Decoding lipolysis through individual free fatty FT-MIR model: Enhancing predictive accuracy through real-sample enrichment and advanced algorithms

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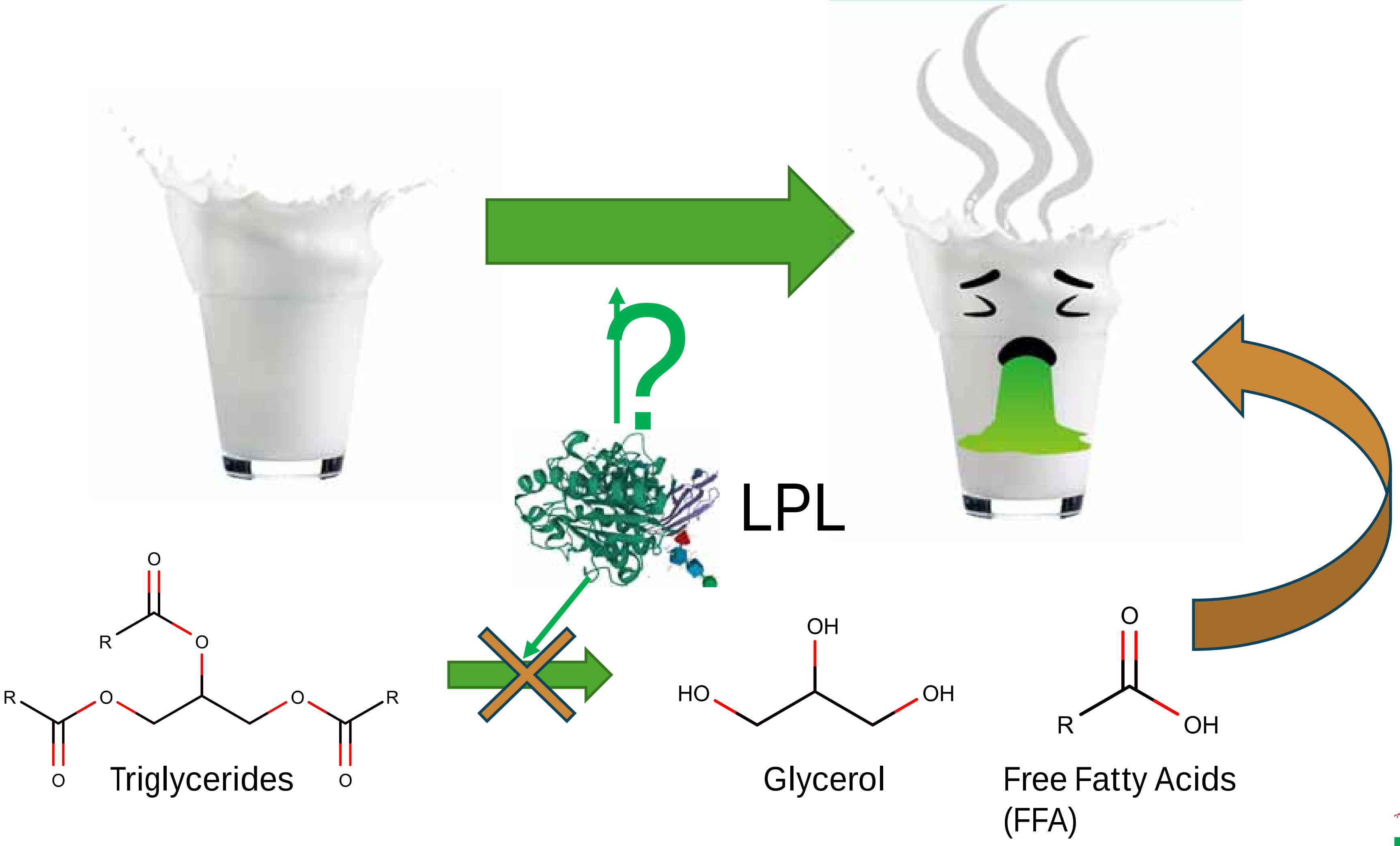
What is lipolysis



LPL

Triglycerides

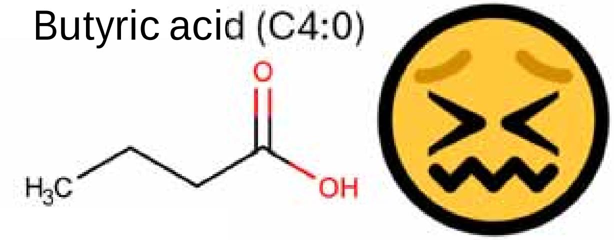
Glycerol

Free Fatty Acids (FFA)

Lipolysis why it’s bad ?

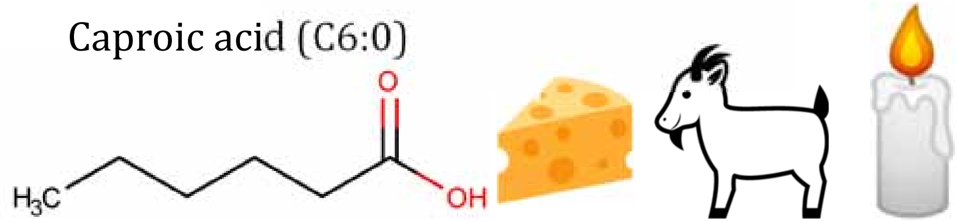
Effect on the taste quality of dairy products

Butyric acid (C4:0)



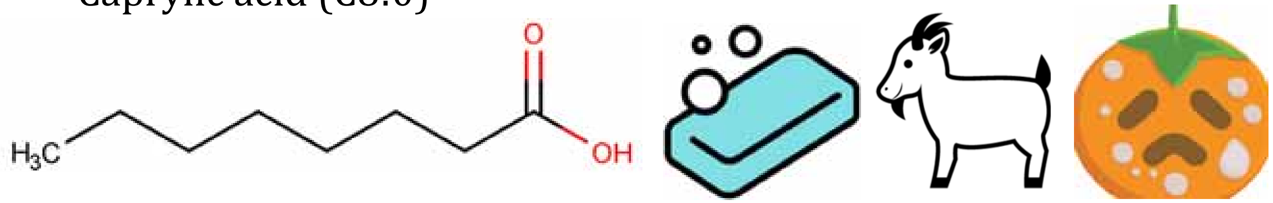
Rancid taste

Caproic acid (C6:0)

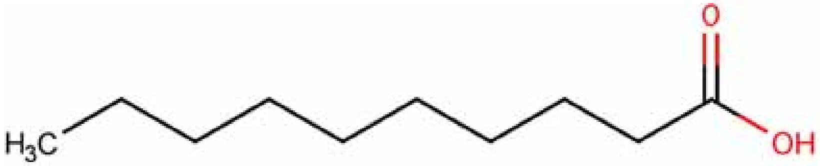


Rancid taste

Caprylic acid (C8:0)



Capric acid (C10:0)



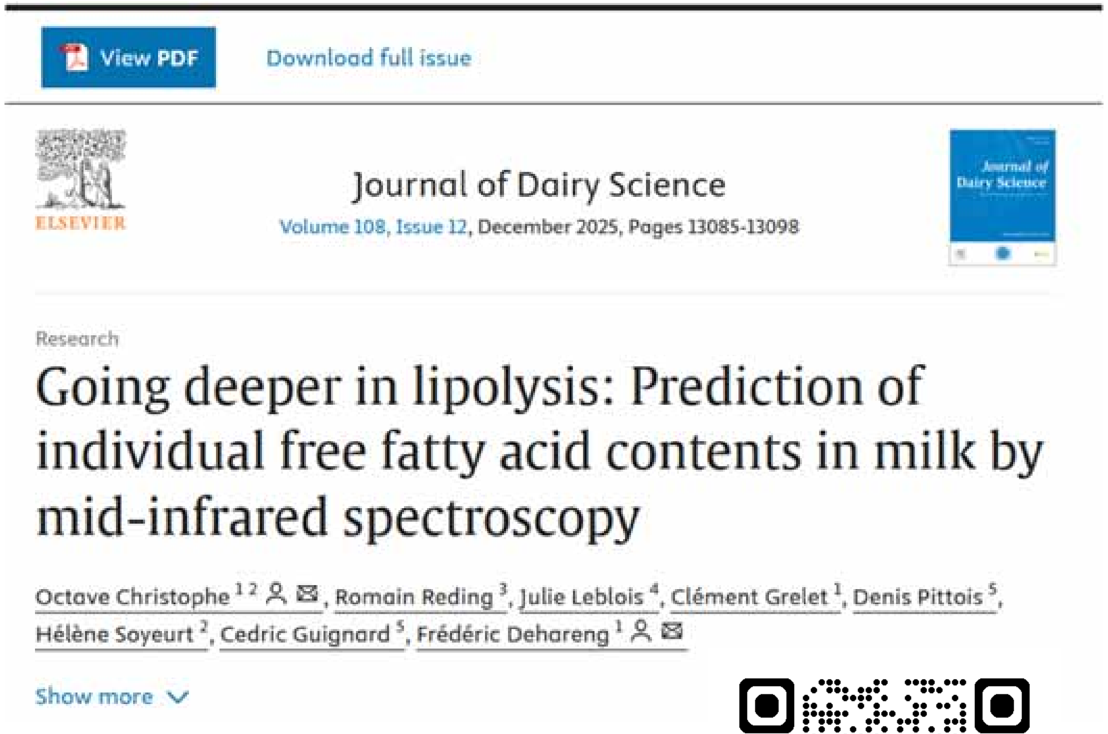
Rancid taste

Other FFA → effect on the milk transformation

Inhibition of bacteria C12:0 → C18:3

→ Each FFA could have different effect on the milk taste

Previous work



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Research

Going deeper in lipolysis: Prediction of individual free fatty acid contents in milk by mid-infrared spectroscopy

Octave Christophe [1,2], Romain Reding [3], Julie Leblois [4], Clément Grelet [1], Denis Pittois [5], Hélène Soyeurt [2], Cedric Guignard [5], Frédéric Dehareng [1]

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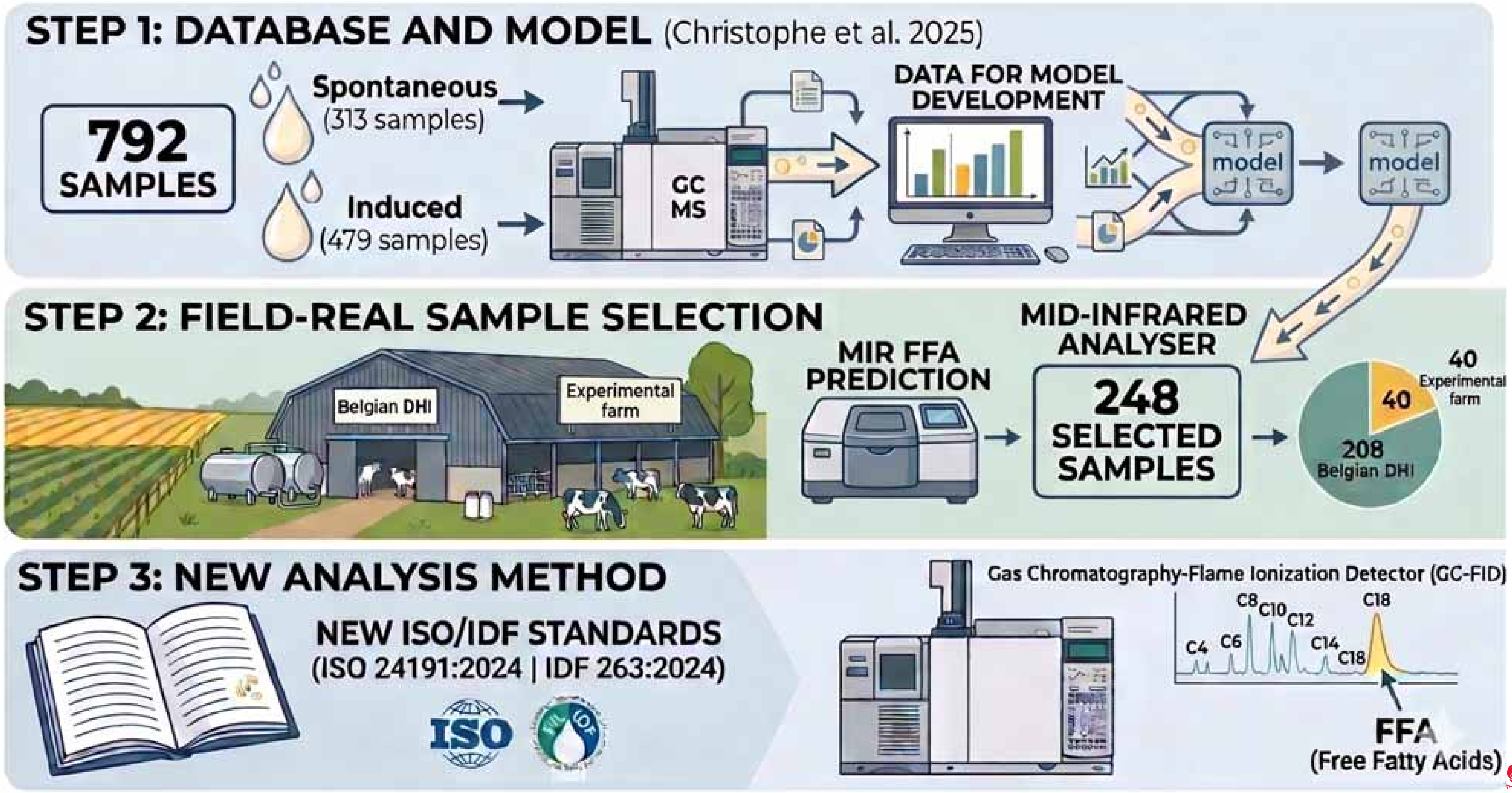
794 samples: samples coming from spontaneous and induced lipolysis

Cons:

- The models was not developed using a method certified by ISO/IDF
- Models not include a lot of real-field samples
- Only PLS algorithm is used

Material et methods: ISO/IDF analysis



@NanoBanana

Material et methods: ISO/IDF analysis

Samples from DHI

FT-MIR Analysis

High FFA

Selection of samples
throught FFA prediction

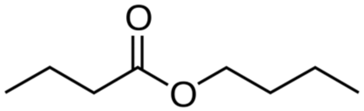
Lab

Extraction

3 extraction with 50/50
Diethylether/n-hexane
SPE

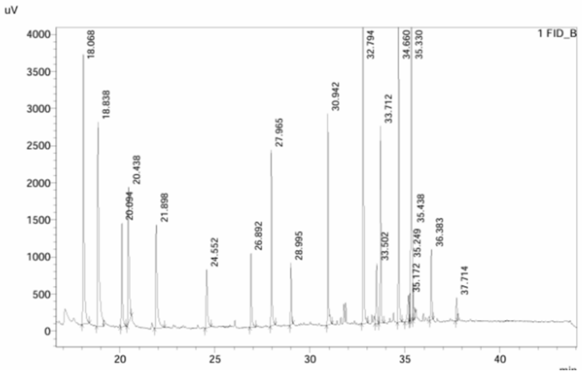
Derivatization

BF3 + butanol
FFA → FABE



Washing with
water/Methano

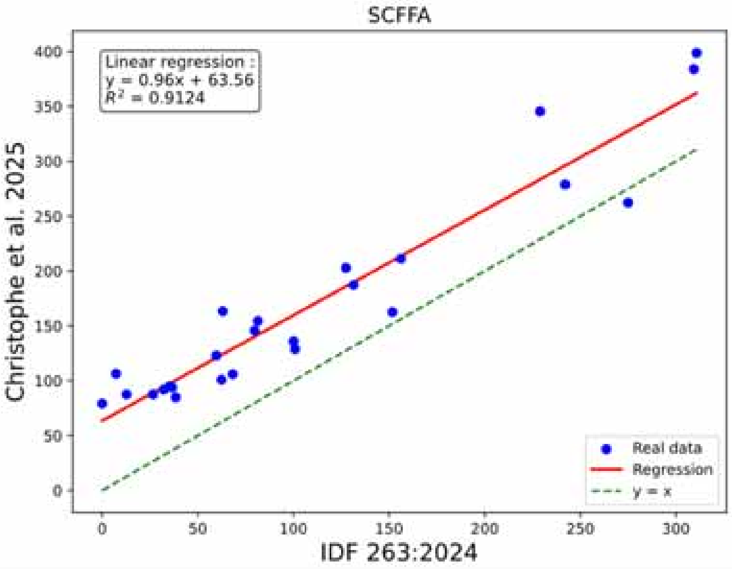
GC-FID analysis

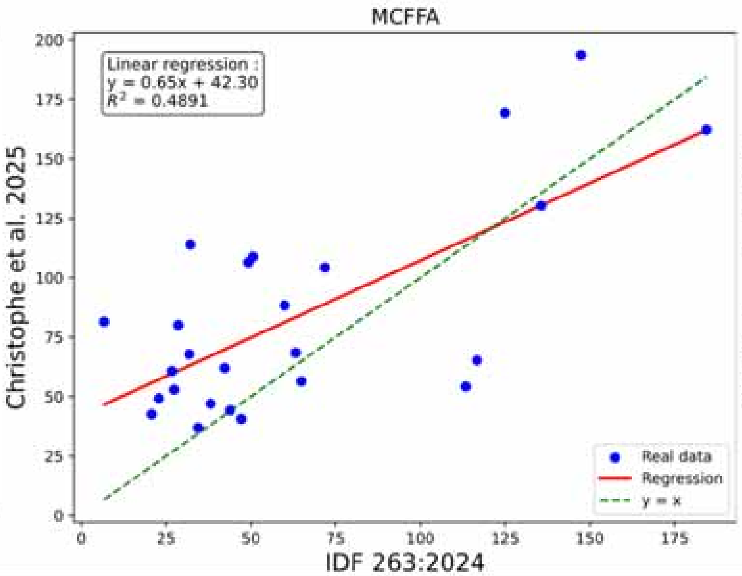


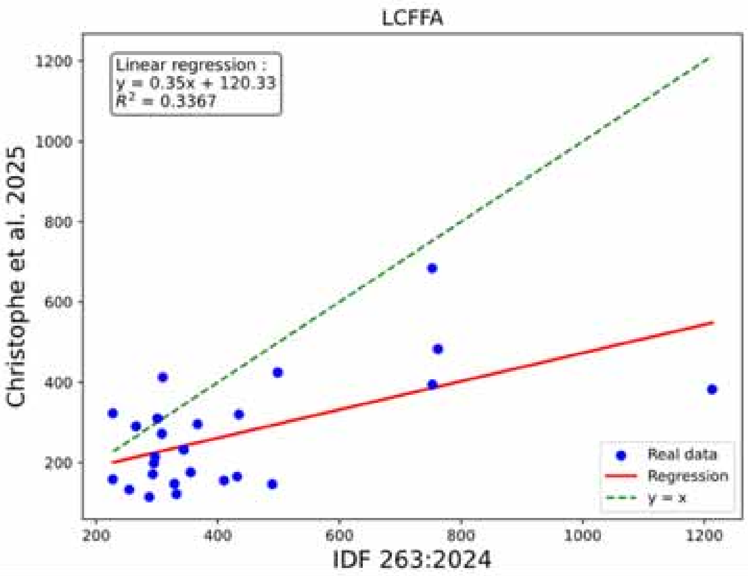
Material et methods: Comparison between method

25 samples with high lipolysis content are used to compare method from Christophe et al 2025 and ISO/IDF reference analysis

	C4	C6	C8	C10	C12	C14	C16	C18	C18_1	C18_2	SCFFA	MCFFA	LCFFA
Slope	0.94	1.00	0.92	0.82	0.61	0.65	0.75	0.73	0.52	0.88	0.96	0.65	0.35
Bias	30.08	17.73	16.11	16.82	13.71	11.24	-9.88	7.35	23.30	13.31	63.56	42.30	120.33
r2	0.91	0.93	0.80	0.74	0.38	0.53	0.47	0.59	0.46	0.25	0.91	0.49	0.34

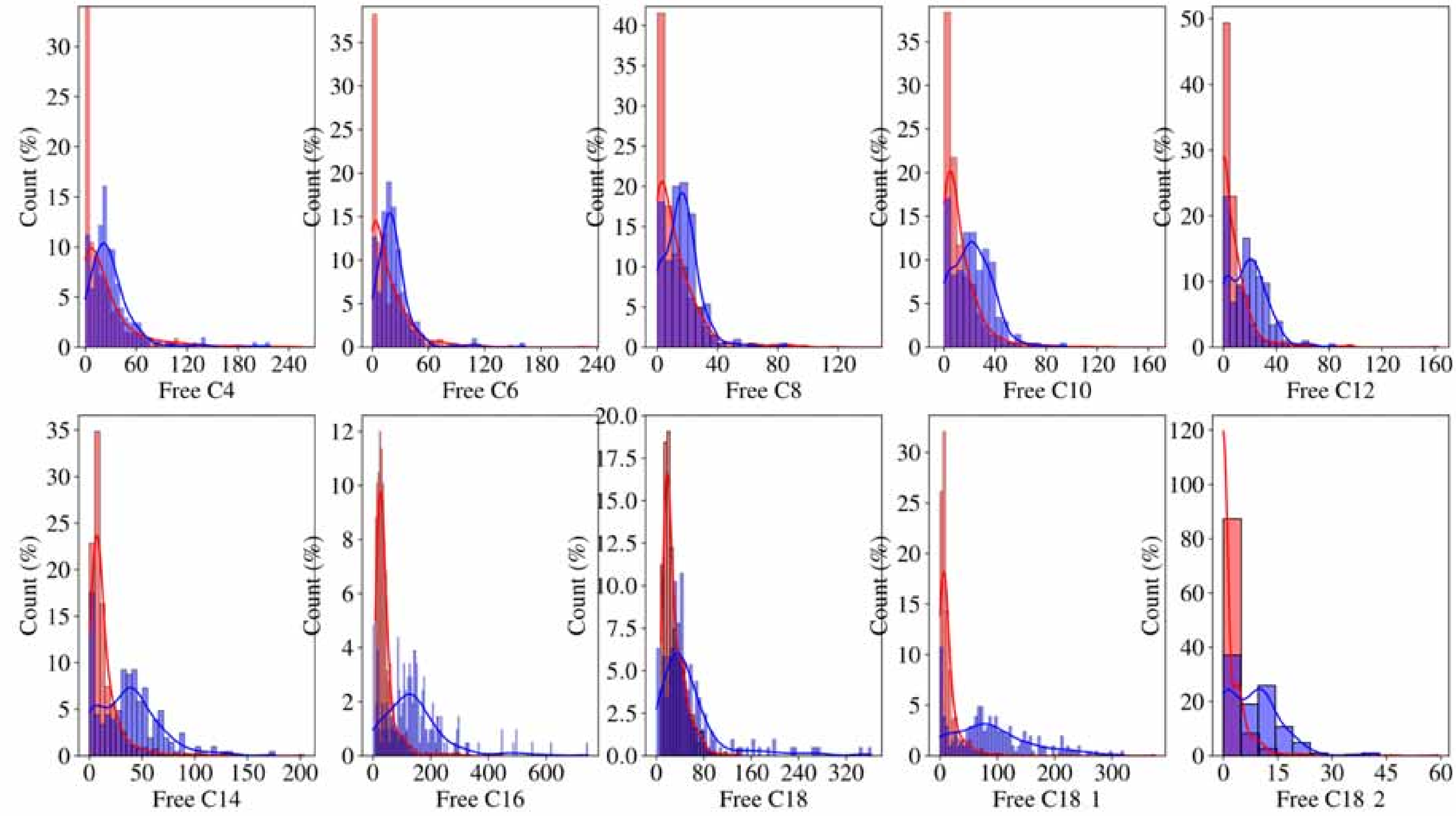






Results: data comparison

Data Distribution

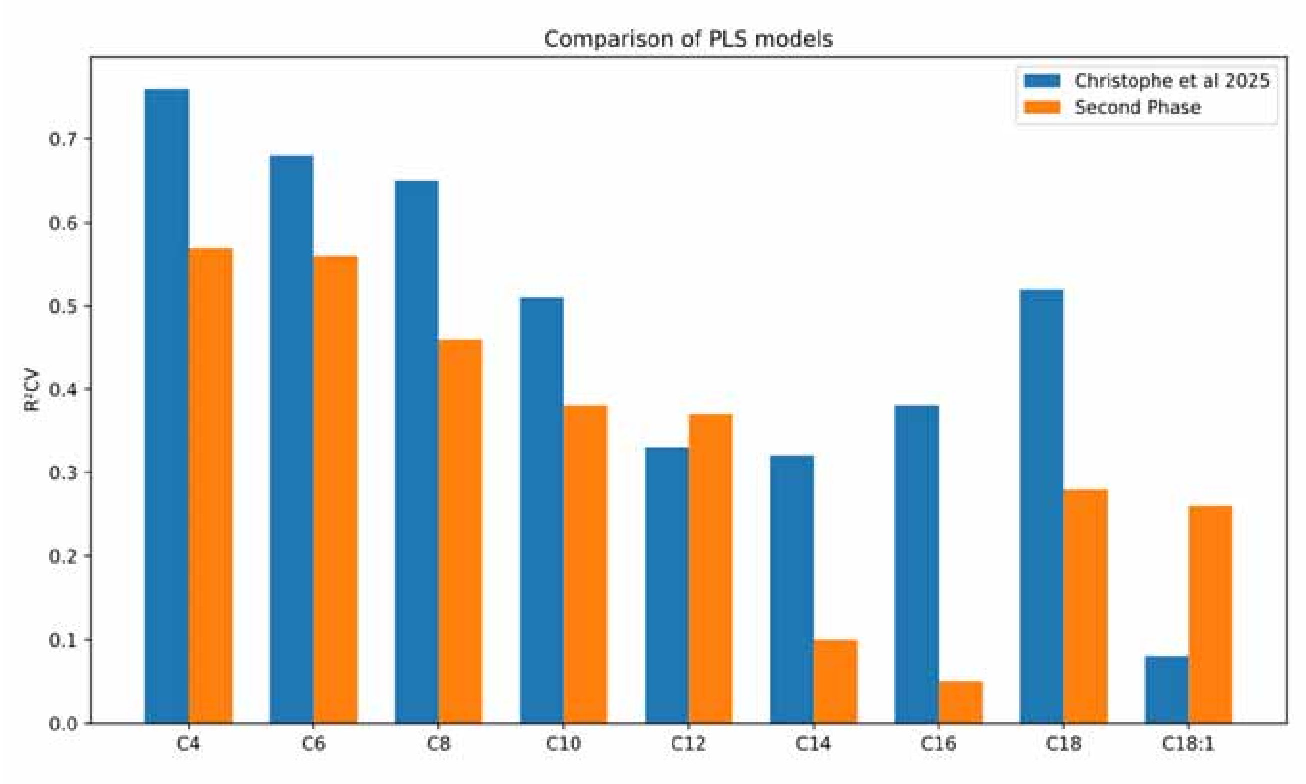


Data from Christophe et al 2025

Second phase Real Field data

Results: Model only ISO/IDF references

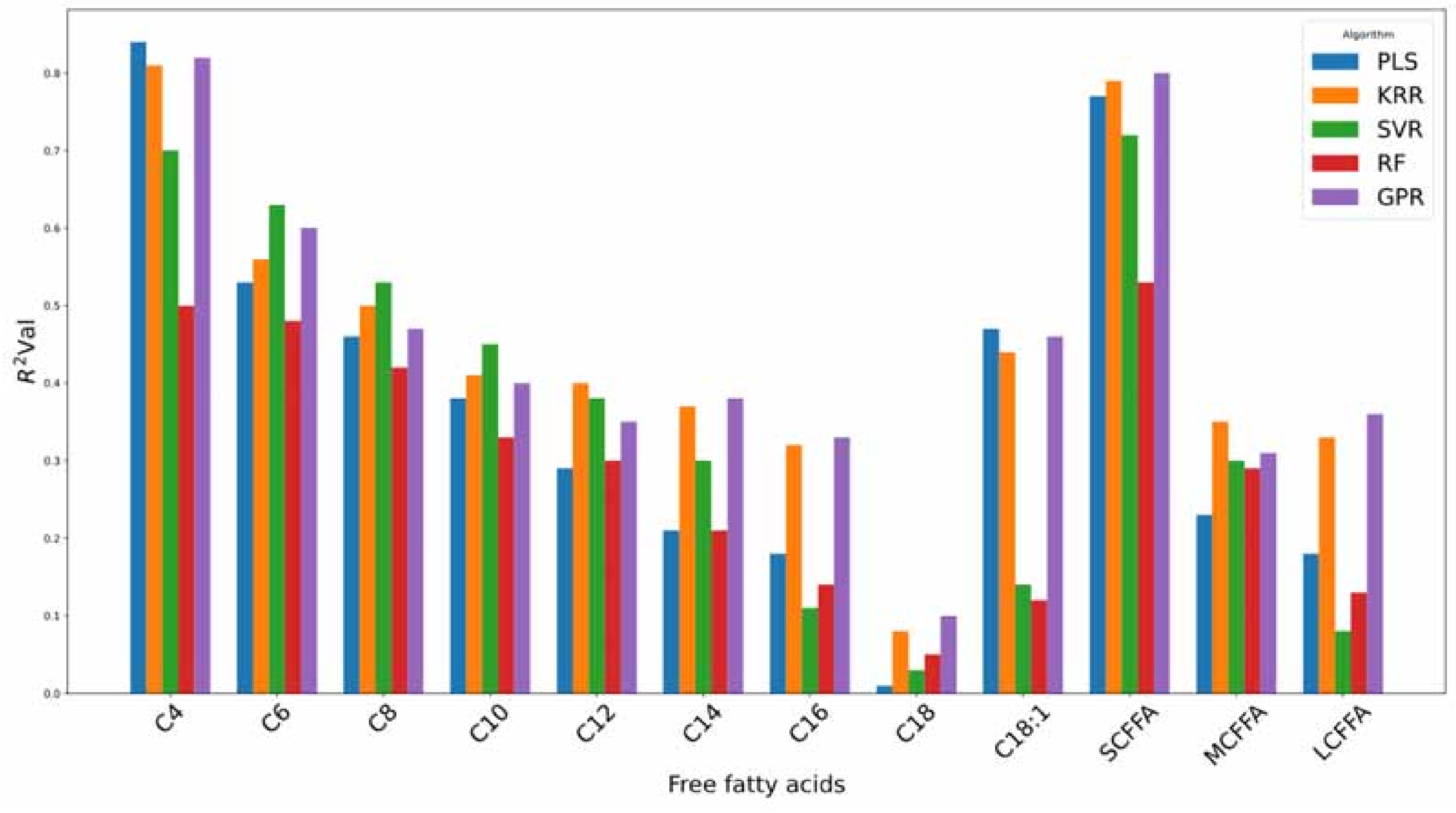
Comparison of PLS models from Christophe et al 2025 and models from field real samples with ISO/IDF quantification



- Good variability of the data but second phase include 248 samples and 792 from Christophe et al
- SCFFA expose Comparable performance
- Long chain FFA very bad for second phase especially C14 and C16
- Improvment of C18:1 model

Results: Global quantitatif Models

Complete Dataset: 1040 samples Cal=832 Val= 208 (60% first Phase , 40% second Phase)
Different algorithms used : Partial least squares regression (**PLS)**, Kernel Ridge regression (**KRR**), Random Forest (**RF**), Support vector regression (**SVR**), Gaussian process regression (**GPR**)



→ No type of algorithm consistently outperforms the other

→ Random Forest overfitting

→ SVR does not work for LCFFA

Results: Global quantitatif Models

Selected algorithm and performance through Cross-validation and Validation

	C4	C6	C8	C10	C12	C14	C16	C18	C18_1	C18_2	SCFFA	MCFFA	LCFFA
Algo	PLS	SVR	SVR	KRR	KRR	GPR	GPR	GPR	PLS	KRR	GPR	KRR	GPR
R²CV	0.71	0.68	0.64	0.57	0.49	0.41	0.44	0.43	0.42	0.36	0.72	0.50	0.45
R²Val	0.84	0.63	0.53	0.41	0.40	0.38	0.33	0.10	0.47	0.27	0.80	0.35	0.36
RMSECV	16.72	10.29	7.22	9.53	8.26	13.77	37.65	14.71	26.24	3.65	34.42	30.80	81.50
RMSEval	17.84	11.79	8.61	10.83	8.77	15.10	63.25	33.82	32.11	4.62	32.08	32.67	117.65
RPDCV	1.87	1.77	1.68	1.52	1.40	1.30	1.33	1.33	1.31	1.25	1.89	1.41	1.34
RPDval	2.49	1.65	1.46	1.30	1.30	1.27	1.22	1.05	1.37	1.17	2.22	1.24	1.25

Compare to different models of Christophe et al. 2025

→ Improve performance of C18:1
→ RMSE C16 and C18 very increased !

→ Same performances achieved for SCFFA
→ Better performances for MCFFA
→ Bad performances for LCFFA

Results: Classification Models

Classification perfomance on the 208 validation samples → Creation of 2 classes: “Good” and “Rancid”

	C4	C6	C8	C10	C12	C14	C16	C18	C18_1	SCFFA	MCFFA	LCFFA
Algorithm	PLS	SVM	SVM	GPC	GPC	GPC	GPC	PLS	GPC	PLS	GPC	GPC
%Rancid	37%	41%	48%	15%	9%	9%	16%	13%	5%	39%	11%	12%
Threshold (mg/L)	27.5	16	8.3	27.6	26.7	48	118	58.4	137	51.8	102.3	313.4
Accuracy	84%	86%	85%	89%	91%	92%	92%	90%	95%	85%	92%	88%
Sensitivity	94%	87%	82%	98%	98%	99%	96%	99%	100%	90%	99%	99%
Selectivity	63%	84%	89%	35%	21%	16%	70%	26%	9%	78%	32%	4%

Classification threshold for flavor defects based on FFA

→From Kintner et al. 1965 https://doi.org/10.3168/jds.S0022-0302(65)88529-0

→ The algorithm employed could enhance the selectivity for MCFFA and LCFFA

→ Very old threshold ? Reference change ?

Conclusion

Take home message

- This study underscores the importance of harmonizing reference methods
- Reminds us that a method should be viewed as a tool for interpretation rather than an absolute truth

- The use of different method influence the performance and increased the robustness of the models

- Improvement of the models robustness by including real lypolized samples

- The use of new algorithm enhance the performance but one algorithm do not have the significant impact compare to the other

- Unless in classification step where an algorithm could really enhance selectivity

Outlook

- Update the threshold as the method have changed !
- Improve the dataset with lypolised sample coming from different countries, feeding system and milking system

Any questions ??

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