

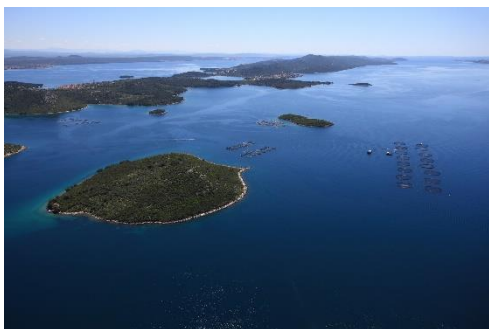
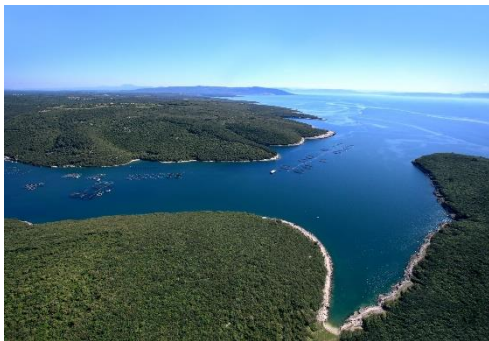


# Fish feed – Quality control

Silvia Križanac

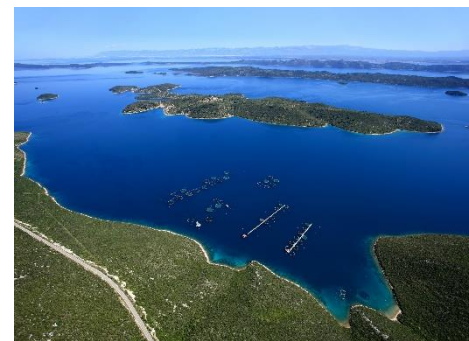
Cromaris d.d., Gaženička cesta 4b, Zadar

14. Međunarodna konferencija o akvakulturi, Vukovar, 30.3.2023



## CONTENT

- ✓ Development of formulations
- ✓ Traceability Control – Audits
- ✓ Fish Feed Control – Internal laboratory
- ✓ Raw materials and fish feed production
- ✓ Objectives and expectations



# Development of formulations

- ✓ First customized fish feed formulation in 2016. – experimental field
- ✓ Continue optimization of the recipes during year
- ✓ Development of new formula for meagre – process last more years – final Croar formula finalized in 2019.
- ✓ Our own Organic Feed Formulations – Crobo and Crosal customized according to Organic Raw Materials Market – in 2018.
- ✓ Development of feed with „antiparasitic” additive
- ✓ Continuously working on feed trails and investigating different approaches for fish feed

Least cost formulation program

The screenshot shows a software interface for feed formulation. At the top, there are tabs for 'Formula', 'Rounding', and 'Run'. Below the tabs, there are input fields for 'Formula' (CRO), 'Plant' (CROMARS), 'Price list' (Cromars 2022), 'Batch size' (1,000.00), 'Batch diff' (0.00), 'Total weight' (1,000.00), 'Tonnage' (Update), 'Price' (1,440.12), 'Cost diff' (0.00), 'Rounded price' (0.00), 'Optim. No' (430), and 'Prod. No' (0). There are also buttons for 'Current', 'Study', and 'Notes'.

Code	Raw material	Add to	Origin	% T	% int	Unit	Value %	Min	Max	Ref value
1	0002	Corn gluten meal, 65% CP		236.9	23.69	22.86				
2	0176	Soy protein concentrate, 62% CP		110.00	11.00	11.42				
3	0132	Soybean meal, 48% CP, solvent extract		110.00	11.00	11.00				
4	0140	Wheat, four		110.00	11.00	11.00				
5	1002	fish meal, not specified		90.00	9.00	9.00				
6	0916	Fish oil, Squaline, Pacific, Codmora		7.64	7.64	7.64				
7	0101	Sunflower meal, solvent extracted, def.		7.00	7.00	7.00				
8	0903	Fish meal, 70% CP, low-temperature, no		6.00	6.00	6.00				
9	0904	Repressed oil		6.00	6.00	6.00				
10	0902	Fish oil, Atlantic salmon, farmed byprod		6.00	6.00	6.00				
11	0137	Wheat gluten meal		2.00	2.00	2.44				
12	0009	L-lysine		1.00	1.00	1.00				
13	0001	Vitamin premix		0.20	0.20	0.67				
14	0040	Vitamin AD3		0.50	0.50	0.50				
15	0011	DL-Met		0.30	0.30	0.30				
16	0006	Soy lecithin		0.10	0.10	0.10				
17	0003	Choline-styC (25%), acetyl-L-homo		0.48	0.48	0.06				
18	0004	Vitamin E		0.20	0.20	0.02				

# Audits – Supplier – Traceability Control

- ✓ Constant audits of fish feed suppliers – depends on quantities by quarter
- ✓ Content of audit report:
  - ✓ Nonconformity
  - ✓ Fish feed and raw material analysis
  - ✓ Traceability – by batch of feed and raw material
  - ✓ Quality control
  - ✓ Production visit

	IZVJEŠTAJ AUDITA DOBAVLJAČA	OB 04/10
		Revizija: 3
		Datum: 01.01.2019

Naziv dobavljača:

Adresa dobavljača:

Datum provedbe audita:

Auditorski tim:

Interni audit je održan prema:

Audit su prisustvovali:

Rezultati audita:

ZAKLJUČAK INTERNOG AUDITA

Mjesto i datum

*(Ime, prezime i potpis glavnog auditora)*

Num.	Date	Comments
01/2018	10.01.2018	Minor non-compliance with the inclusion of fish oil, which is always less than the required and increased inclusion of rapeseed oil.
02/2018	16.01.2018	Inclusion of two different fish oil – FO trimmings and FO NA STD 18 – different inclusions in total according to specifications – conform. Without analysis on raw materials – fish oil 3014604 Agreed batches without guar meal – accepted.
03/2018	09.03.2018	After checking the production there were no evident non-conformities. There are problems that they are trying to solve it. The most manifest themselves in the physical characteristics of the feed – currently an objection to the oil leakage.
04/2018	15.05.2018	In collaboration with R&D department, we will continue to establish physical and nutritive parameters of feed. Reports will be prepared quarterly from both laboratories. We expect to increase the quality parameter to a higher level and unique form for each producer. Raw materials and feed have very good quality control and frequent analysis on each batch. Everything according to specification and certification.
05/2018	28.06.2018	Inclusion of raw material – minor non-conformity on traceability – differences between 0,6 – 1%.
07/2018	10.07.2018	Raw materials meet the required specifications. There are some deviations but not significant, also everything conforms during production check. here is no deviation in production. A new specification was created that changed the 9mm size parameters.

# Traceability Control

✓ Technical specification, certification and analysis

## IDENTIFICATION:

- Product name: Krill™ Antarctic Krill Meal
- Product number: 43010000 (25 kg)  
43011000 (500 kg)
- Batch number: 10-02-2018
- Production date: 26/DEC/2017-05/JAN/2018

## DESCRIPTION:

- Composition: Dry krill meal
- Additives: FEQ 500 (Ethoxyquin)
- Country of origin: Norway, Produced on board F/T Saga Sea and F/V Antarctic Sea

## ANALYTICAL SPECIFICATION:

Parameter	Method	Actual value	Limit	Unit
Appearance	Visual	Orange powder/meal	Orange powder/meal	
<b>Composition</b>				
Fat	Bligh & Dyer (A56)	28	≥15	g/100 g
Crude Protein	ISO 16634-1	54	≥54	g/100 g
Ash	ISO 5984	10	≤13	g/100 g
- incl. Salt	AOAC 937.09	2,6	≤4,5	g/100 g
Moisture	ISO 6496	7	7±2	g/100 g
Astaxanthin	NOFIMA method (A101)	119	≥80	mg/kg
Sum Fat & Protein		83	≥ 77	g/100 g
<b>Freshness</b>				
Total Volatile Nitrogen	AOAC 920.03	<0.1	≤0.1	g/100 g
Cadaverine	NOFIMA method (A55)	<0.1	<0.1	g/kg
Histamine	NOFIMA method (A55)	<0.1	<0.1	g/kg
Peroxide	AOCs Cd 8b-90	4	≤20	meq
Meat Bone Meal	2009/152/EC	Not present	Not present	g/100 g
<b>Mineral level</b>				
Fluorine	NOFIMA method (A98)	1700	≤2500	mg/kg
<b>Additives</b>				
Ethoxyquin	AOAC 963.07	143	≥120	mg/kg
<b>Microbiology</b>				
Total plate count	AFNOR 3M 1/1-9/89	100	≤100 000	cfu/g
Salmonella negative	NordVal 023	Negative	Negative	1 sample
Enterobacteriaceae	ISO 21528-2	<10	≤ 300	cfu/g

**Analytical testing:** The analytical test data reported in this CoA is produced by Nofima AS, Bergen, Norway.

**Application:** For animal nutrition: To be used in formulated diets. The product contains phospholipid bound Omega -3 fatty acids, high quality marine protein and Astaxanthin. The product is added antioxidant FEQ 500 (Ethoxyquin).

**Storage:** The product is best kept at temperatures <25°C during storage in its original packaging container.

**Best before date:** 2 years from production date when stored in unopened packaging at recommended storage conditions.

## RAPPORT D'ESSAI FINAL DRY FISH - FARINE DE POISSON SPR68

Date réception client : 28/05/2018  
Date fabrication : 22/05/2018  
N° lot client : 3015616  
Fournisseur :  
N° lot fournisseur :  
Tonnage :  
DLUO :

Demandeur :  
N° commande :  
N° client :  
N° optim :  
N° étude :  
Réf. commerciale :  
Tiers :

Date réception labo : 30/05/2018

Masse brute (g):

Observations :

Commentaires :

## ANALYSES CHIM

Détermination	Rés/brut	Rés/sec	Incerti
HUMIDITE	6,9		0,1
Méthode interne EAU-H 14/02 adaptée du Règlement CE 152/2009 du 27-01-2009 (103°C/4h) - SN	g/100g		g/10
CENDRES BRUTES	16,2	17,4	0,1
Méthode interne CEND-H 13/02 adaptée du Règlement CE 152/2009 du 27-01-2009 - SN	g/100g	g/100g MS	g/10
PROTEINES KJELDAHL (Nv6.25)	66,7	71,6	2,1
Méthode interne PROT-H 14/01 adaptée du Règlement CE 152/2009 du 27-01-2009 - SN	g/100g	g/100g MS	g/10
CELLULOSE BRUTE (avec prétraitement)	0,9	1,0	0,1
Méthode interne - CELLFIBR 16 - CT	g/100g	g/100g MS	g/10
MATIERES GRASSES BRUTES TOTALES	9,6	10,3	0,1
Méthode interne - MGRA-H - Procédé B - SN	g/100g	g/100g MS	g/10

Dénomination : HUILE DE POISSON  
T° à réception (°C) : Température ambiante  
Masse (g) : 126

Lot : 3015666  
Fabriqué le : 07/03/21

Détermination	Méthode	Unité	Résultat
<b>CHIMIE</b> N° d'échantillon : 2 593 009 - _AA00-180309-C-89			
Plomb	NF EN 15763*	mg/kg	<-0.01
Mercurure	NF EN 15763*	mg/kg	<-0.01
Cadmium	NF EN 15763*	mg/kg	<-0.01
Arsenic	NF EN 15763*	mg/kg	9.99
Minéralisation digestion sous pression	NF EN 13805*		-
Métaux lourds	calcul		
Plomb mg/kg		mg/Kg 12% Humidité	<-0.01
Mercurure mg/kg		mg/Kg 12% Humidité	<-0.01
Arsenic mg/kg		mg/Kg 12% Humidité	8.81
Cadmium mg/kg		mg/Kg 12% Humidité	<-0.01
Teneur en eau	M160 (Gravimétrie)	g/100g	<-0.2

Dénomination : MATIERE PREMIERE SPC 62  
Conditionnement : Sachet plastique  
T° à réception (°C) : Température ambiante  
Masse (g) : 180

Lot : 3016900  
Fabriqué le : 08/06/20

**CHIMIE**  
N° d'échantillon : 2 709 820 - \_AA00-180614-C-60

Détermination	Méthode	Unité	Résultat
Mycotoxines - CT	Méthode interne		
Désoxynivalénol µg/kg		µg/kg	<125
Aflatoxine B1 µg/kg		µg/kg	4.4
Aflatoxine B2 µg/kg		µg/kg	0.7
Aflatoxine G1 µg/kg		µg/kg	<0.1
Aflatoxine G2 µg/kg		µg/kg	<0.1
Aflatoxines B et G		µg/kg	5.1
Ochratoxine µg/kg		µg/kg	<0.5
Fumonisine B1 µg/kg		µg/kg	<50
Fumonisine B2 µg/kg		µg/kg	<50
Zéaralénone µg/kg		µg/kg	<6

## Fish feed control / Sampling

- ✓ Sampling of fish feed – for nutritional and physical control
- ✓ Sampling method – collect feed samples from **different bags** of the **same batch** to have referent sample of batch.
- ✓ Minimum quantity for analysis is 500 grams per sample batch.
- ✓ During sampling is **REQUIRED** to enclose label of the batch.



## Fish Feed Control / Analyses

- ✓ Measuring nutritional parameters
  - ✓ Control of labeling – crude protein, crude lipid, crude fiber, ash and moisture (NIR spectrophotometer)
- ✓ All samples bigger than 3 mm are grinding before measuring on NIR.
- ✓ New update of NIR – advance course for technical calibration the NIR
- ✓ Measuring physical parameters:
- ✓ Vibratory sieve shaker AS 200 digit with new sieves – separating dust, broken and different sizes



# Fish Feed Control / Analyses

- ✓ Measuring physical parameters
  - ✓ Control of agreed technical parameters – pellet size, dust, broken pellets, floatability, oil leakage, durability and hardness
- ✓ Holmen NHP 100 – new device for measuring durability / hardness of the pellets – PDI %
- ✓ Floatability – measuring of each batch in the laboratory according to stable salinity and temperature of the water



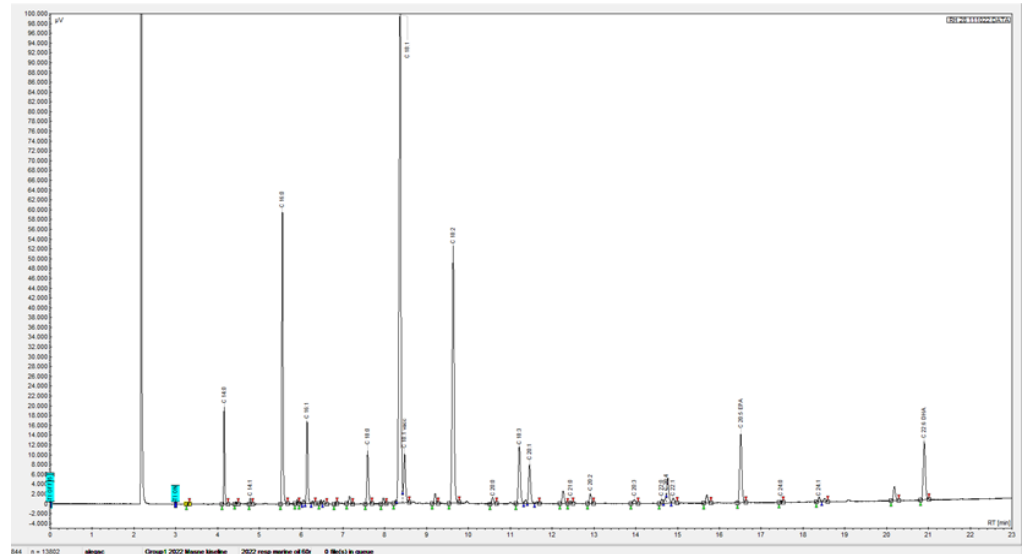


# Chemical analysis on fish feed – FATTY ACID

- ✓ Essential to control EPA and DHA – omega-3 and omega-6 FA.
- ✓ Implemented in internal laboratory

Method for determination of fatty acids:

- ✓ Oil preparation by Soxlet-type extraction in petrol ether after acid hydrolysis, derivatization by rapid transmethylation with KOH in methanol, isooctane layer transferred to vial and separated on Rtx-2330 capillary column.
- ✓ Instrumentation: Gas chromatography system with FID detection, Scion 436-GC.



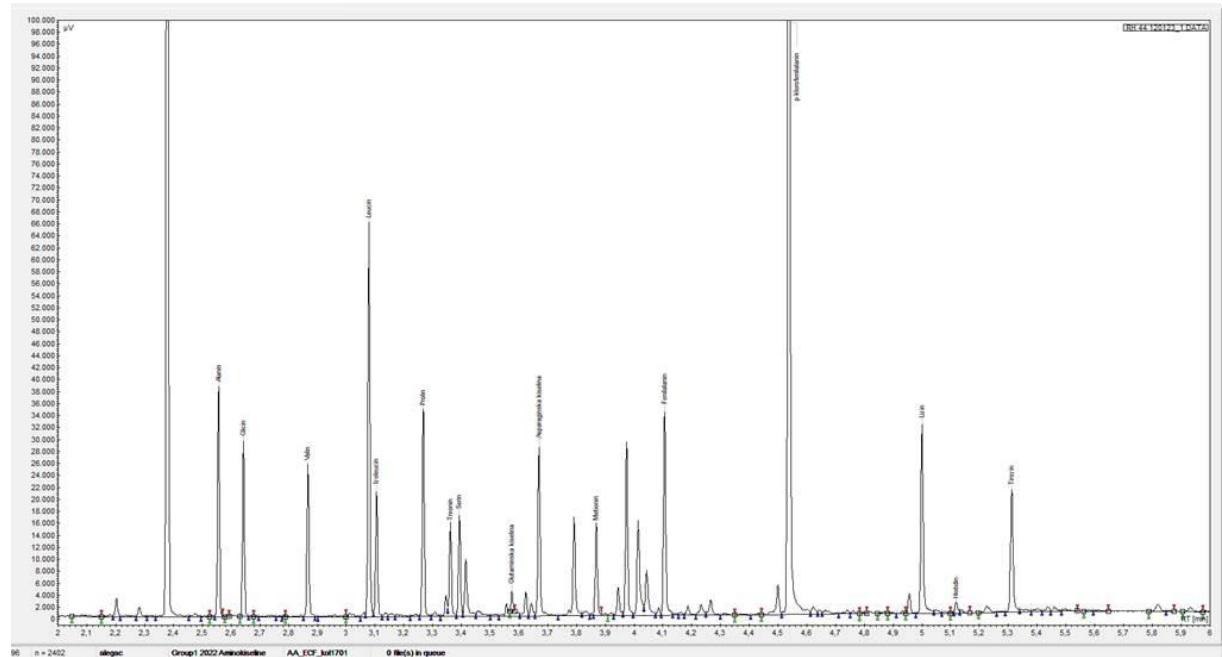
# Chemical analysis on fish feed – AMINO ACID

✓ Essential to control limited AA Lys and Met.  
Implemented in internal laboratory

✓ Method for determination of amino acids:

Acid hydrolysis at 110°C for 24-hour,  
derivatization with ethyl chloroformate at pH 2,  
chloroform layer transferred to vial and separated  
on Rtx-1701 capillary column.

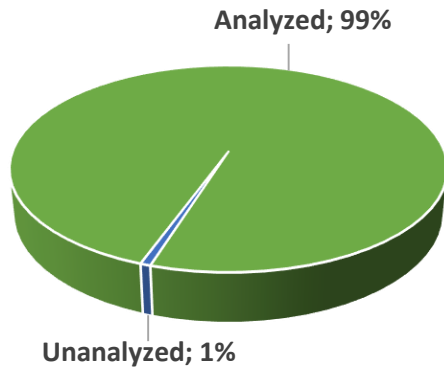
✓ Instrumentation: Gas chromatography system  
with FID detection, Scion 436-GC.



# Review of analyzed batches

- ✓ The batch analysis is intensively implemented throughout the year

Analysis of fish feed in 2022



- ✓ Increased number of analyzed batch significant for all suppliers

% ANALYZED BATCH	Supplier 1	Supplier 2	Supplier 3	Supplier 4
2016	89%	70%	49%	/
2017	90%	85%	87%	/
2018	94%	88%	90%	/
2019	100%	92%	97%	93%
2020	87%	82%	50%	87%
2021	100%	99%	95%	95%
2022	/	99%	99%	99%

- ✓ Review of analyzed batch through years

Year	N. Input batch	Analyzed	% Analyzed batch	% Nonconformity batch
2016	1.012	752	70,8%	21,9%
2017	916	796	86,9%	14,8%
2018	1.147	1.031	89,9%	4,8%
2019	1.021	955	93,5%	5,1%
2020	781	639	81,8%	6,3%
2021	939	913	97,2%	19,2%
2022	1.219	1.211	99,3%	2,9%

- ✓ Development of program monitoring the results of measured parameters in SAP
- ✓ Development for credit note calculation for nonconformities
- ✓ Creating reports and interfaces for faster and easier control monitoring of analyzed batch
- ✓ Objectives for future:
  - ✓ Automatic updating results from NIR to SAP / in progress
  - ✓ Purchasing equipment – fast control oxidative changes on feed
  - ✓ Device for measuring oil leakage
  - ✓ DORIS test

# Raw materials in fish feed production

- ✓ Monitoring of price movements – Holtermann index
- ✓ Impact of RMC on fish feed cost
- ✓ Research RM – market situations
- ✓ Technical and nutritional specification
- ✓ Possibilities in purchasing RM – sourcing of RM
  - ✓ Fish meal and oil – traders Europe, producers – South America, Africa...
  - ✓ Soy and soy protein concentrate – origin Brazil, traders Europe
  - ✓ Purchasing RM through traders and producers – safety risk
- ✓ Calculation of prices fish oil – EPA+DHA
  - ✓ Latest price for EPA+DHA on market **+160 USD** per percentage point per mt oil
  - ✓ Fish oil Peru – 26% EPA+DHA, better quality
  - ✓ Fish oil Scandinavia – 18% EPA+DHA, higher content of contaminant, refining is obligatory
  - ✓ Standardization of fish oil specification – mixed oil according to defined specification from buyer (fish feed producer)

Guar protein Soybean meal Wheat gluten



Fish oil

Rapeseed oil



# Raw materials in fish feed production

## ✓ Raw material in fish feed production divided in three groups:

- ✓ Macro ingredients: Marine ingredients and vegetable proteins (inclusions more than 1%)
- ✓ Micro ingredients: Additives and premix
- ✓ Liquid – marine or vegetable oils

## ✓ Category group I.

- ✓ Fish meal and fish oil – continuous presence on the market – follow physical movements and price

## ✓ Category group II.

- ✓ Soy protein concentrate – FM replacer, **issue NGMO is falling**
- ✓ Guar protein and wheat gluten – decreasing in fish feed production
- ✓ Corn gluten, Soybean meal, Wheat, Rapeseed oil, Sunflower meal – commodity
- ✓ Land animal protein – increasing demand for fish feed
- ✓ New raw material: macroalge oil, bacterial single cell protein, camelina oil, insect meal...

## ✓ Category group III.

- ✓ Micro ingredients – Vitamins, minerals and functional additives



# Raw materials / Holtermann index

✓ Raw material prices from 2015 till today

✓ Fish oil:

- 2021/2022 = +90%

- 2022/today = +35%

✓ Rapeseed oil:

- 2021/2022 = +33%

- 2022/today = -30%

✓ Fish meal:

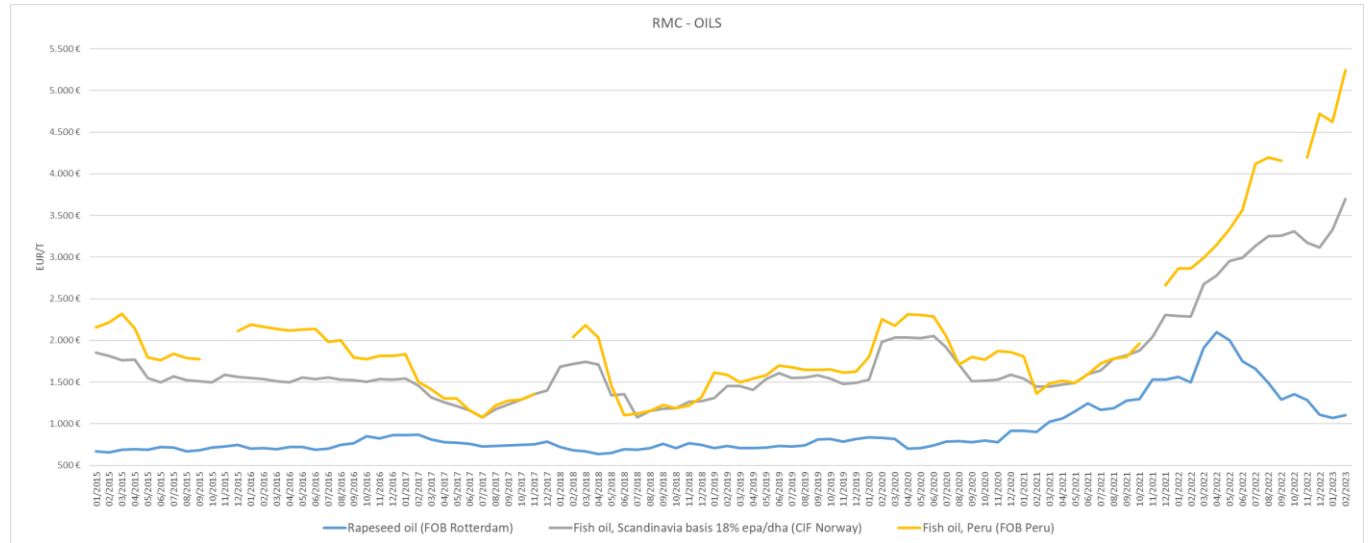
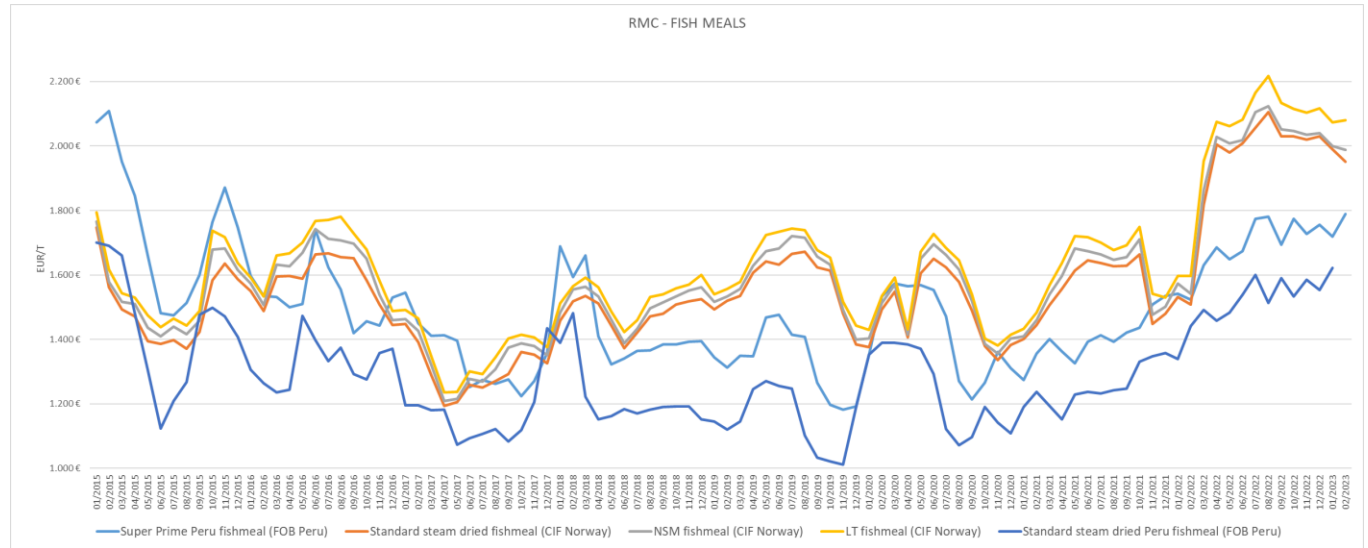
- 2021/2022 = +23%

- 2022/today = +4%

✓ Vegetable proteins

- 2021/2022 = +36%

- 2022/today = -14%



## Conclusion and further aims

- ✓ Constant improvement in raw material database
- ✓ New calibration packages for RM on NIR
- ✓ Research of new raw materials
- ✓ Optimization and improvements in formulations
- ✓ Education and courses related with production and control of fish feed







**THANK YOU**