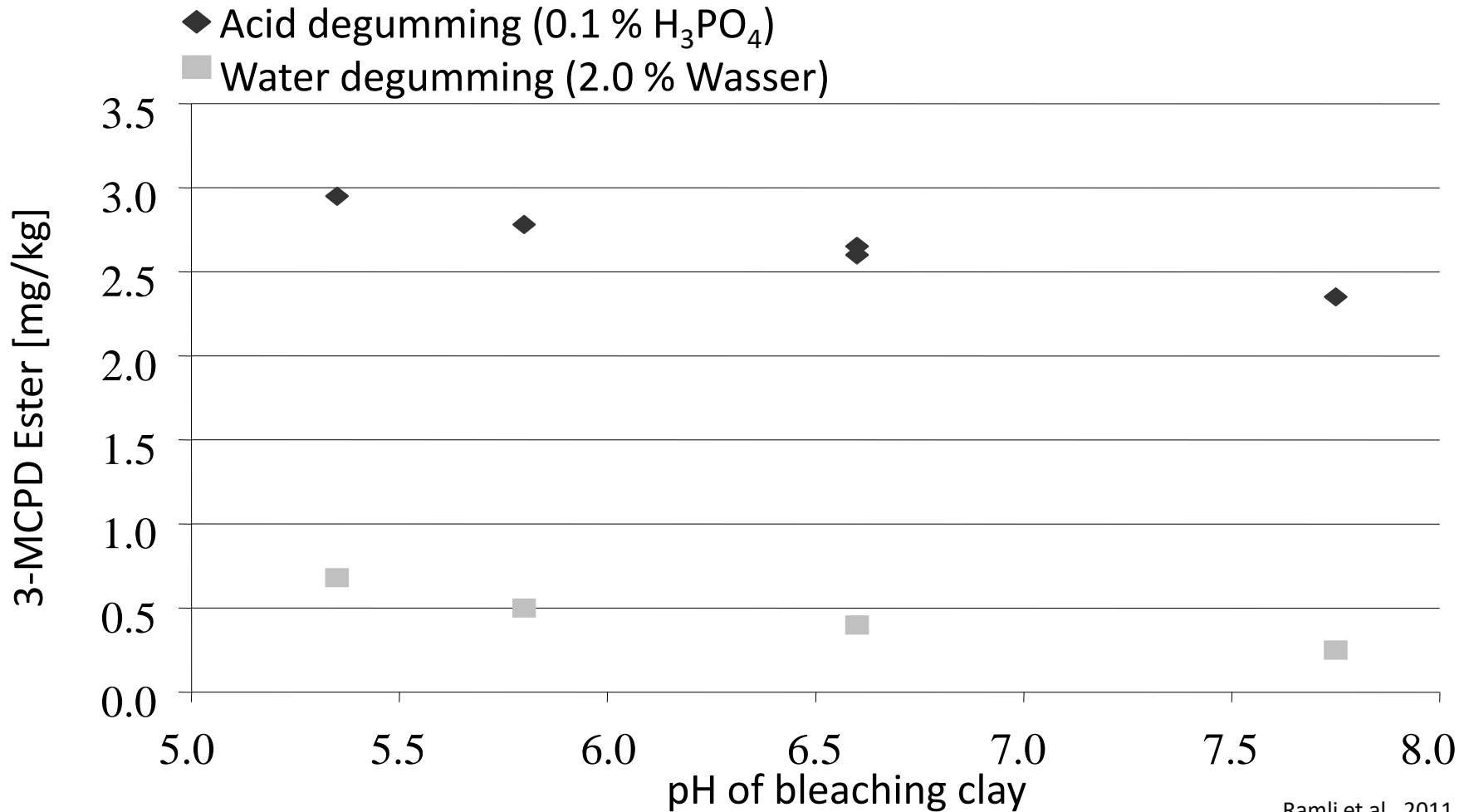
Minimize the use of chloride containing substances in cultivation

- Fertilizers
- Pesticides
- Irrigation water

Avoid cultivation on saline soils

Destailats et al., Food Additives & Contaminants 1 (2012) 29-37; Craft et al., Food Additives & Contaminants 29 (2012) 354-361; Abdollah, TU Malaysia, 2010

Influence of pH



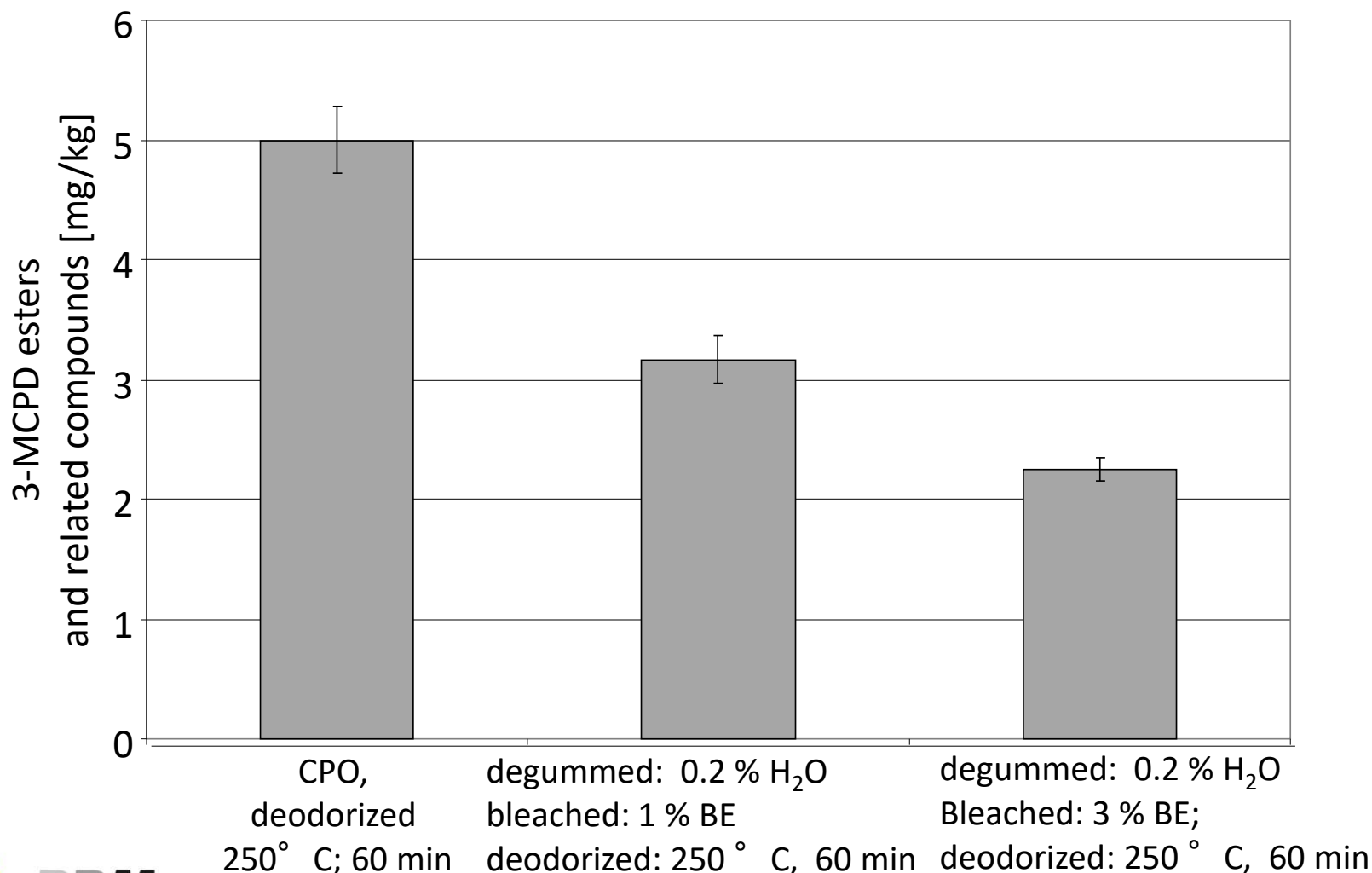
Ramli et al., 2011



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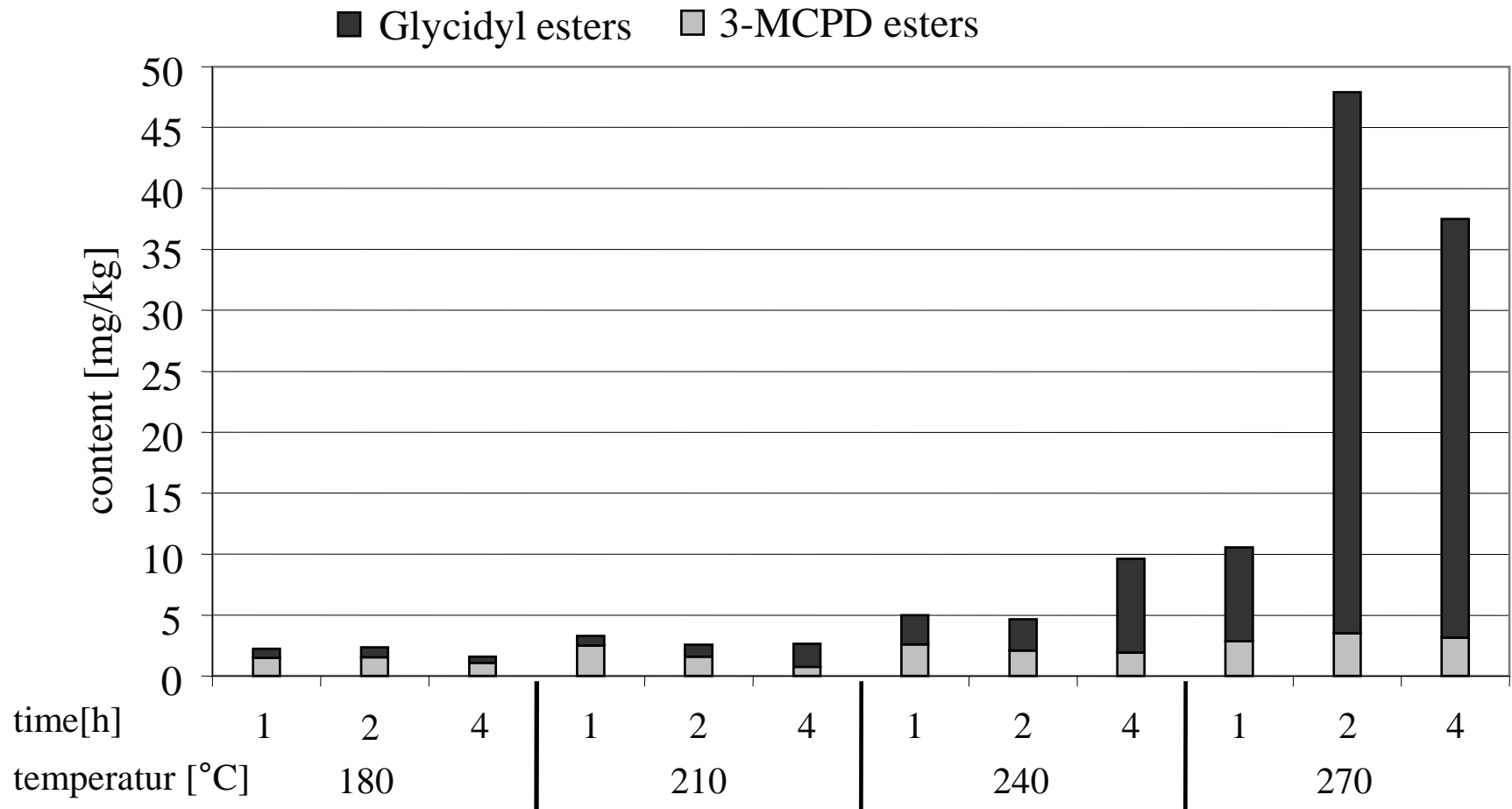
Influence of bleaching clay amount



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Influence of deodorization temperature and time



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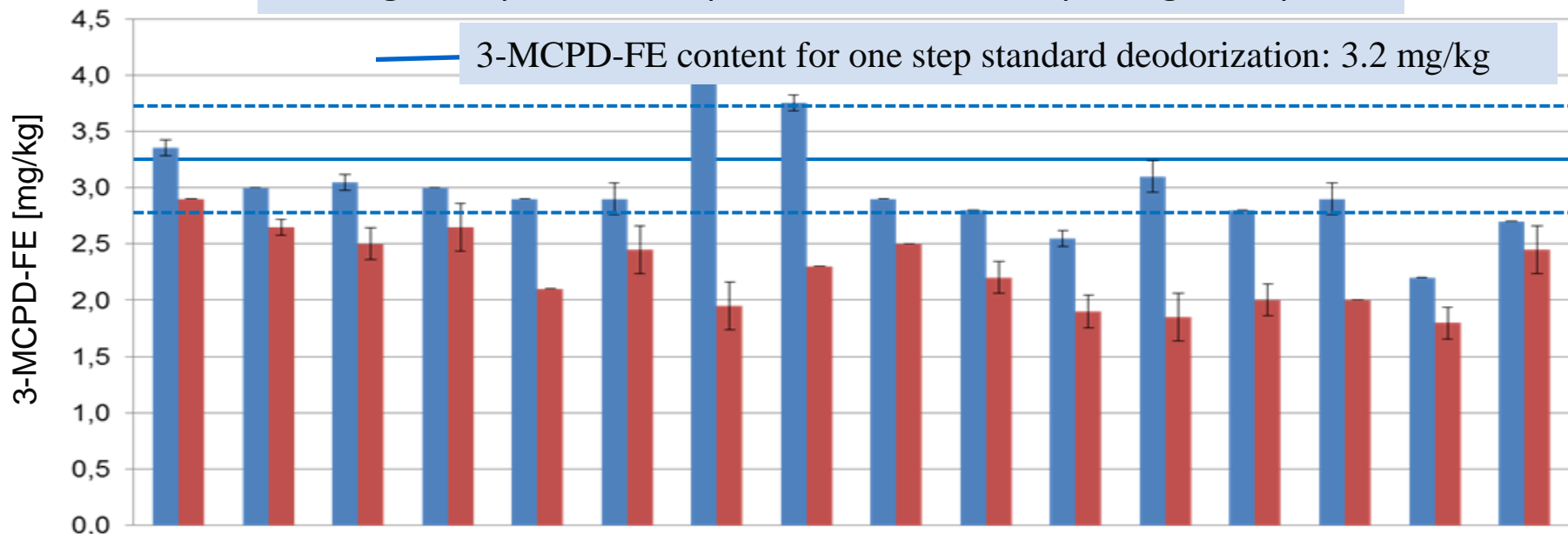
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2-step deodorization – Effect on 3-MCPD esters

■ high temp. / low temp.

■ low temp. / high temp.

— 3-MCPD-FE content for one step standard deodorization: 3.2 mg/kg



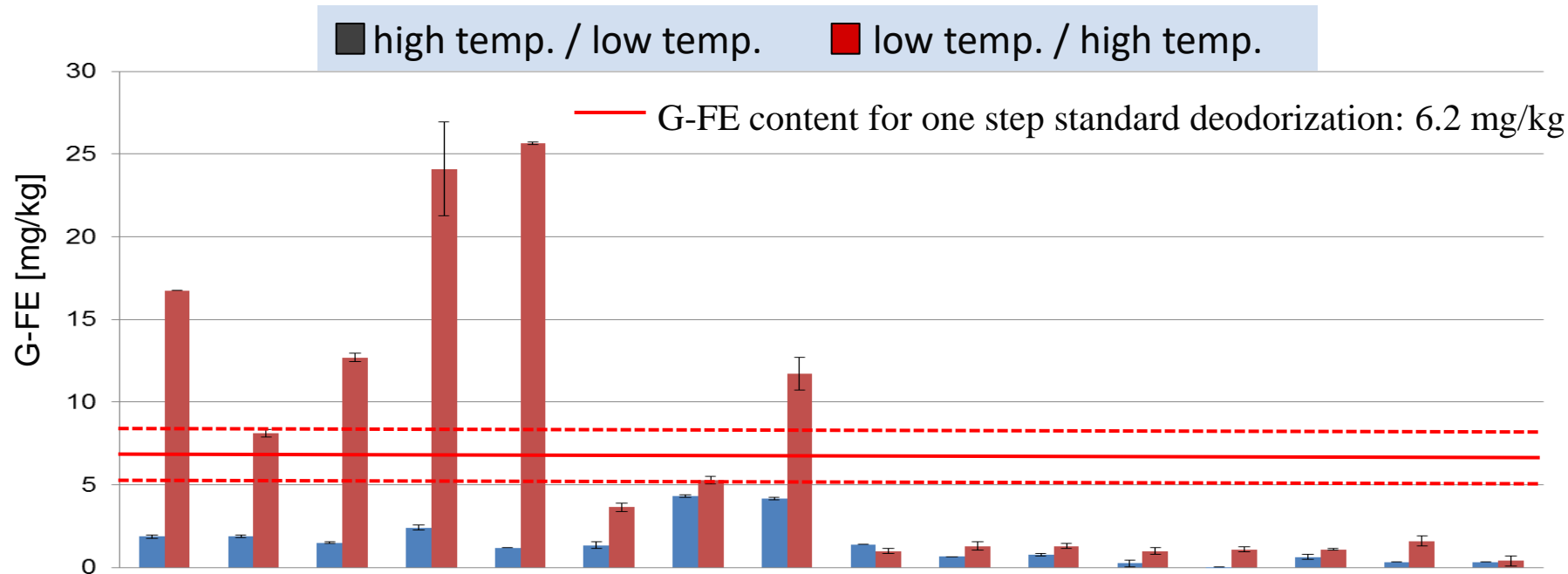
° C min	280 20	280 20	280 20	280 20	280 5	280 5	280 5	280 5	240 20	240 20	240 20	240 20	240 5	240 5	240 5	240 5
° C min	230 120	230 60	200 120	200 60	230 120	230 60	200 120	200 60	230 120	230 60	200 120	200 60	230 120	230 60	200 120	200 60
AV (High/low)	0.14	0.09	0.09	0.04	0.08	0.19	0.53	1.98	0.08	0.11	0.17	0.54	0.11	0.89	1.09	2.86
AV (Low/high)	0.21	0.45	0.5	0.3	0.18	0.6	0.53	0.65	0.09	0.62	1.19	1.47	0.07	0.53	0.74	2.31



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2-step deodorization – Effect on Glycidyl esters



° C min	280 20	280 20	280 20	280 20	280 5	280 5	280 5	280 5	240 20	240 20	240 20	240 20	240 5	240 5	240 5	240 5
° C min	230 120	230 60	200 120	200 60	230 120	230 60	200 120	200 60	230 120	230 60	200 120	200 60	230 120	230 60	200 120	200 60
AV (High/low)	0.14	0.09	0.09	0.04	0.08	0.19	0.53	1.98	0.08	0.11	0.17	0.54	0.11	0.89	1.09	2.86
AV (Low/high)	0.21	0.45	0.5	0.3	0.18	0.6	0.53	0.65	0.09	0.62	1.19	1.47	0.07	0.53	0.74	2.31

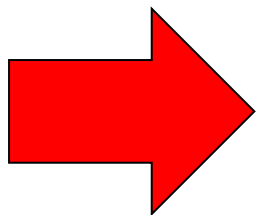


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Results of 2-step deodorization experiments

- **Most promising:** Short deodorization at higher temperature first with following longer deodorization at lower temperature results in:
 - Remarkable lower contents of G-FE
 - Comparable contents of 3-MCPD-FE
 - Slightly lower peroxide values
 - Slightly lower oxidative stability



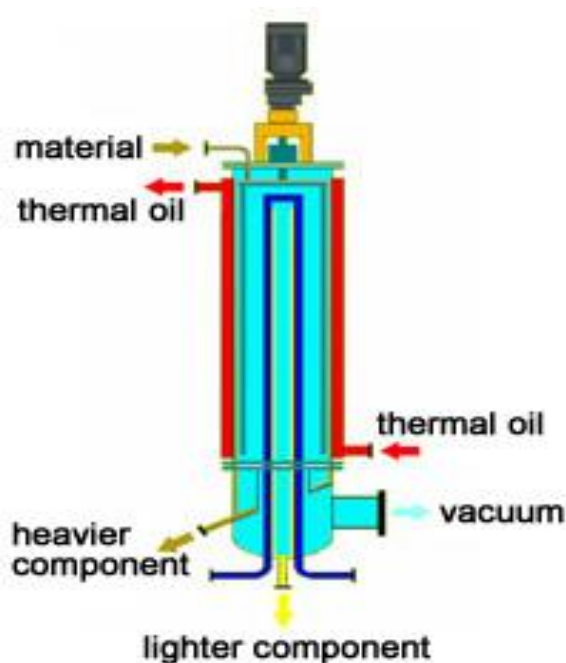
But: Target conflict between low acid value and low content of 3-MCPD-FE/G-FE; but compromise is possible for low G-FE and constant acid value and 3-MCPD-FE



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Short path distillation instead of deodorization



Feature of SPD	Advantage
High vacuum (up to 0.001 mbar)	Decreased boiling point
Short heating time	Reduced exposure to heat
Evaporation from a thin layer	No static pressure loss
	More efficient heat transfer
	Large surface area per volume element
Turbulent circulation	No thermal gradient Permanent surface renewal
Short steam path	Only one evaporation No gas veil
No stripping steam	No waste water No hydrolytic splitting
Tight system Condensation of vapors	Less energy use



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Results of short path distillation experiments



A B C D

A : Crude palm oil (CPO)

B : degummed palm oil

C : Palm oil after SPD

D : Palm oil after conventional
deodorization

- Short path distillation (SPD) is a suitable method to produce a refined palm oil without 3-MCPD & Glycidyl esters.
- Its chemical quality parameters are comparable with a conventionally refined palm oil.
- SPD results in a red colored palm oil.
- Sensory is negatively influenced.
- Depending of crude oil quality a subsequent gentle deodorization at 160 - 180°C eliminates sensory disadvantages. This leads to slightly enhanced 3-MCPD esters.



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How to refine

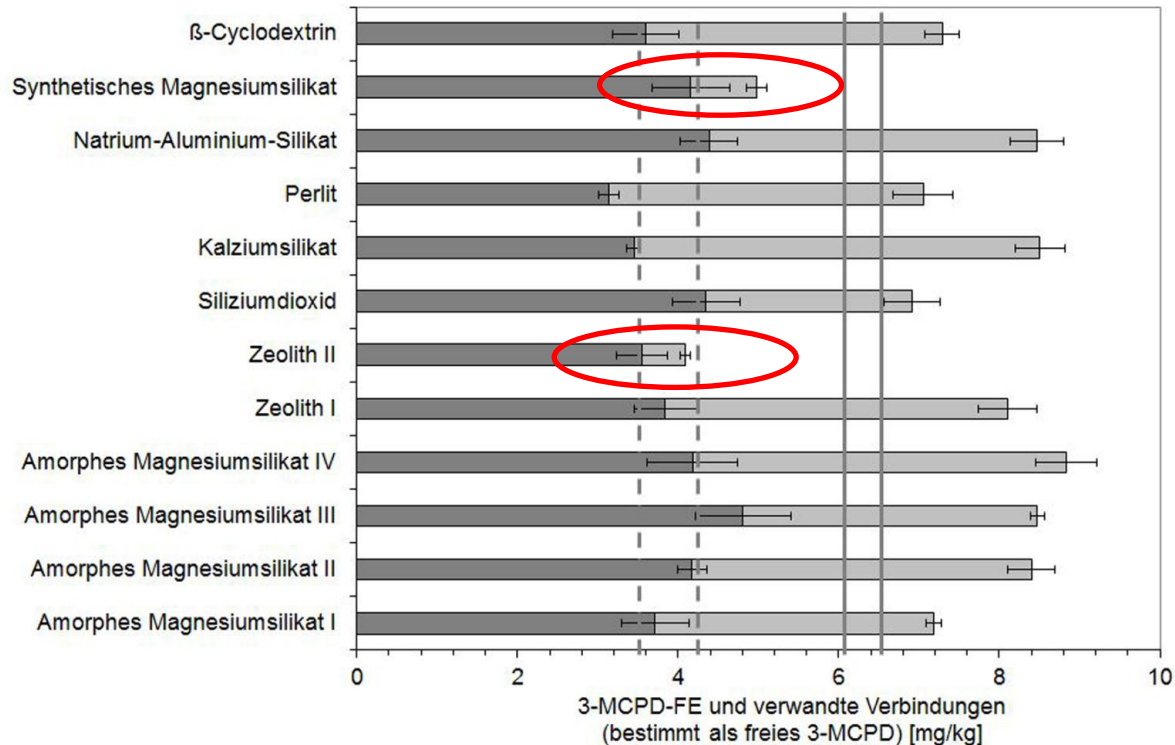
- Choose / use suitable crude oil (low in DAG and ffa)
- Use chemical refining instead of physical refining
- Avoid low pH (e.g. by degumming with low acid amounts; neutralisation with calcium oxide)
- Use significant increased amounts of bleaching clay
- Use natural bleaching clays
- Use lower temperatures in deodorizing
- Use 2-step deodorizing (HTST followed by LTLT)
- Use short path distillation followed by gentle deodorization



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Post bleaching by adsorbents



3-MCPD Esters
 Glycidyl Esters

3-MCPD Esters, RBD oil
 Glycidyl-Esters, RBD oil

Strijowski et al., 2011

3-MCPD esters are removed by carboxymethyl cellulose or cation exchange resin under nitrogen (WO2011009841-A1)



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Post refining methods

- Post bleaching with different adsorbents followed by gentle deodorization
- Double bleaching / deodorization
- Use of short path distillation after deodorization

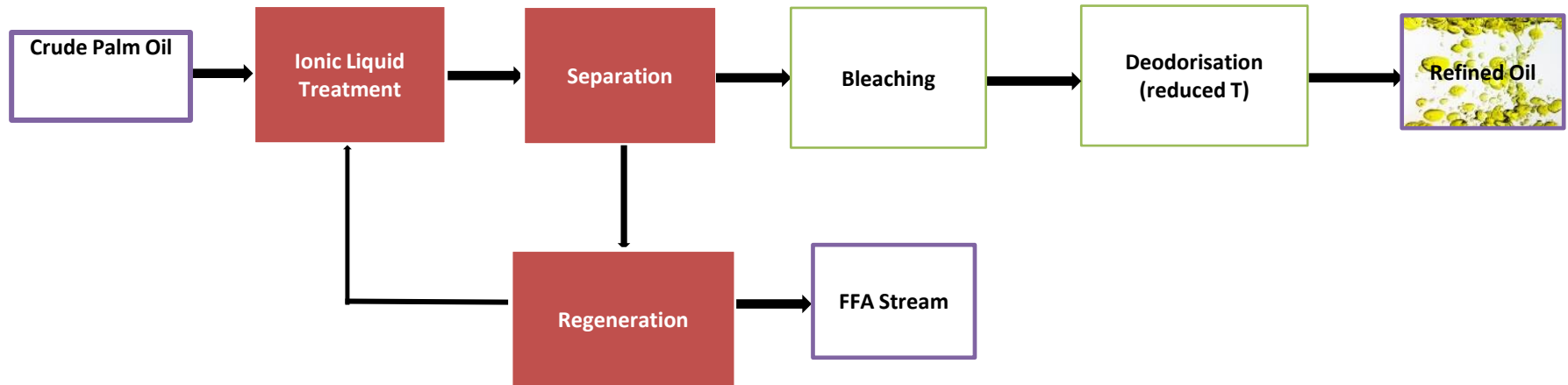
(WO2015/073359 – Cargill)



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Treatment with ionic liquids

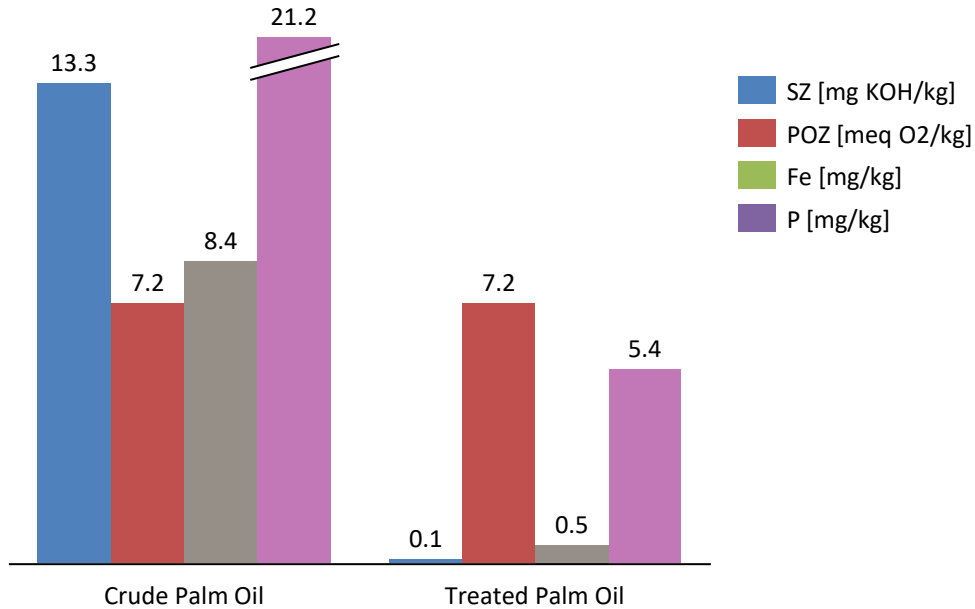


Advantages:

- Ionic Liquid Treatment allows **mild conditions and less resources** in degumming, bleaching and deodorization
- The treatment with ionic liquid is a **reactive extraction of free fatty acids** and similar to the chemical refining, but **can be regenerated**
- Lower energy demand and waste reduction have **positive impact on planet category of sustainability**
- Formation of potential carcinogenic by-products is reduced resulting in **health benefits for the customer**

WO2016189115, WO2016189114, WO2015079262

IL treatment: oil quality



- Reduction of phosphor and iron content is observed
- Free fatty acids are almost completely removed after treatment step

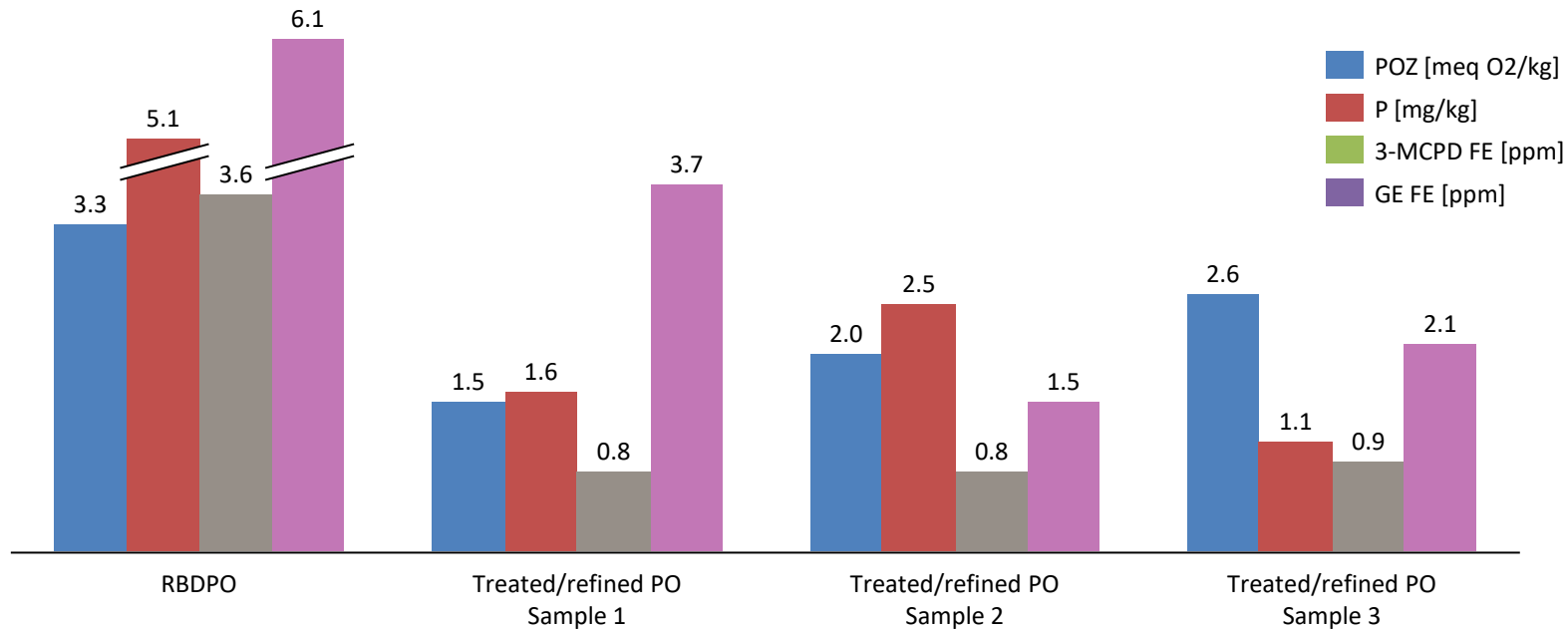


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IL treatment: 3-MCPD and Glycidyl esters



- With the treated palm oil a reduction of deodorization temperature < 230 °C is possible
- The amount of GE and 3-MCPD content is significantly reduced in comparison to conventional refined Palm oil (RBDPO)



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Questions?

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