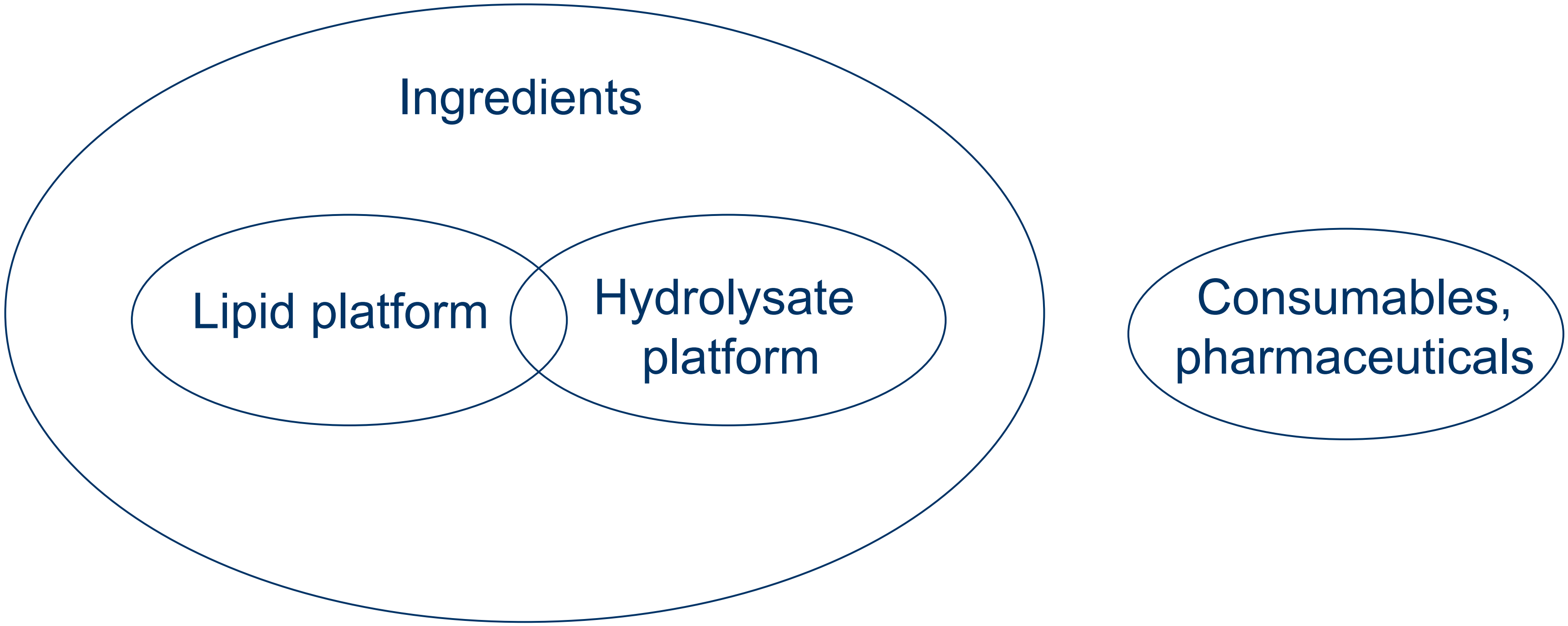
Lipidplatform og hydrolysplatform Sintef Fiskeri og havbruk

Ivar Storrø
Trondheim

Structure of rest raw material platform

Marine rest raw materials



To produce oil and protein hydrolysate of premium quality

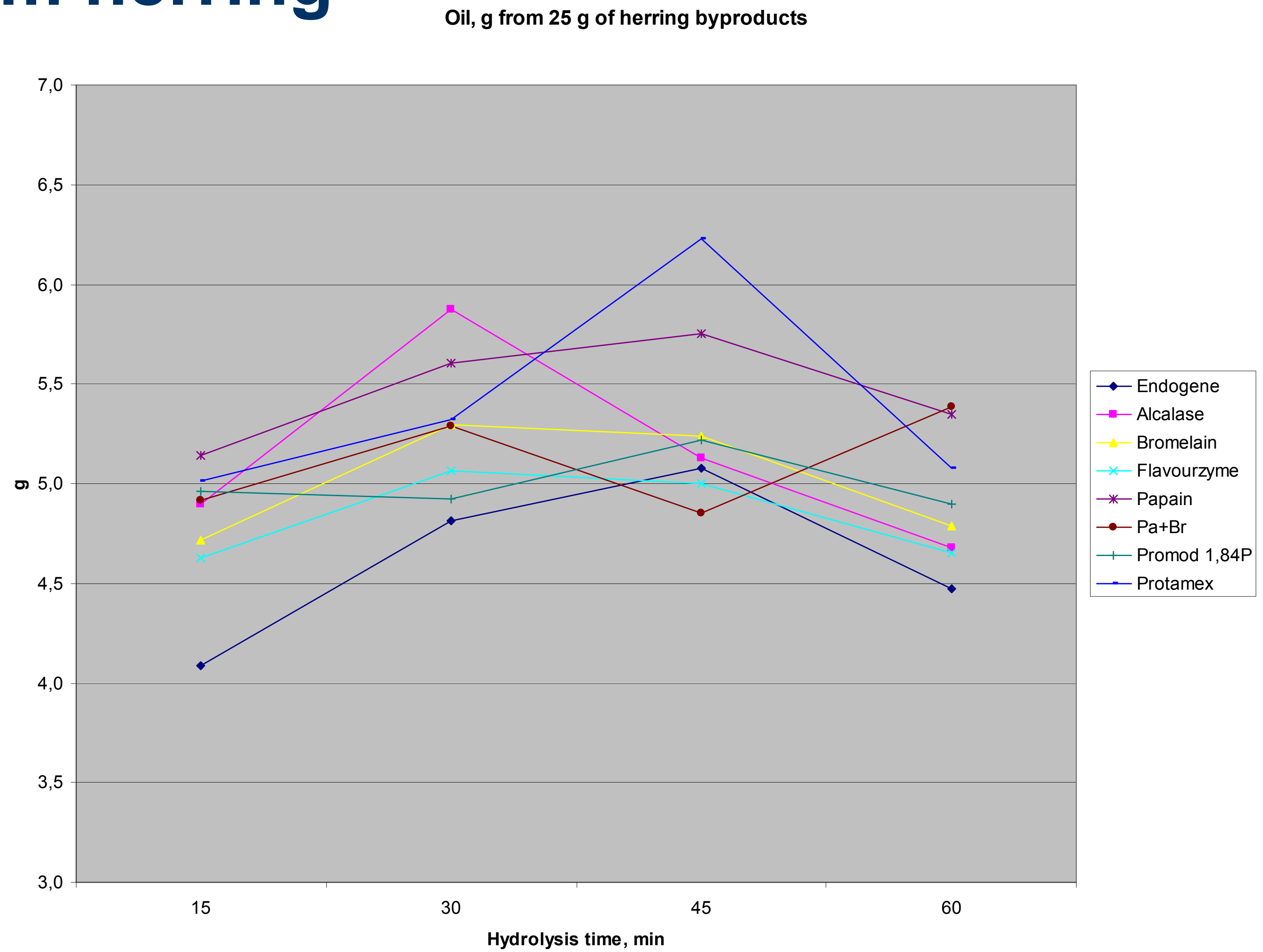
Hydrolysis platform, overview

Biochemistry of hydrolysis	Process optimization	Process verification	Process design
Choice of enzyme(s) Characterisation of raw material and product Reactors, 6 of 250 ml, 32 of 3L	Selection of conditions for best profit Yield, quality Temperature, water content pH, time… 32 reactors of 3L	The best hydrolysis conditions are tested on site with Mobile SeaLab. 1 reactor of 800L. Capasity 500kg/h	Scale up data . Dewatering and drying in cooperation with SINTEF Energy and Enginering companies

Analytical capabilities:
Characterisation: Protein, lipid, ash and water content, Amino acid composition, free and bound, Molecular weight distribution, Water holding capasity, Foaming and emulsifying capability, Fat absorption, Antioxidative properties.
Instrumentation: GC, FPLC, NMR (Low-high field), GC-MS-MS, LC-TOF-MC, electrophoresis

Oljeseparasjon og proteinhydrolyse forsøk

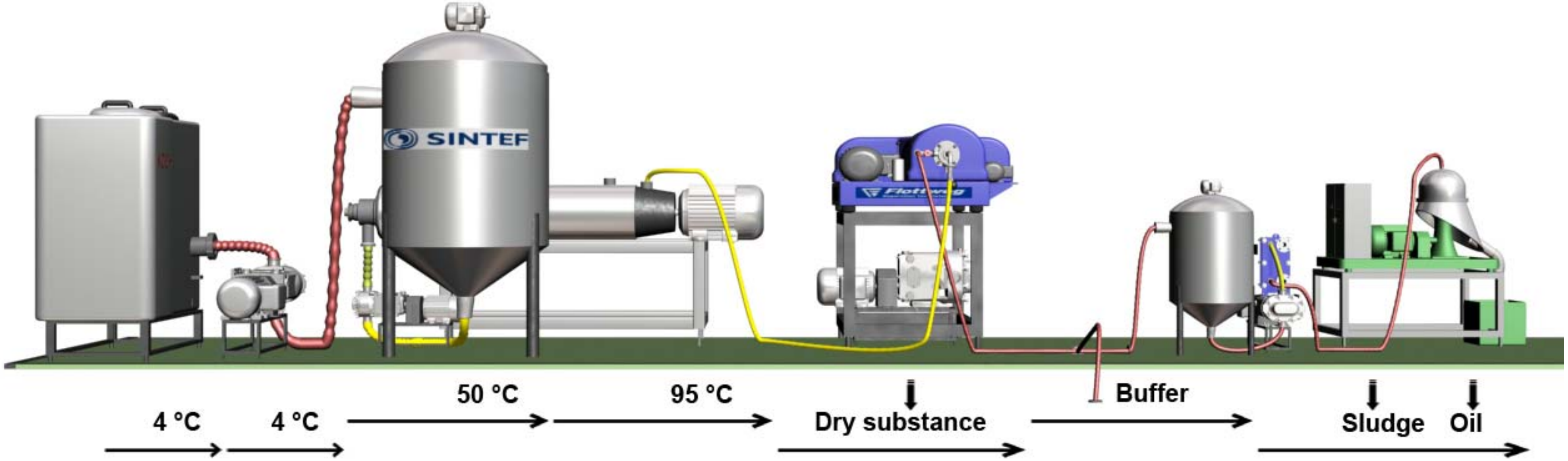
Oil yield from hydrolysis of byproducts from herring



Mobile SeaLab

Mobile Sea Lab

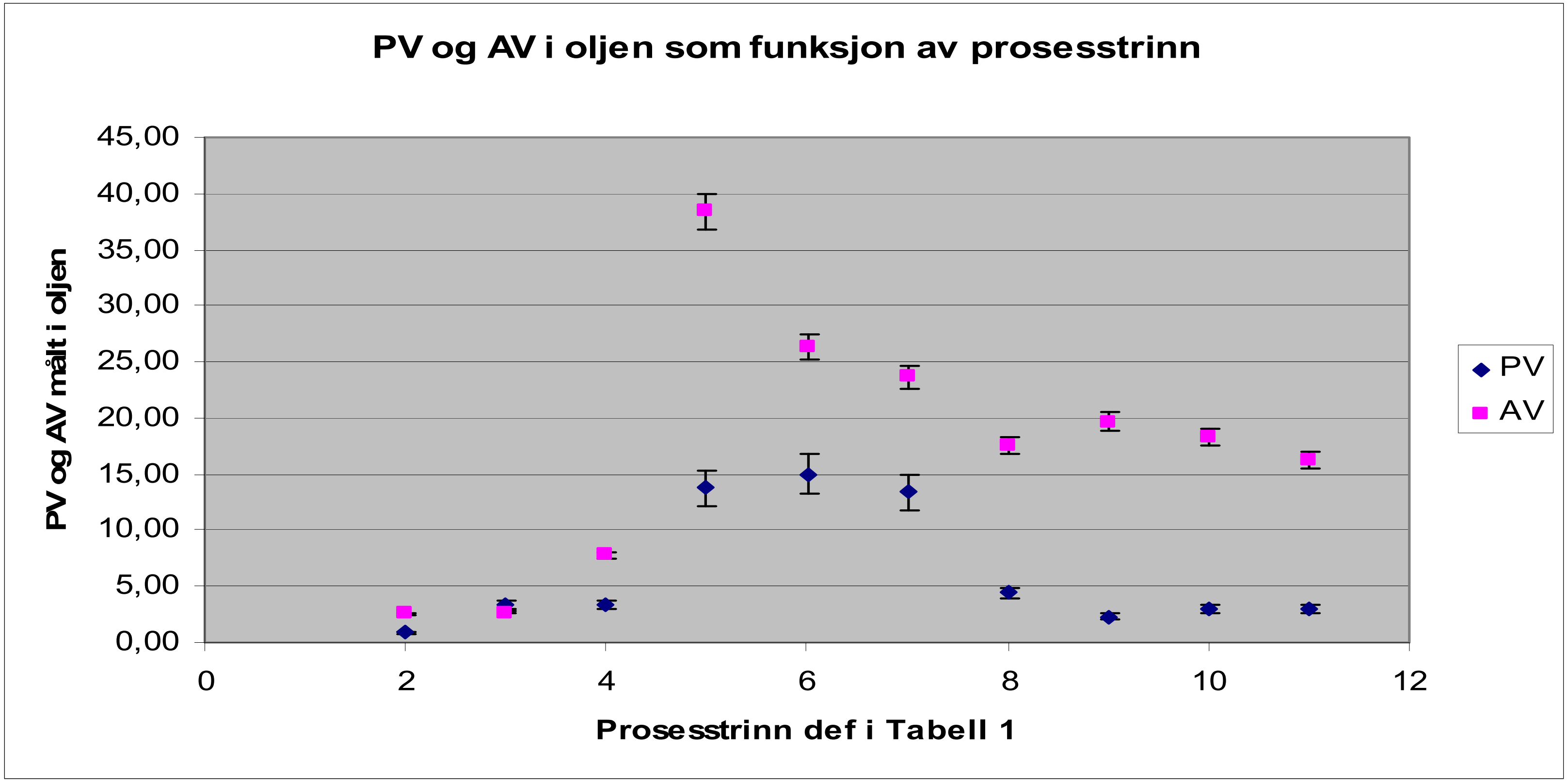
Batch hydrolysis- and oil production with decanter



SINTEF

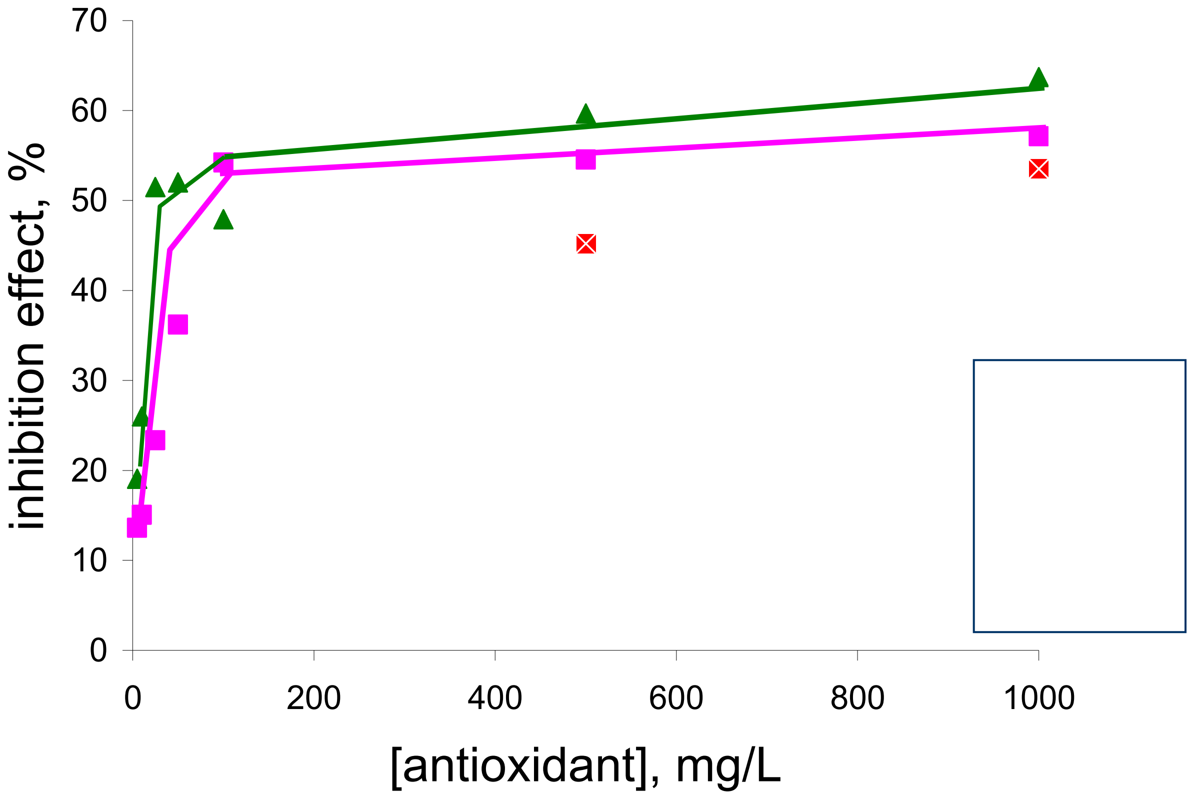
http://www.sintef.no/Projectweb/Mobile-Fish-Refinement/

Oksidasjon av lipidene under hydrolyseprosesssen



Select optimal antioxidants :

- XX, YY – inhibiting effect



Lipid platform, overview

Extraction	Purification	Transformation	Stabilisation
Production of raw oil	Production of highly purified oil	Production of new lipids	Prevent oxidation in oil and food
Extraction equipment in lab and pilot scale. From 50 ml batch to 1000 kg/h continuous (Mobile SeaLab)	Reactors (100L) for: Degumming, bleaching Deodorization (1000L) Short path distillation (lab and pilot 10l/h) Infrastructure (0,5 mill €)	Reactors: 6 reactors of 250 ml 32 reactors of 3 L Several reactors from 8 L to 1500L	Equipment for measuring oxidation kinetics (OSI, and oxygen consumption). Effect of pro oxidants and anti oxidants

Analytical capabilities:
Characterisation: Fatty acid composition, lipid classes, phospholipid classes, positioning of fatty acids
Wet chemical: PV, Ansidin value, TBARS,
Instrumentation: GC, Iatroscan, HPLC-Corna, NMR (Low-high field), GC-MS-MS, LC-TOF-MC

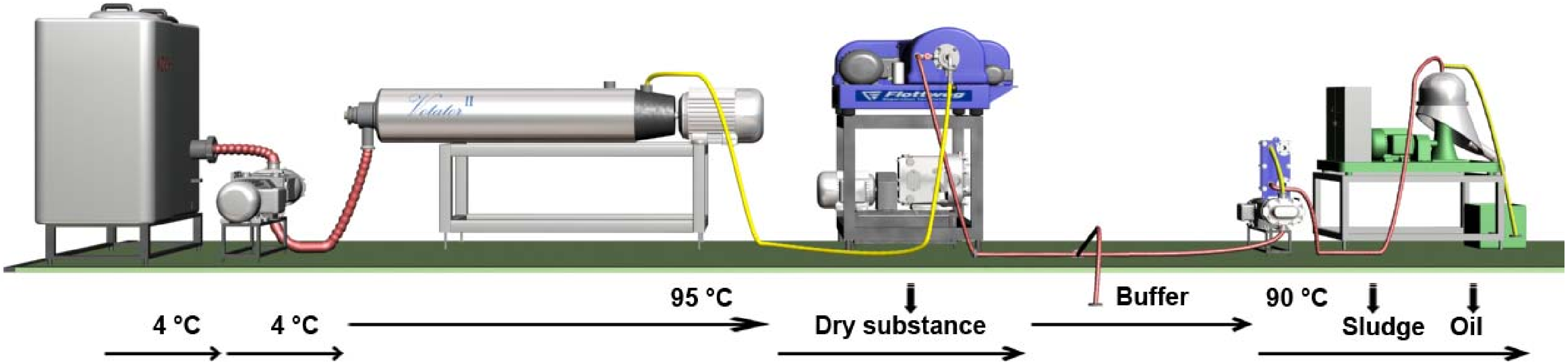
ω3-oil-Extraction

- Optimal conditions for extraction (pH, temp.)
- Stabilisation of oil during extraction.
- Selection of best antioxidant for use during extraction.
- Selection of best process design for extraction combined with production of protein hydrolysate.
- Understanding of lipid oxidation in raw oil.
- Selection of best antioxidant to be used in raw oil.

ω3-olje-Extraction hardware

Mobile Sea Lab

Continuous oil production with decanter



SINTEF

ω3-oil-Purification

- Equipment in-house:
  - Glass reactors for refining.
  - A 1000 L reactor for deodorization (200°C, full vacuum)
  - VTA Short path distillation. Capacity 1-10 l/h.

- Equipment to be installed:
  - Five 100 L steel reactors ordered.
  - Granted 4,4 mill NOK for equipment inc bleaching equip and infrastructure.

ω3-oil-Transformation

- Modification of lipids.
  - Chemical modification
  - Use of immobilized enzyme.
- Inhouse hardware:
  - 32 fully equipped enzymatic reactors (2 L)
  - Other reactors 8L, 100L, 300L and1500 L.
  - (Secret: These are fementers at SINTEF Materials and chemistry)

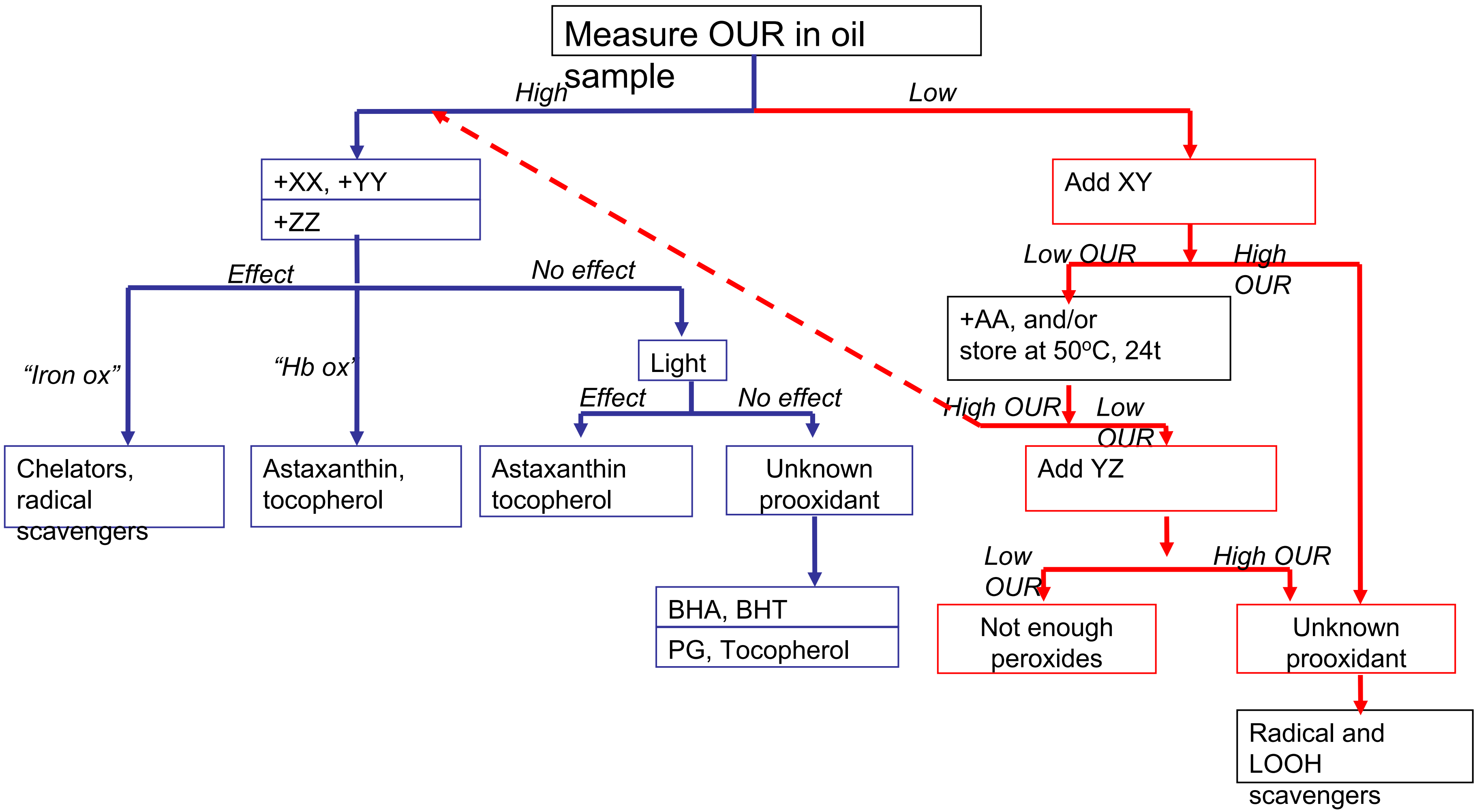
- Competence: Several years of work with immobilized biocatalysts.
- Special competence: Mass transfer, Rx kinetics
- One student (NTNU) working in this area.

Enzyme reactors for lipid transformation

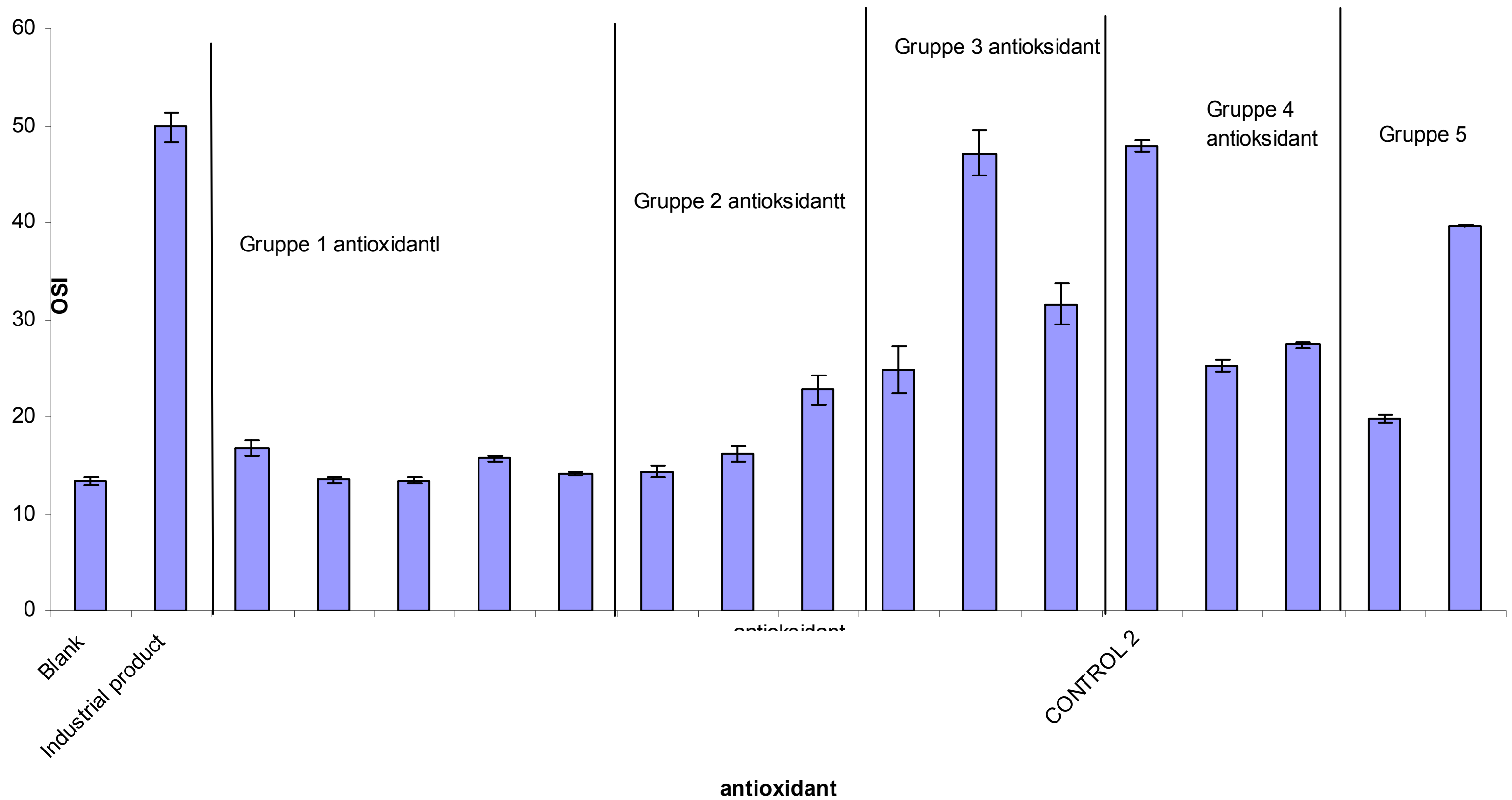
Ω-3 Oil - Stabilising

- Understanding the lipid oxidation!
- How do prooxidants work?
  - Reduction of the concentration of prooksidants.
- How do antioxidants work?
  - The right antioksidant
  - Optimal concentration of the antioxidant.

Strategy for selecting effective antioxidant



Riktig valg av antioksidant



Analysis

- We shall have minimum two independent methods for analysing the same components.
- Fatty acids
  - GC, NMR, LC-MS
- Lipid classes
  - Iatroscan (TLC med FID), HPLC with Corona detector, Camag (HPTLC)
- Phospholipid classes
  - HPLC with Corona detector, ^{31}P-NMR
- Fatty acid positioning (sn1, sn2, sn3)
  - NMR

The Mass Spectrometric Laboratory

- 3 GC-MS
- 4 LC-MS (Singelquadropole)
- 1 LC-Ion Trap
- 1 LC-QQQ (Trippelquadropole)
- 1 LC-TOF
- 1 LC-QTOF
- 1 GCxGC-QTOF

- Laboratory Manager: Kolbjørn Zahlsen
  - SINTEF Materialer og kjemi.

Compounds found in purified high omega-3 oil

Analytt	% prob
2-Butanone	87
Propanoic acid-3, ethoxy-, ethyl ester	59
1,4-Cyclohexadiene	86
Propane-1,1-dimetoxy	72
2-Butanone-3-methyl	80
Propanoic acid ethyl ester	74
Disulfide dimethyl	98
2,2-Dimethoxybutane	74
Toluene	95
Propane,1,1 dimetoxy-2-methyl	64
2,4-Octadiene	90-93
Butanoic acid, ethyl ester	93
Benzene 1,3 dimethyl / p-Xylene	90
Pentanoic acid ethyl ester	93
Hexanal dimethyl acetal	72
Tetradeconic acid, ethyl ester	91-95
Ethyl 9-hexadecanoate	95
Hexadeconic acid, ethyl ester	97

Analytt	% prob
3-Heptanone-5-methyl	96
Trans-2-(2-Pentenyl)-furan	94
2,5-Furandione, 3-methyl-	91
Propane,1,1,3,3 tetrametoxy-	64
Cyclopentanepropanol-2-methylene	72
Benzenaldehyde dimethyl acetal	93
2-Nonanone	64
Octanal dimethyl acetal	72
Octanoic acid, ethyl ester	90
Nonanal dimethyl acetal	78
Decanoic acid, ethyl ester	94
Pentadecane	97
Ethyl trideconate	86
Pentadecane ,2,6,10,14 tetramethyl	99
Heptadecane	89
Ethyl tridecanoat	90
Methyl tetradecanoat	95

Personel resources in the lipidplatform

- Marit Aursand (Research director, Prof II NTNU, PhD)
  - NMR
- Inger Beate Standal (Researcher, PhD stud)
  - NMR, Multivariate data analyses.
- Revilija Mozuraityte (Researcher, PhD)
  - Prooxidanter, Antioxidanter.
- Ana Carvajal (PhD-student, NTNU)
  - SPD, Prooxidanter, Haemoglobin
- Vera Kristinova (PhD stud, Univ of Brno, Chek Republic)
  - Natural antioxidants. Oxidation in stomach.
- Ivar Storrø (Group leader, PhD)
  - Lipid oxidasjon, Mathematical modelling)
- Ingrid Overein (Researcher, PhD student)
  - Lipidomics. Feeding of copepodes and fish fries.
- Laboratory personnel and master students