Aplicação de técnica de metabolômica ao estudo de alimentos

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FORMAÇÃO ACADÊMICA

- 1998: Bacharel em Química Industrial pela UFPB

- 1993: Mestrado em Ciência e Tecnologia de Alimentos (UFPB)

- 2000: Doutorado em Tecnologia de Alimentos (Unicamp)

- 2001: Pesquisador Embrapa Agroindústria Tropical (Fortaleza-CE)

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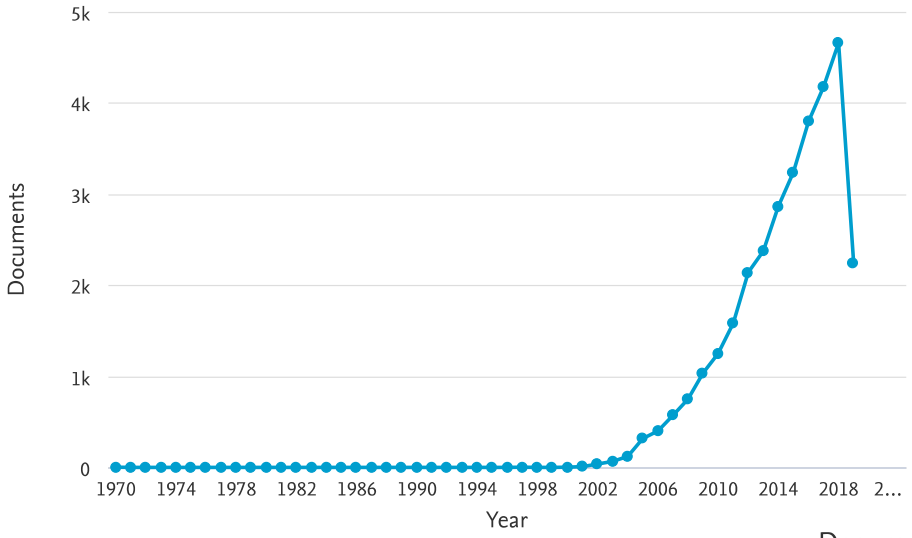
I-3587-2012

metabolomic

Scopus junho 2019

31,643 document results

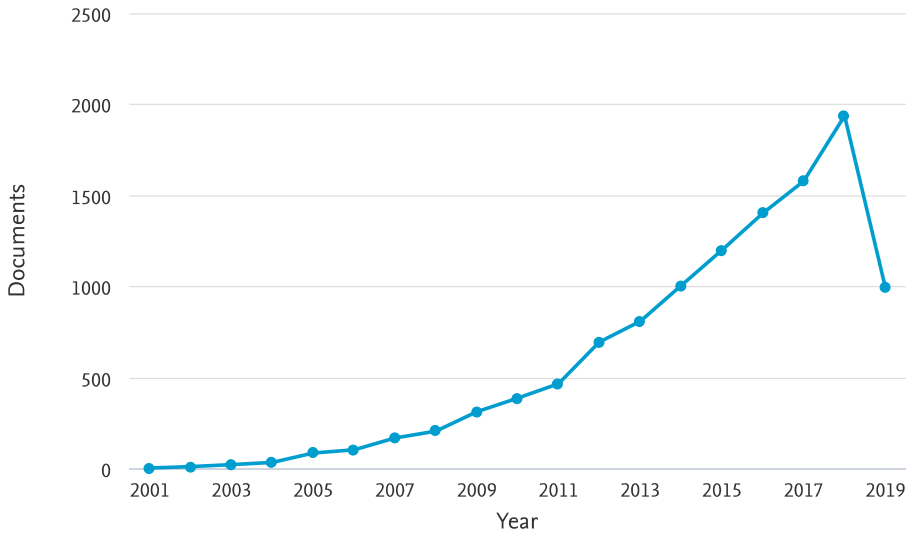
Documents by year



metabolomic AND food

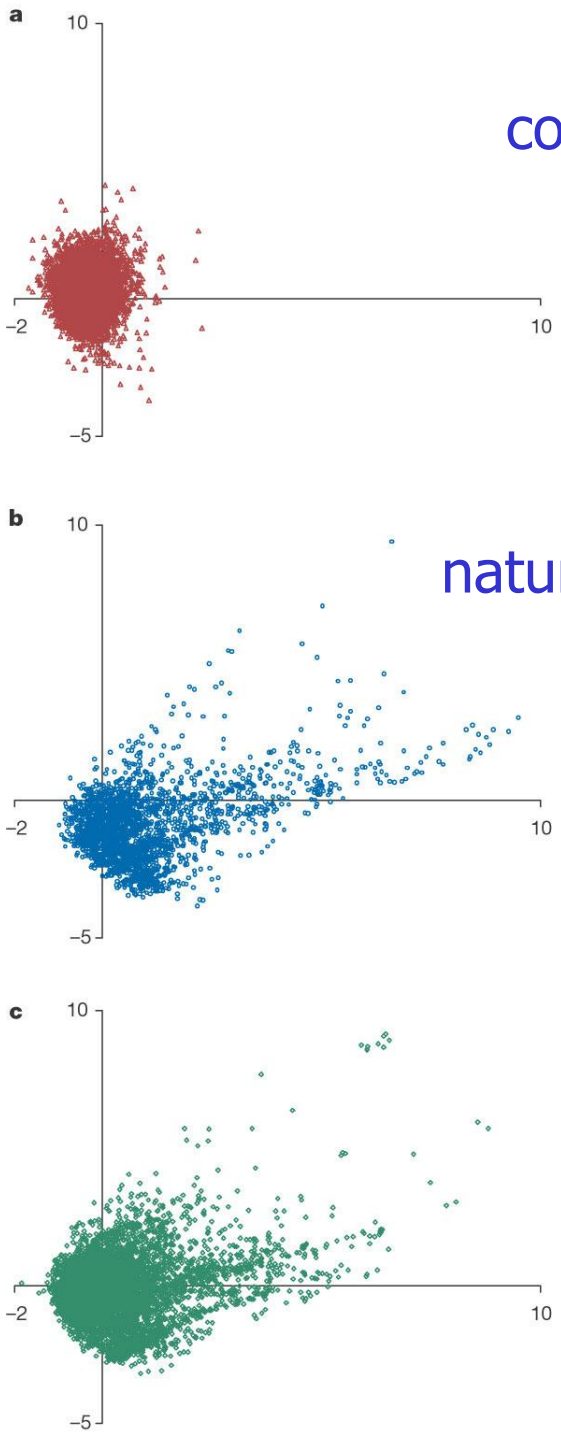
11,401 document results

Documents by year



Espaço Químico

basis of a variety of molecular properties

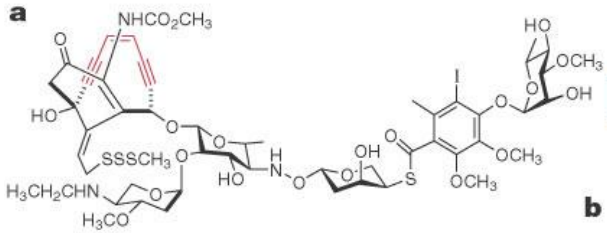


combinatorial chemistry

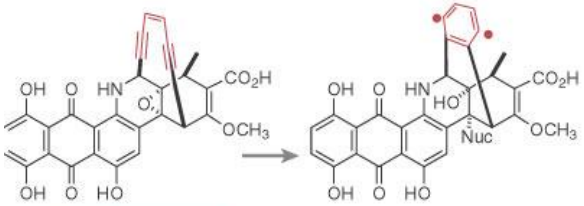
natural products

drugs

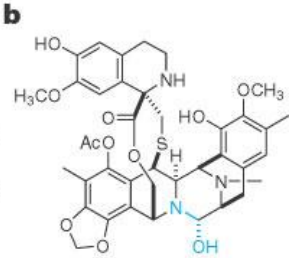
*J. Chem. Inf. Comput. Sci.*2003431218-227



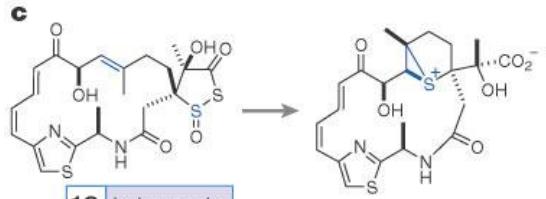
9 Calicheamicin



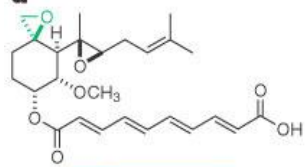
10 Dynemicin



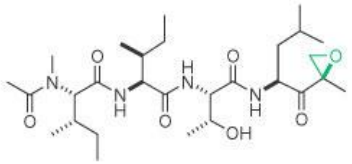
11 Ecteinascidin



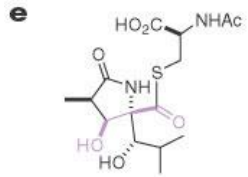
12 Leinamycin



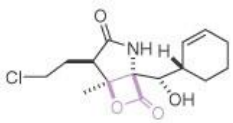
13 Fumagillin



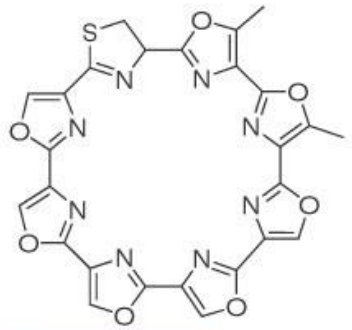
14 Epoxomicin



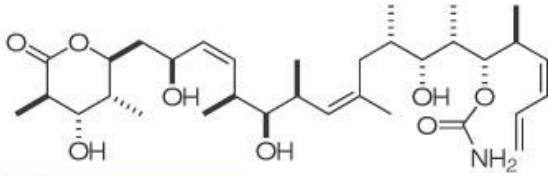
15 Lactacystin



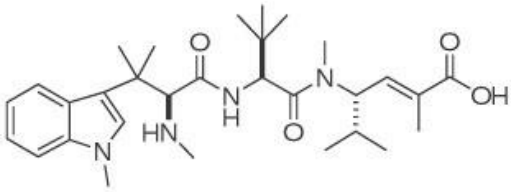
16 Salinosporamide



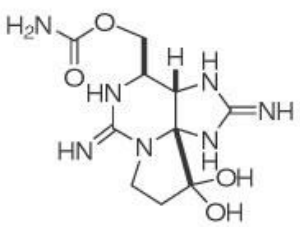
17 Telomestatin



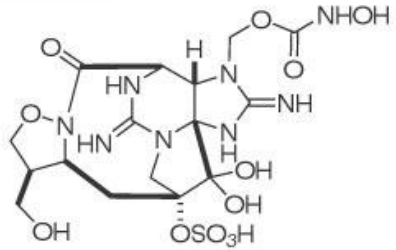
18 Discodermolide



19 Hemiasterlin



20 Saxitoxin



21 Zetekitoxin AB

Molecular Plot

File

DATA SOURCES

Abbott kinase dataset

PLOTS

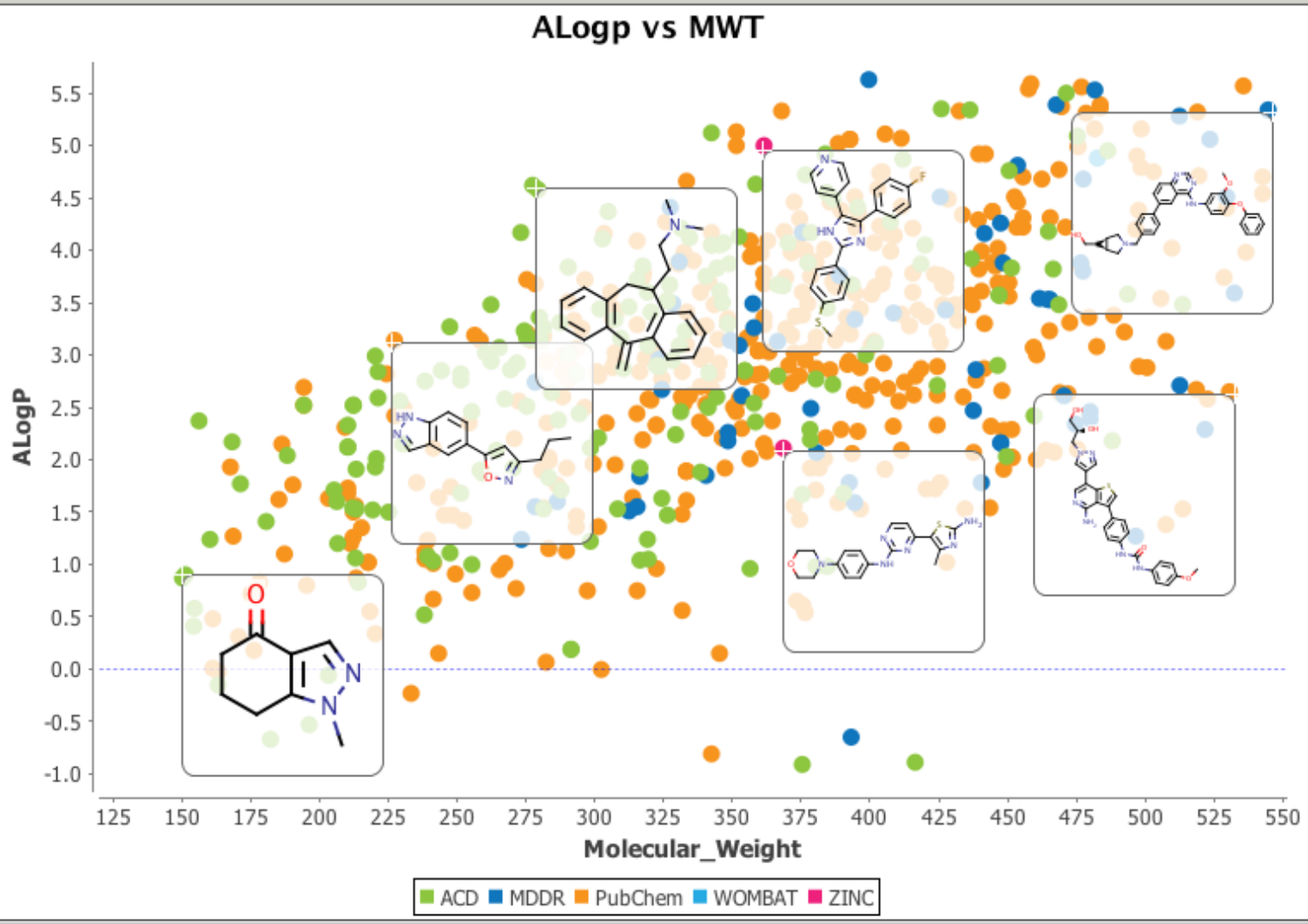
FLT1 vs FLT3

ALogp vs MWT

AKT1 vs AKT2

JAK2 vs JAK3

DYRK1A vs DYRK1B



Metabolômica: Desafios

Metabólitos

Primários

Secundários

Lipídeos, açucares,

ácidos graxos

Policetídeos, alcaloides,

NRP, terpenos

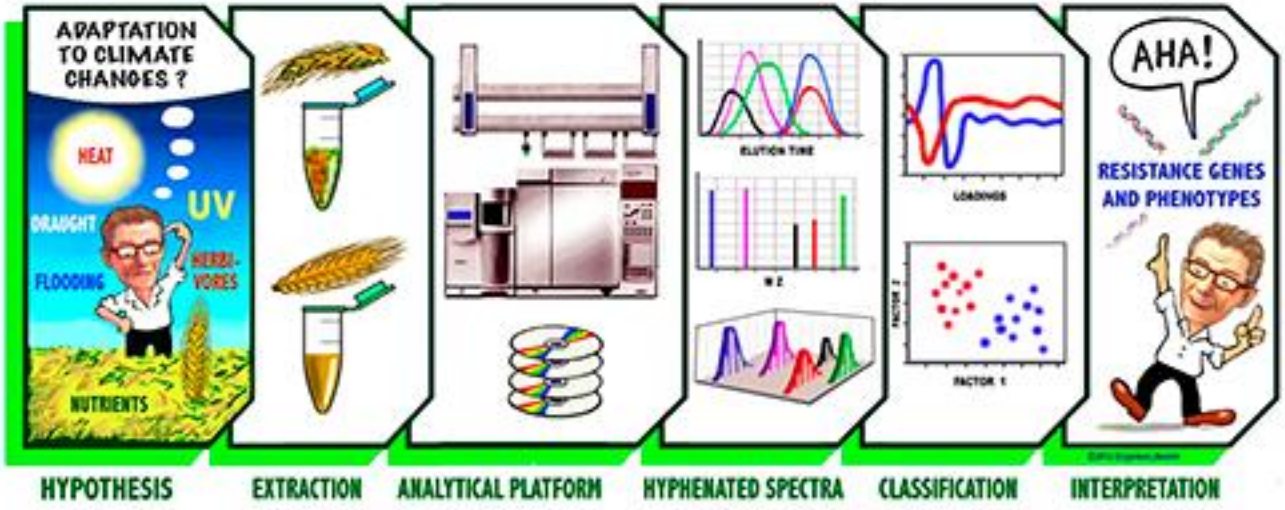

Metabolomica

Foodomics é uma disciplina que examina todo o conjunto de substâncias presentes em nossos alimentos (foodome). A disciplina utiliza plataformas analíticas avançadas para investigar a composição do alimento e, assim, suas propriedades nutricionais e impacto na saúde. As novas técnicas também fornecem uma imagem detalhada da qualidade dos alimentos e podem ser usadas para detectar fraudes em alimentos e encontrar soluções para outros desafios na produção de alimentos. Os resultados de pesquisas de foodomics têm um impacto direto sobre os consumidores, a indústria de alimentos e a sociedade.

Engelsen
(https://food.ku.dk/english/research_at_food/research_fields/foodomics/

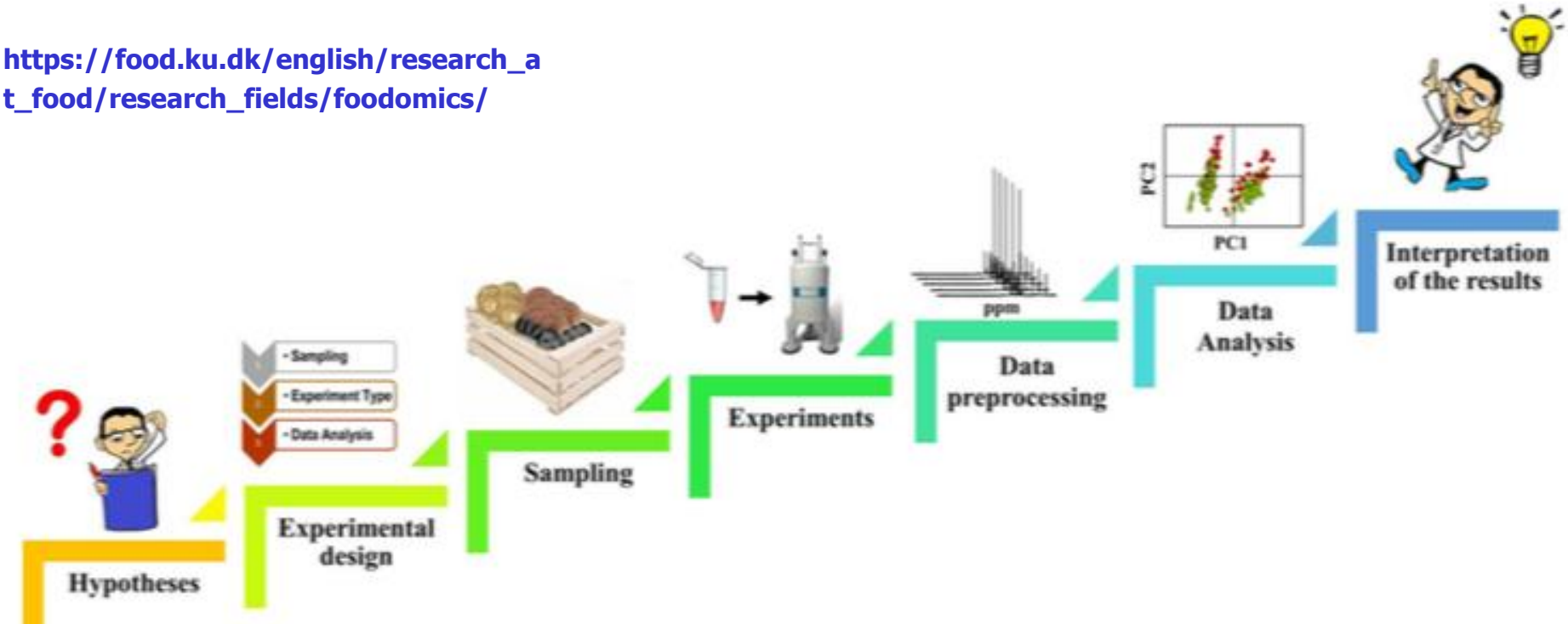
***Foodomics*—a discipline that studies the food and nutrition domains through the application of advanced omics technologies to improve the consumer's well-being, health, and knowledge.**

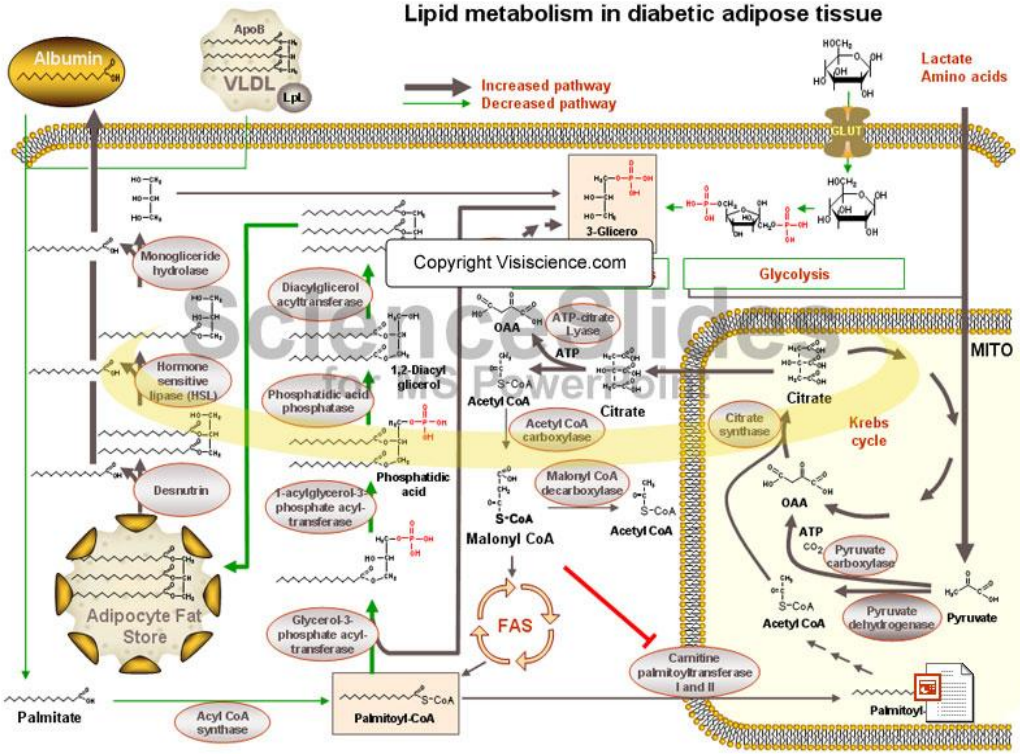
Khakimov & Engelsen, 2017

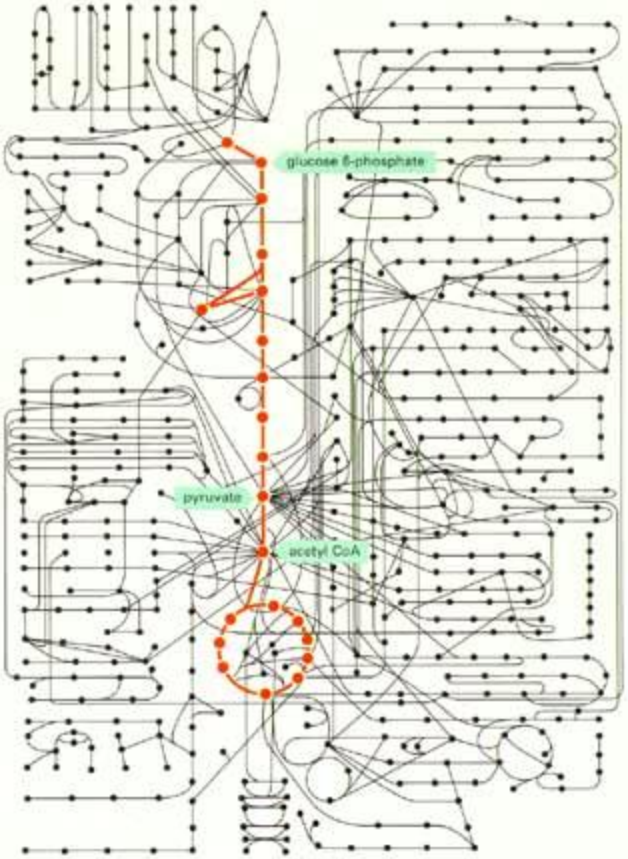


Foodomics. Illustration by Søren B. Engelsen and Tim Newlin ©

https://food.ku.dk/english/research_at_food/research_fields/foodomics/




Lipid metabolism in diabetic adipose tissue
Albumin
ApoB
VLDL
LpL
Increased pathway
Decreased pathway
Lactate
Amino acids
GLUT
3-Glicero
Glycolysis
Copyright Visiscience.com
Monoglyceride hydrolase
Diacylglicerol acyltransferase
Hormone sensitive lipase (HSL)
1,2-Diacyl glicerol
Phosphatidic acid phosphatase
Phosphatidic acid
Desnutrin
1-acylglycerol-3-phosphate acyl-transferase
Adipocyte Fat Store
Glycerol-3-phosphate acyl-transferase
Palmitate
Acyl CoA synthase
Palmitoyl-CoA
OAA
ATP-citrate Lyase
ATP
Acetyl CoA
Citrate
Acetyl CoA carboxylase
Malonyl CoA decarboxylase
S-CoA
Malonyl CoA
Acetyl CoA
FAS
Carnitine palmitoyltransferase I and II
Palmitoyl-
MITO
Citrate
Citrate synthase
Krebs cycle
OAA
ATP
CO2
Pyruvate carboxylase
Pyruvate
Acetyl CoA
Pyruvate dehydrogenase


glucose 6-phosphate
pyruvate
acetyl CoA

Metabolite Profile

fingerprint

Table 1. Main Features of Most The Popular Techniques for Metabolomics

technique	CE−MS	GC−MS	LC−MS	NMR 1D	NMR 2D
universality	++	+	+/++	+++	+++
accuracy	−/++[a]	−/++[a]	−/++[a]	+++	+++
reproducibility	++/−[a]	++/−[a]	++/−[a]	+++	+++
sensitivity	+	++/+++[a]	+++	−	−
resolution	++	+++	+++	+	++
efficiency	++	−	+	++	++

[a]Accuracy, repeatability, and sensitivity mainly depend on the analyzer. GC−MS and LC−MS are usually performed either with a quadrupole or time-of-flight analyzer. "+" or "−" in front of and behind "/" represent quadrupole and time-of-flight analyzer, respectively.

*J. Agric. Food Chem.*201967236445-6457

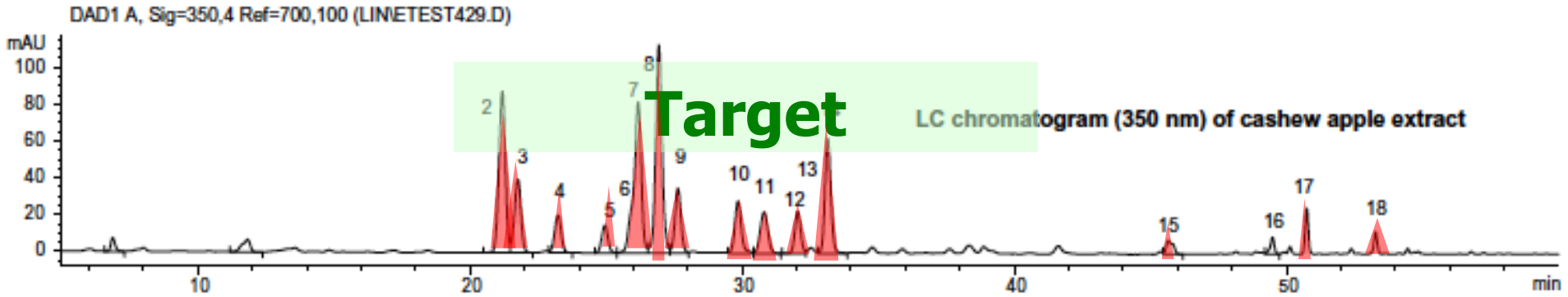
ABORDAGEM?

COMPOSTOS

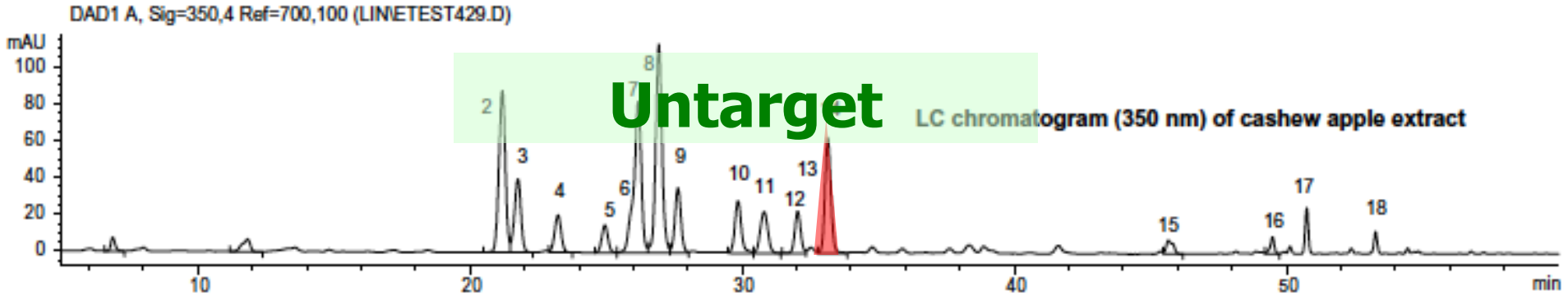
ALVO

NÃO ALVO

Caracterização completa e quantificação



Definição de marcadores e sua posterior quantificação



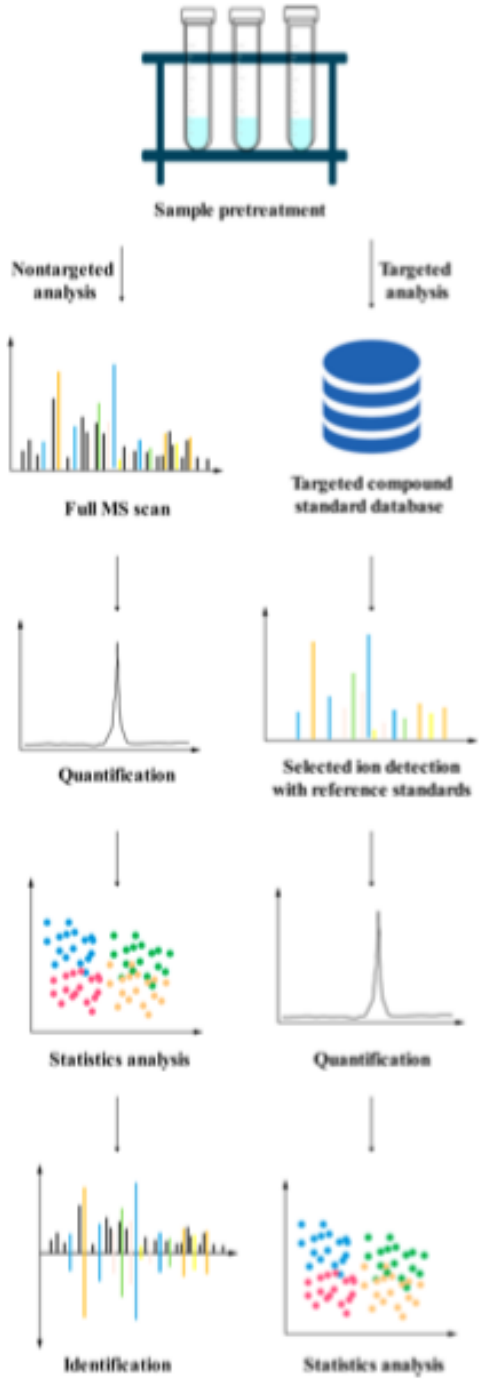


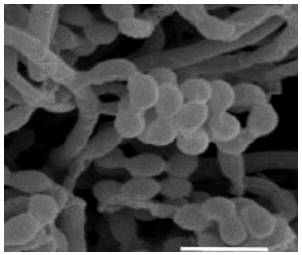
Figure 1. Workflow of metabolomics.

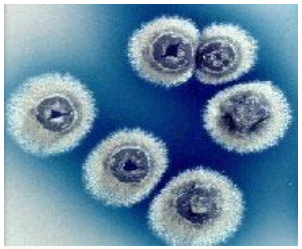
*J. Agric. Food Chem.*201967236445-6457

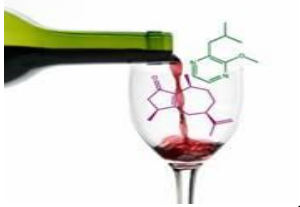
Table 1
Reported applications of metabolomics in life sciences research and practice

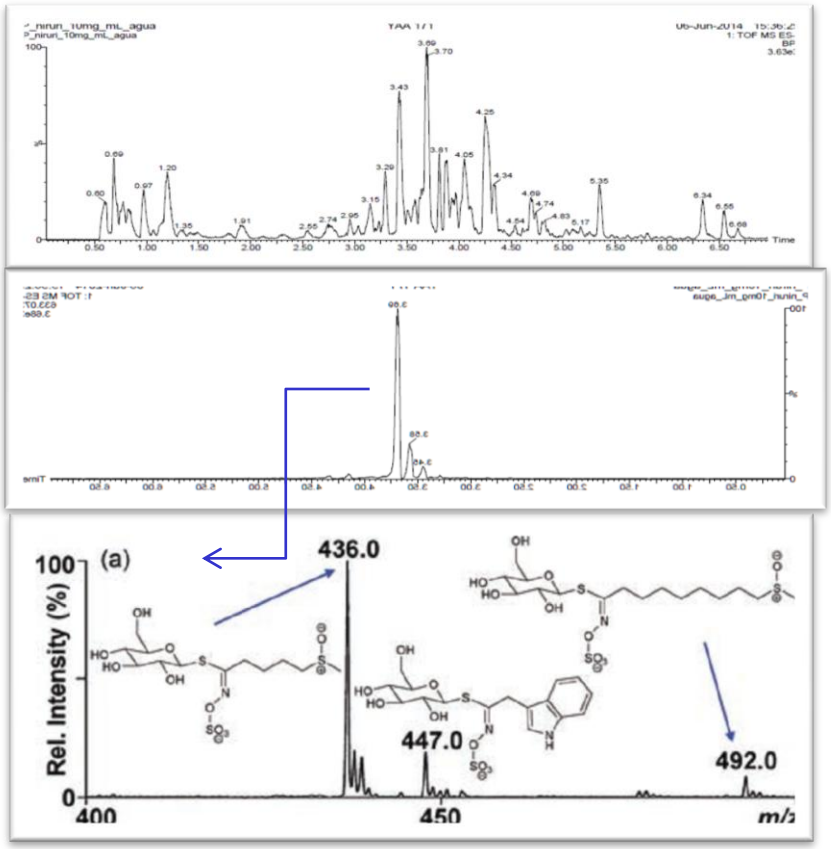
Basic research	References
Functional genomics	[2,5,6]
Interaction between metabolome, transcriptome and proteome	[7,8]
Discovery of new biochemical pathways	[9]
Interaction between species	[10]
Analysis of metabolic regulation	[11]
Applied research	**References**
Medical applications	
Understanding of disease pathophysiology	[12,13]
Disease biomarker identification	[14,15]
Early diagnosis	[16]
Personalized medicine	[17–19]
Clinical trial monitoring	[32,33]
Drug discovery	[34,35]
Toxicology—Drug safety	[36]
Agricultural/Nutrition Applications	
Identification of metabolic engineering targets	[20,21]
Understanding of stress response	[22,23]
Classification of special varieties of produce (e.g. tea, ginseng, fish)	[24–26]
Genetically modified (GM) food certification	[27–29]
Human nutrition	[30,31]
Industrial Applications	
Identification of metabolic engineering targets in *Escherichia coli*, yeast, algae	[37]
Fermentation process improvement	[38]
Biologics production and fermentation process optimization	[39]

Instrumentação: UPLC – QTOF – MS/MS









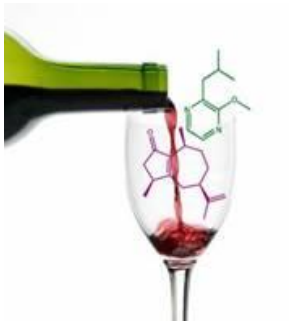
Agumas aplicações:

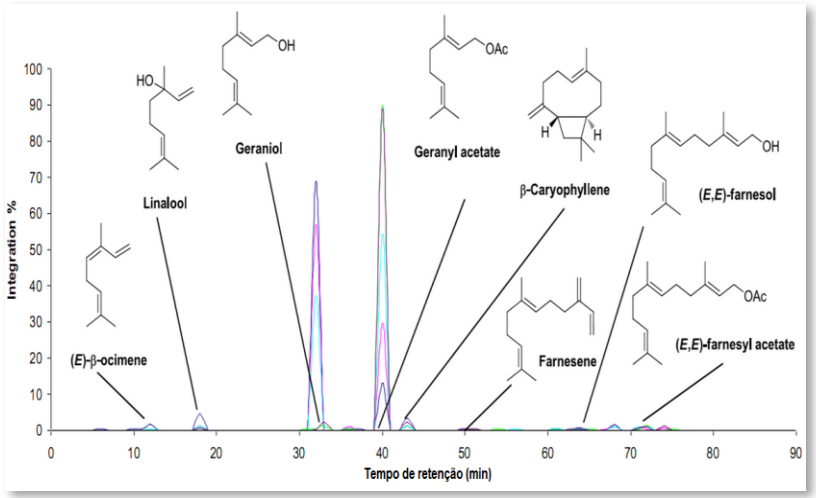
Produtos naturais -

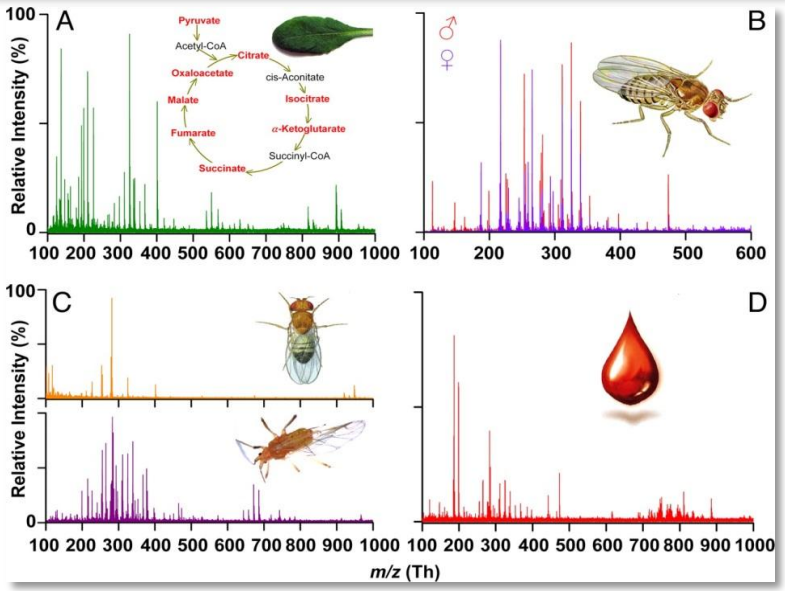
Insumos agroindustriais

Determinação estrutural de substâncias de origem vegetal, animal e de microorganismos

Instrumentação: GC–MS/MS







Agumas aplicações:

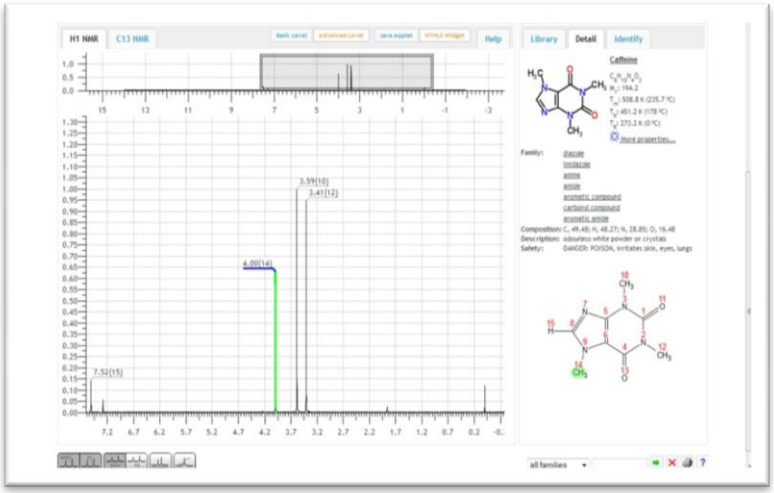
Análises de aromas de vinhos

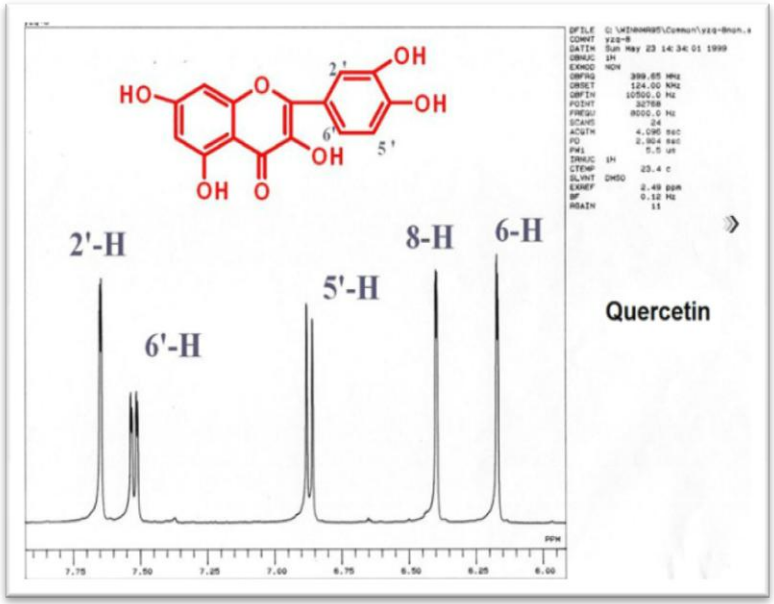
Óleos essenciais

Feromônios

Estudos de ecologia química

Instrumentação: Ressonância Magnética Nuclear (RMN)

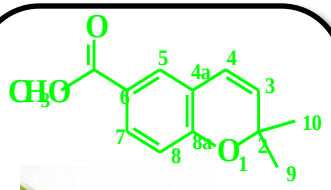


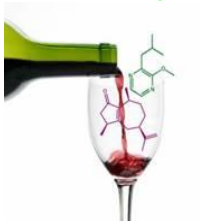


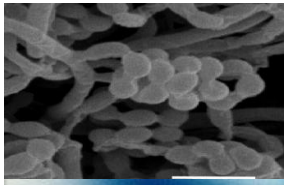
Sistema de detecção universal

Estudos de Metabolômica e Elucidação estrutural

Quantificação sem necessidade de padrões analíticos

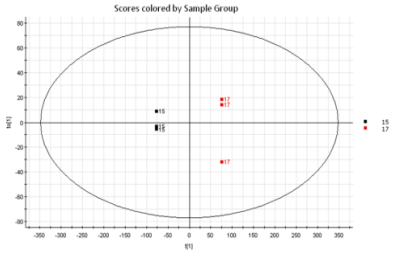


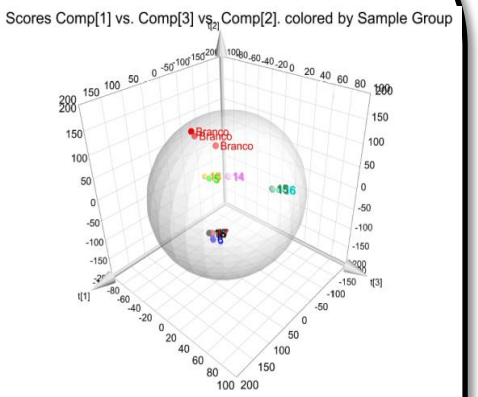




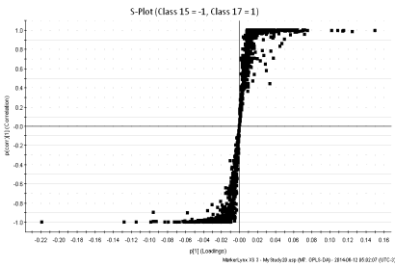
Métodos Quimiométricos

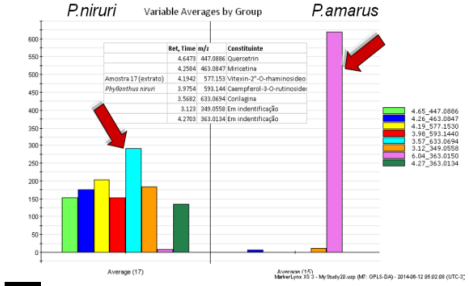
Análise de Componentes Principais (PCA)





OPLS-DA





Determinação de Biomarcadores

Interpretação de grande volume de dados

Metabolite profiling by GC-EI-TOF covers ~ 50-550 Da

R-OH
R-COOH
$R\text{-}NH_2$
$R_2\text{-}NH$
R-SH

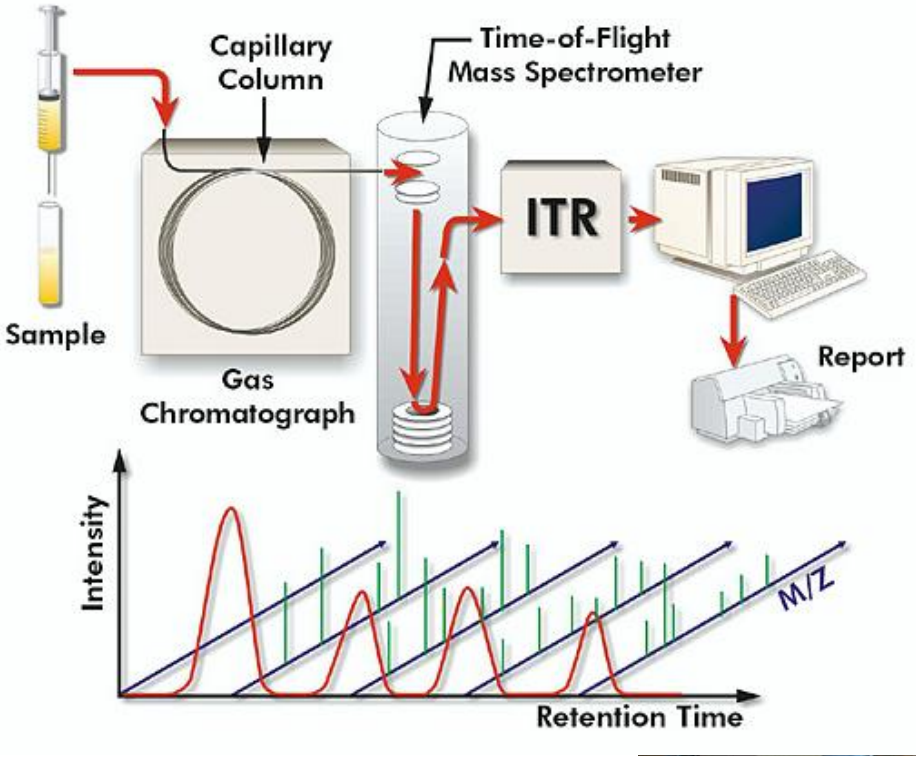
(a) methoxymation

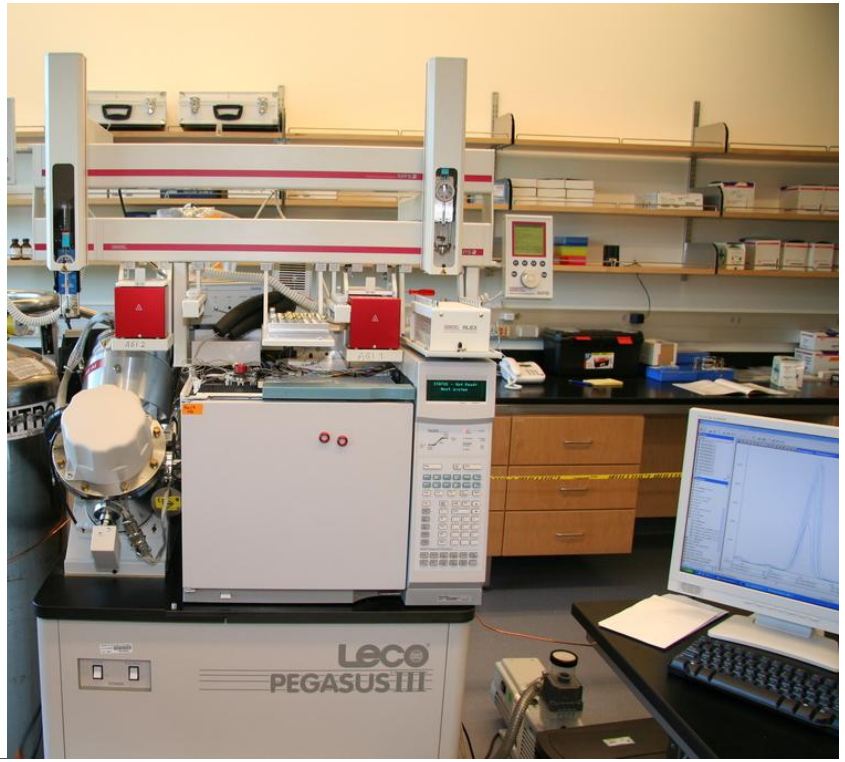
(b) silylation

(manually or automated)

R-OTMS
R-COOTMS
$R\text{-}NTMS_2$
$R_2\text{-}NTMS$
R-STMS
$R_2\text{-}CNOCH_3$

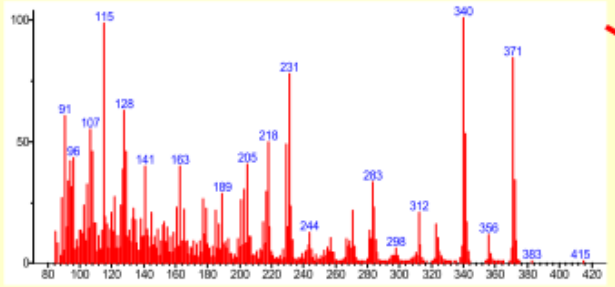
Exchange of H against TMS removes H-bridges to increase volatility



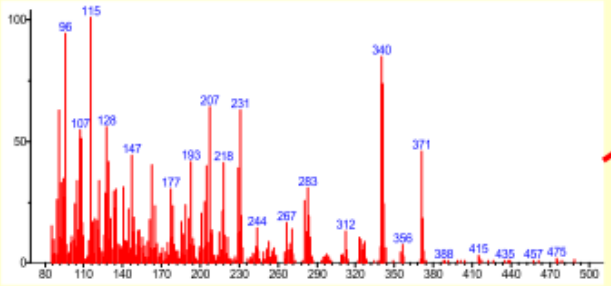


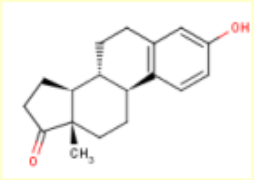
Library – Naming convention must be consistent but is not trivial

Estrone major
FiehnRI 948753



Estrone minor
FiehnRI 950990



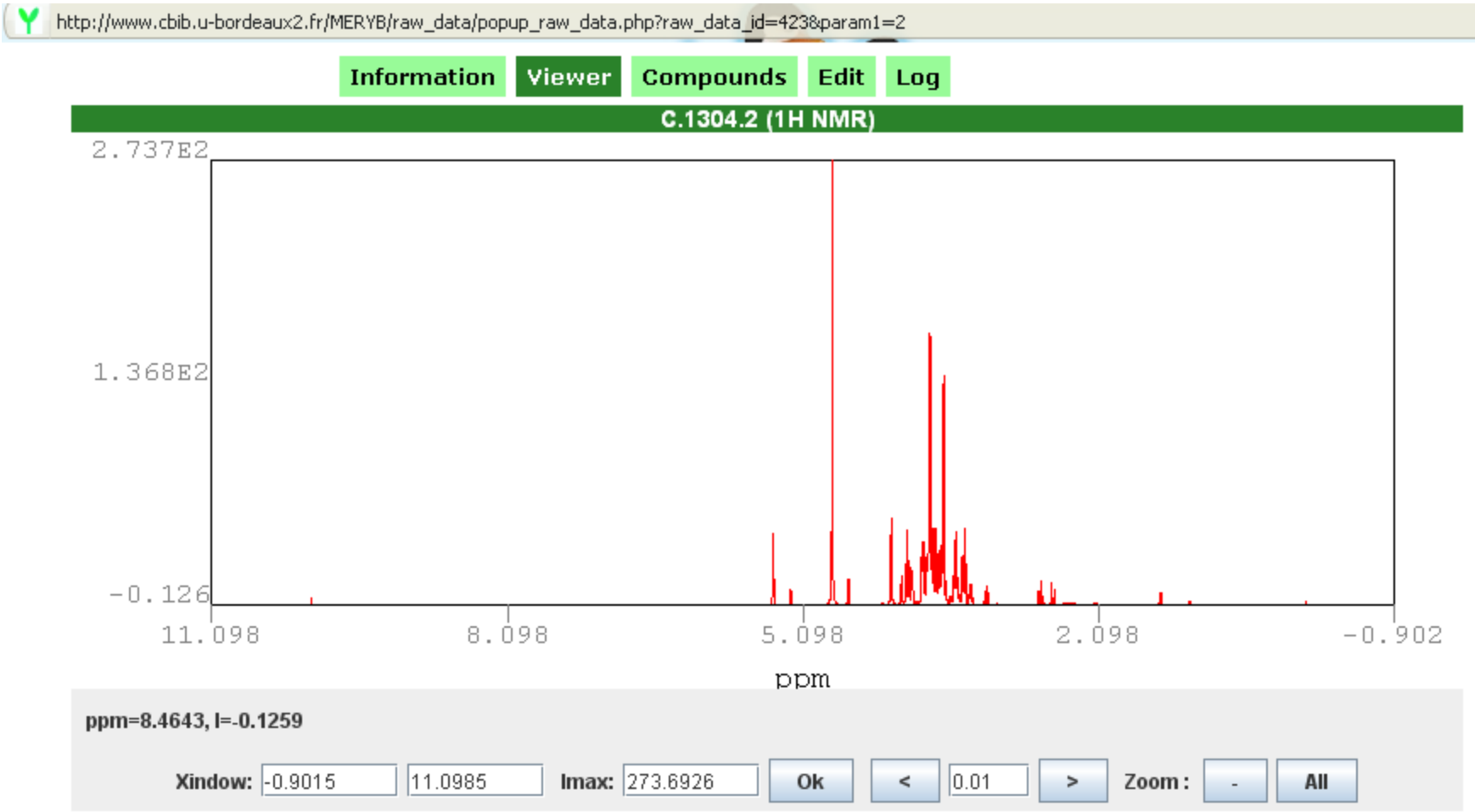


Estrone

Solution:

mass spectra + retention time
point to the biological name estrone

with PubChem CID: 5870 and KEGG ID and InChIKey=DNXHEGUUPJUMQT-UHFFFAOYAI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Information
Viewer
Compounds
Edit
Log
C.1304.2 (1H NMR)
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1.368E2
-0.126
11.098
8.098
5.098
2.098
-0.902
ppm
ppm=8.4643, I=-0.1259
Xindow:
-0.9015
11.0985
Imax:
273.6926
Ok
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>
Zoom :
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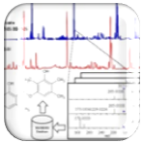
All

RMN

Bioprospecção de Moléculas com Potencial Terapêutico

Biodiversidade



Metodologia de Desreplicação

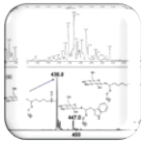
Aumento da sistematização, informação, maiores chances de sucesso

Preparo dos extratos

Armazenamento em Extratoteca

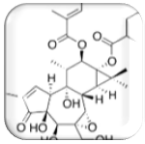
Fracionamento e caracterização por HPLC

Triagem de alta velocidade (HCS)



Identificação cromatográfica

Armazenamento Biblioteca de frações e substâncias puras



Novas moléculas

Banco de dados

Novos medicamentos

Estratégia de Desreplicação

Dinâmica de Pesquisa

Coleta do material vegetal (Bancos de Germoplasma – Embrapa)

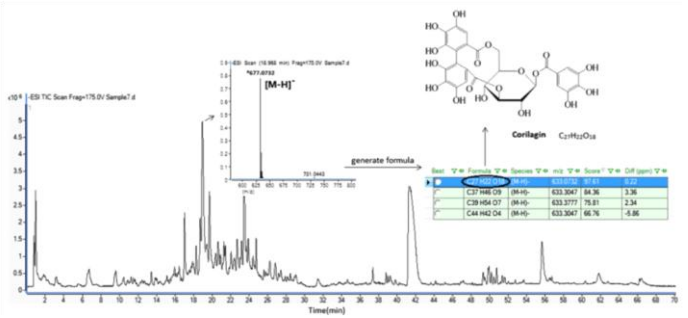
Extração do Material	Preparação das Amostras

Metodologia de Desreplicação – Determinação do perfil Químico do Extrato Bruto

UPLC-QToF-MS/MS/ HPLC - PDA	RMN

Fracionamento – Obtenção de 10 frações por planta

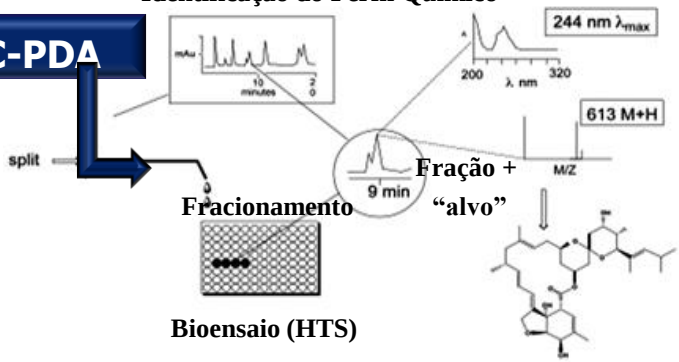
Preparo das amostras	Bioensaio (HTS)



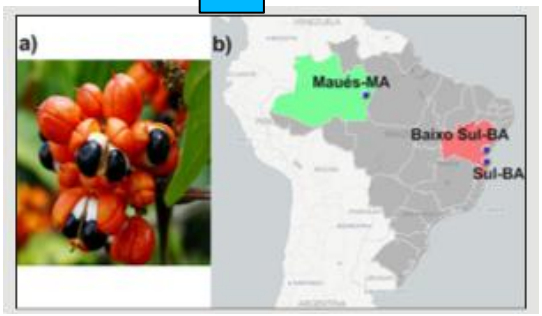
UPLC-HRMS

HPLC-PDA

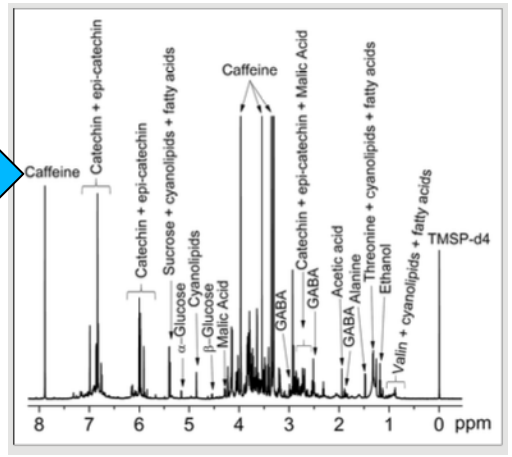
Identificação do Perfil Químico



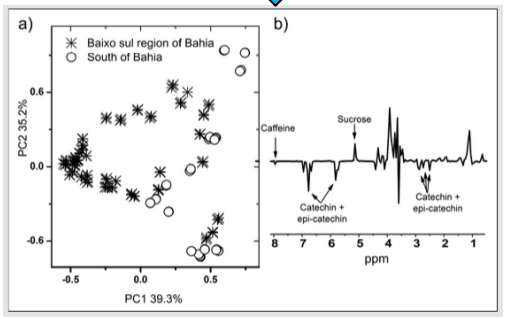
Confirmação estrutural via RMN



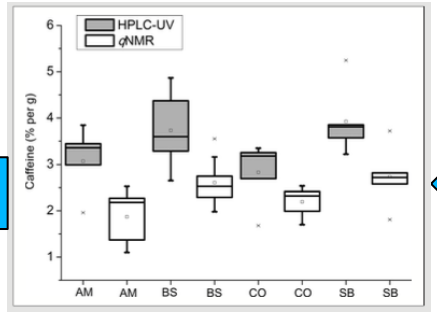
Sample set



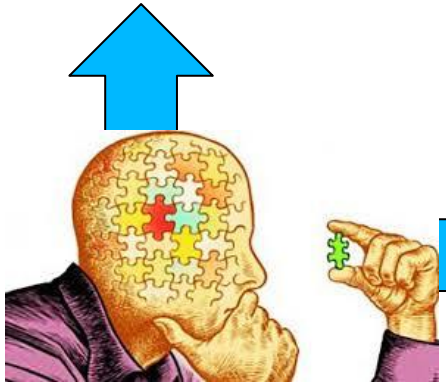
fingerprint



chemometrics



quantitation



Qual a minha hipótese?

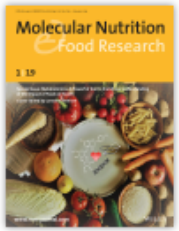
Metabolômica e Nutrição: condições experimentais

Molecular Nutrition & Food Research

Review | Free Access

Nutrimetabolomics: An Integrative Action for Metabolomic Analyses in Human Nutritional Studies

Marynka M. Ulaszewska, Christoph H. Weinert, Alessia Trimigno, Reto Portmann ... See all authors



Volume 63, Issue 1
Special Issue: Metabolomics: A Powerful Tool to Enrich our Understanding of the Impact of Food on Health
January 2019
1800384

REVIEW

Nutritional Metabolomics

Molecular Nutrition & Food Research

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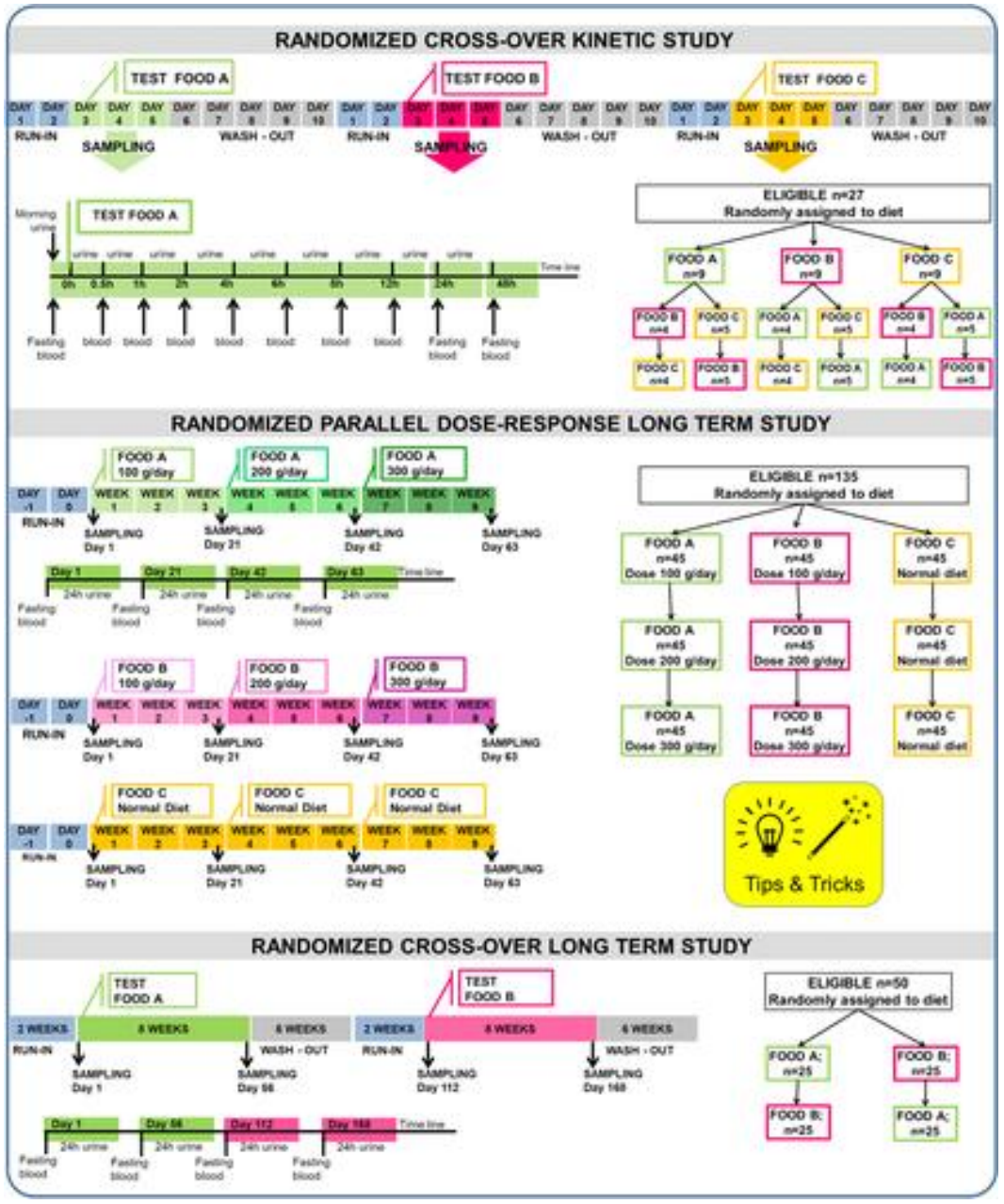
Nutrimetabolomics: An Integrative Action for Metabolomic Analyses in Human Nutritional Studies

*Marynka M. Ulaszewska, Christoph H. Weinert, Alessia Trimigno, Reto Portmann, Cristina Andres Lacueva, René Badertscher, Lorraine Brennan, Carl Brunius, Achim Bub, Francesco Capozzi, Marta Cialiè Rosso, Chiara E. Cordero, Hannelore Daniel, Stéphanie Durand, Bjoern Egert, Paola G. Ferrario, Edith J.M. Feskens, Pietro Franceschi, Mar Garcia-Aloy, Franck Giacomoni, Pieter Giesbertz, Raúl González-Domínguez, Kati Hanhineva, Lieselot Y. Hemeryck, Joachim Kopka, Sabine E. Kulling, Rafael Llorach, Claudine Manach, Fulvio Mattivi, Carole Migné, Linda H. Münger, Beate Ott, Gianfranco Picone, Grégory Pimentel, Estelle Pujos-Guillot, Samantha Riccadonna, Manuela J. Rist, Caroline Rombouts, Josep Rubert, Thomas Skurk, Pedapati S. C. Sri Harsha, Lieven Van Meulebroek, Lynn Vanhaecke, Rosa Vázquez-Fresno, David Wishart, and Guy Vergères**

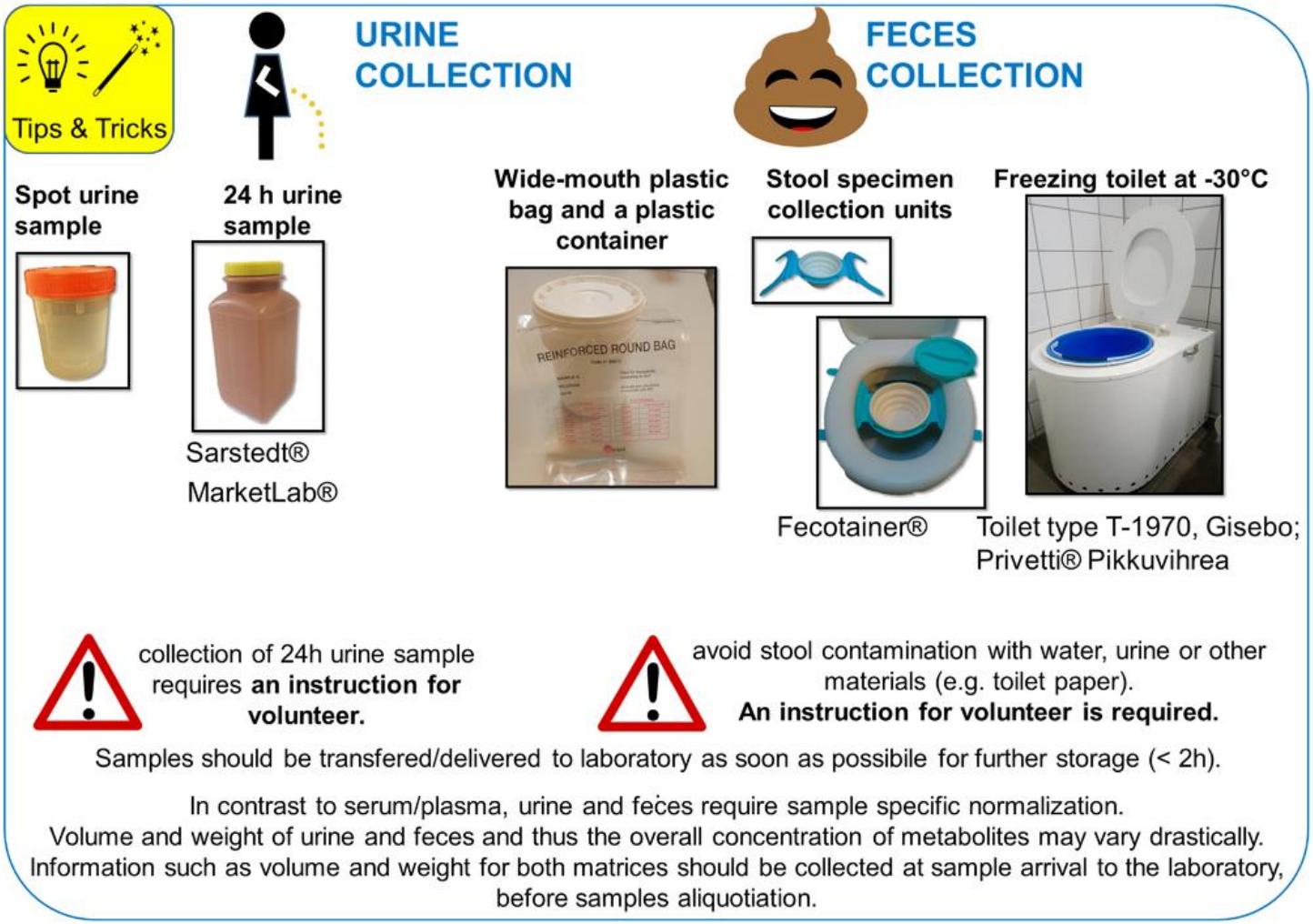
Qual o desenho experimental?

Nutrimetabolomics: An Integrative Action for Metabolomic Analyses in Human Nutritional Studies



Molecular Nutrition & Food Research, Volume: 63, Issue: 1, First published: 03 September 2018, DOI: (10.1002/mnfr.201800384)

Amostras?



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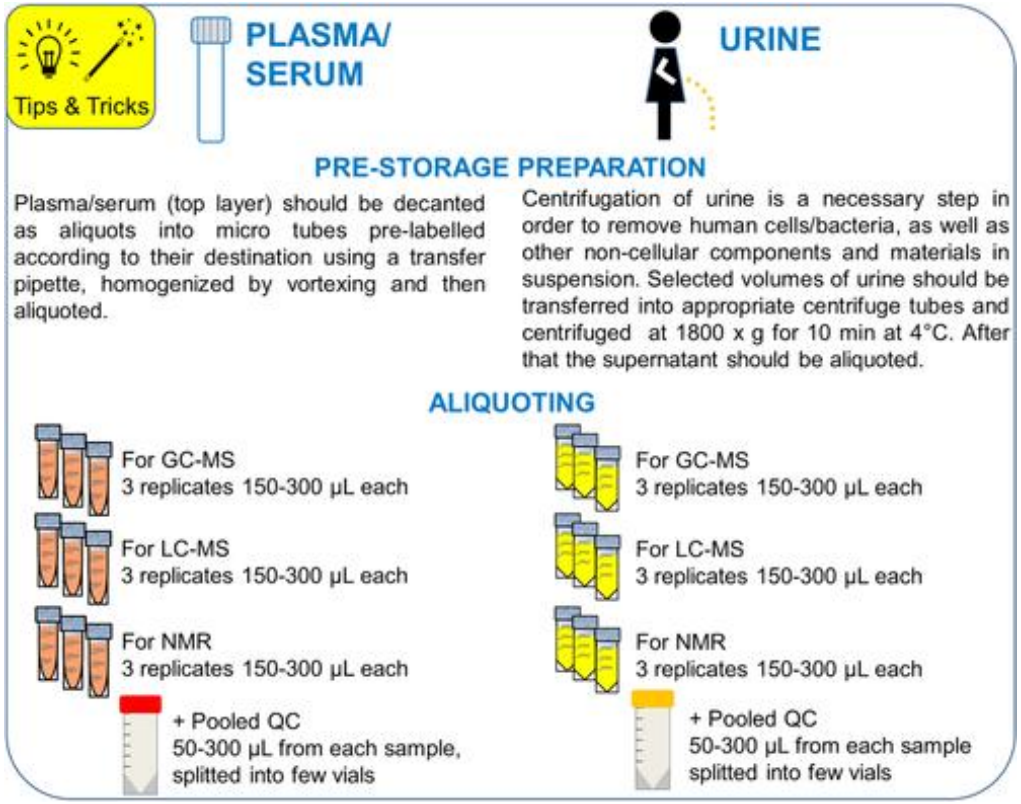
Preparação de amostras

Table 2. Overview of commonly used solvents for microbial metabolite extraction.

Method	Organism	Ref.
For general extraction of different chemical classes		
80:20 methanol:water	*Escherichia coli*	[103–105]
50:50 methanol: water	*Schizosaccharomyces pombe*	[106]
60:40 methanol:water	*Streptomyces coelicolor*	[33]
72:28 methanol	*Saccharomyces cerevisiae*	[107]
30:30:20 methanol:water:chloroform	*Corynebacterium glutamicum*	[108]
60:20:20 methanol:water:chloroform	*Leishmania donovani*	[80]
40:40:20 acetonitrile:methanol:water with 0.1% formic acid	*S. cerevisiae*	[31]
	E. coli	[66]
40:40:20 acetonitrile:methanol:water	*Mycobacterium tuberculosis*	[109]
	E. coli	[110]
	S. cerevisiae CEN.PK 113–7D	[61]
Boiling HEPES-buffered ethanol solution (75:25 v/v ethanol:water; pH=5.2)	*Methylobacterium extorquens*	[111]
60:40 ethanol:water	*Staphylococcus aureus*	[58]
Pure cold methanol	*E. coli*	[66,71,72,112]
Boiling ethanol	*S. cerevisiae* CEN.PK 113–7D	[61]
	E. coli, B. subtilis, S. cerevisiae	[49]
	S. cerevisiae	[60,69,113–115]
	Pichia pastoris	[116]
For specific classes of compounds		
Alkaline extraction (for basic molecules)	*M. extorquens*	[111]
Acid extraction (for organic acids)	*M. extorquens*	[111]
Bligh–Dyer method (for lipids)	*Trypanosoma brucei*	[117]



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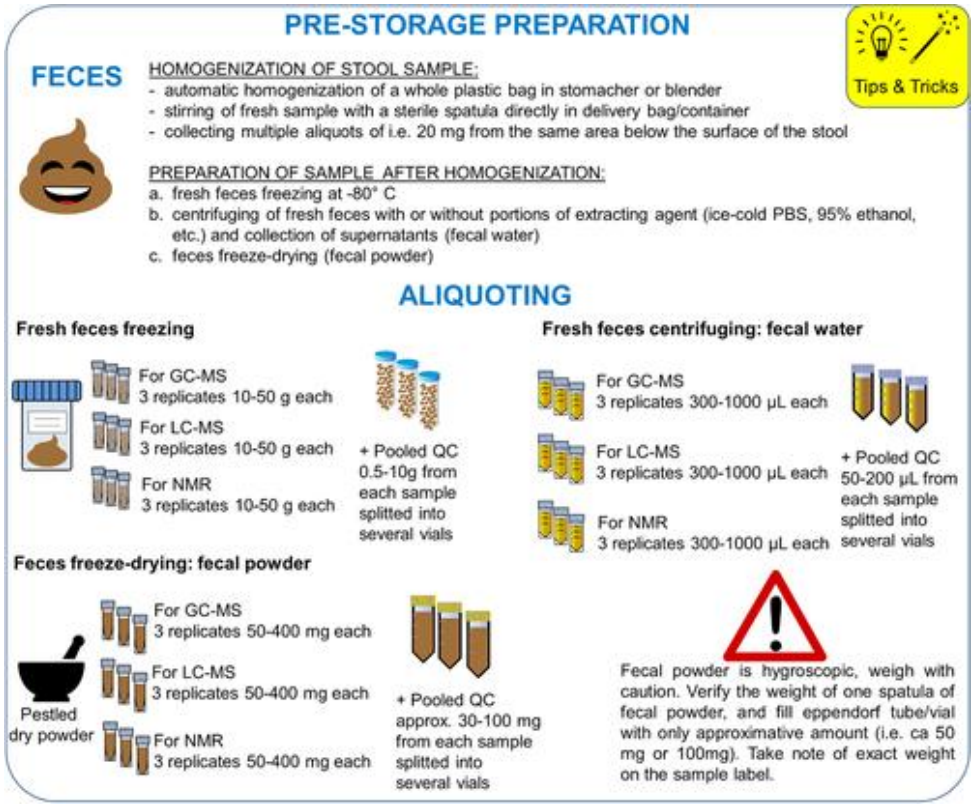

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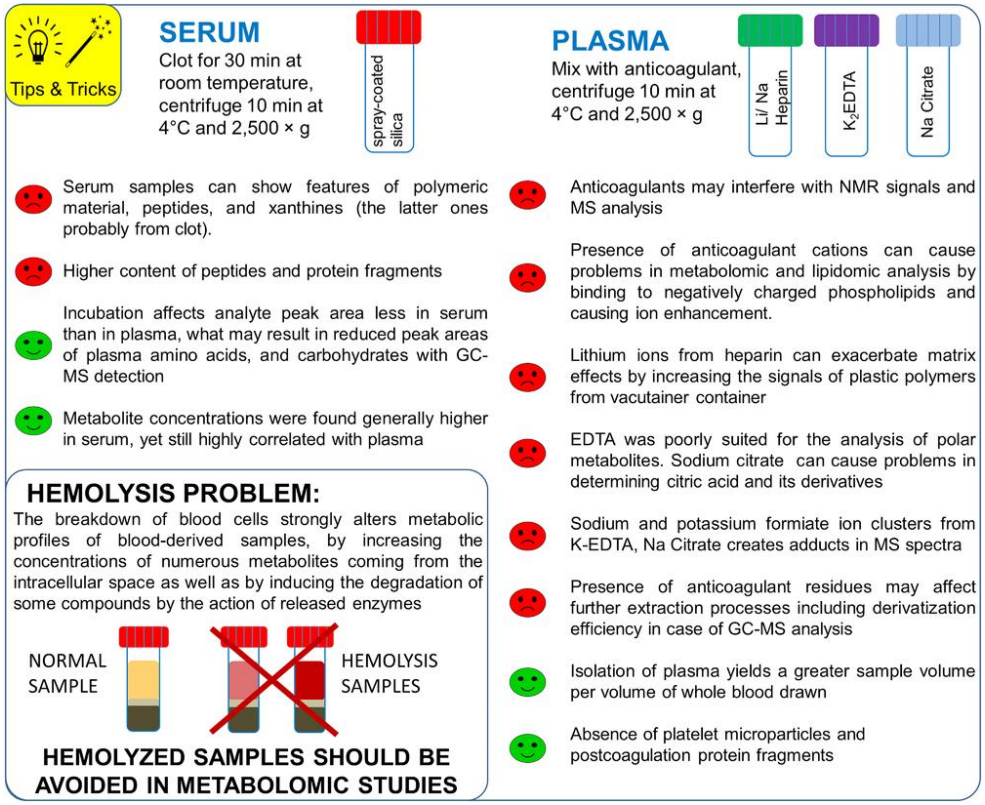

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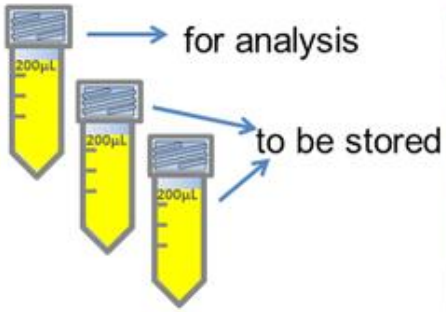
Qual abordagem analítica?

UNTARGETED ANALYSIS

- LC-MS: polar and medium-polar metabolites (i.e., polyphenols and its metabolites, amino acids)
- LC-MS: non-polar metabolites (i.e., lipidomics)
- GC-MS: volatile metabolites
- GC-MS: metabolites after derivatization
- NMR



MINIMUM REQUIREMENT

3 ALIQUOTS FOR ONE TYPE OF ANALYSIS (i.e., GC-MS)

TARGETED ANALYSIS

- LC-MS (kits, etc)
- GC-MS quantitative analysis with/without derivatization (i.e., SCFA, sugars etc.)
- Clinical analysis (multiple choices)

In general multiple aliquots of smaller volume (i.e., 200 µL) are recommended, rather than few aliquots of high volume.

Higher number of aliquots is recommended, which allows for additional analysis (i.e., additional analytical technique that arises in the future)

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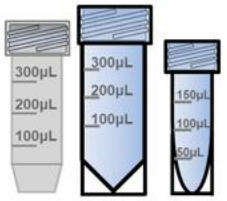
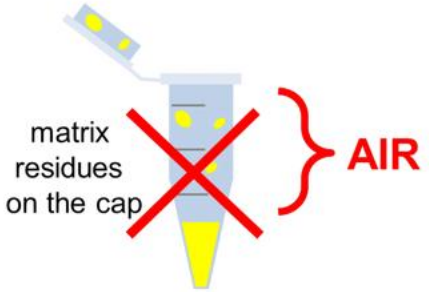
Use containers adapted for the volume of matrix that will be stored. Matrix should fill possibly 80%-90% of container with minimum amount of air above its surface. Use containers with low adherence in the interior of the tube, and twisted caps that avoid splashing while opening

Tips & Tricks

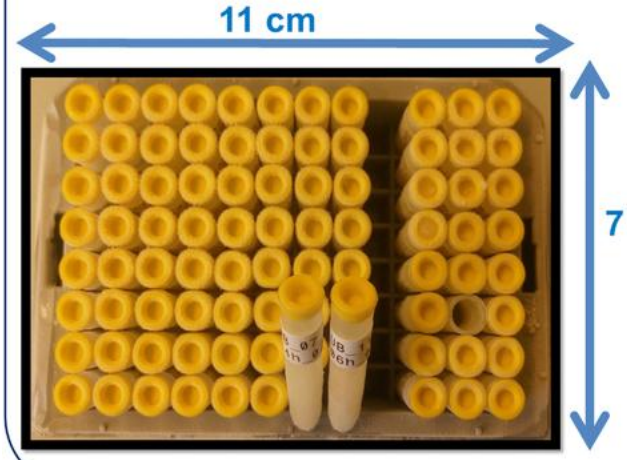
Conical/skirted vials for better recovery of matrix, twisted caps.
Recommended cryovial

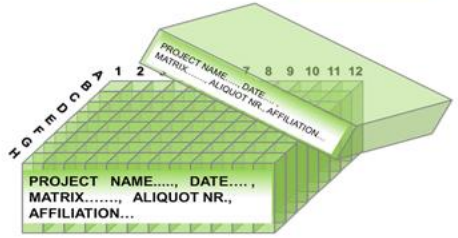


-80 °C
and lower



DON'T STORE AIR





Optimization of storage capacity in freezers of small aliquotes in tight boxes. Avoid long term storage in paper boxes.

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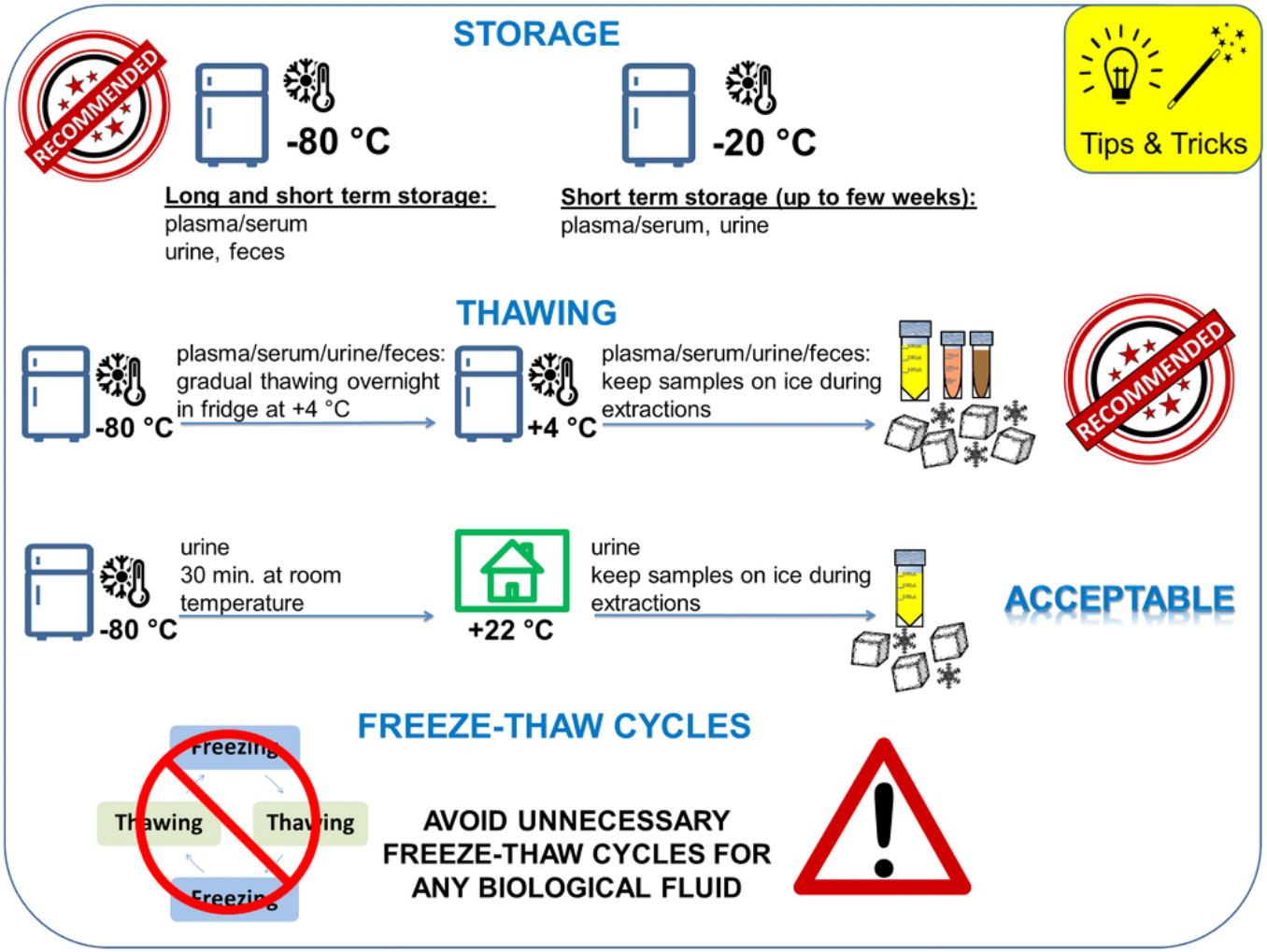

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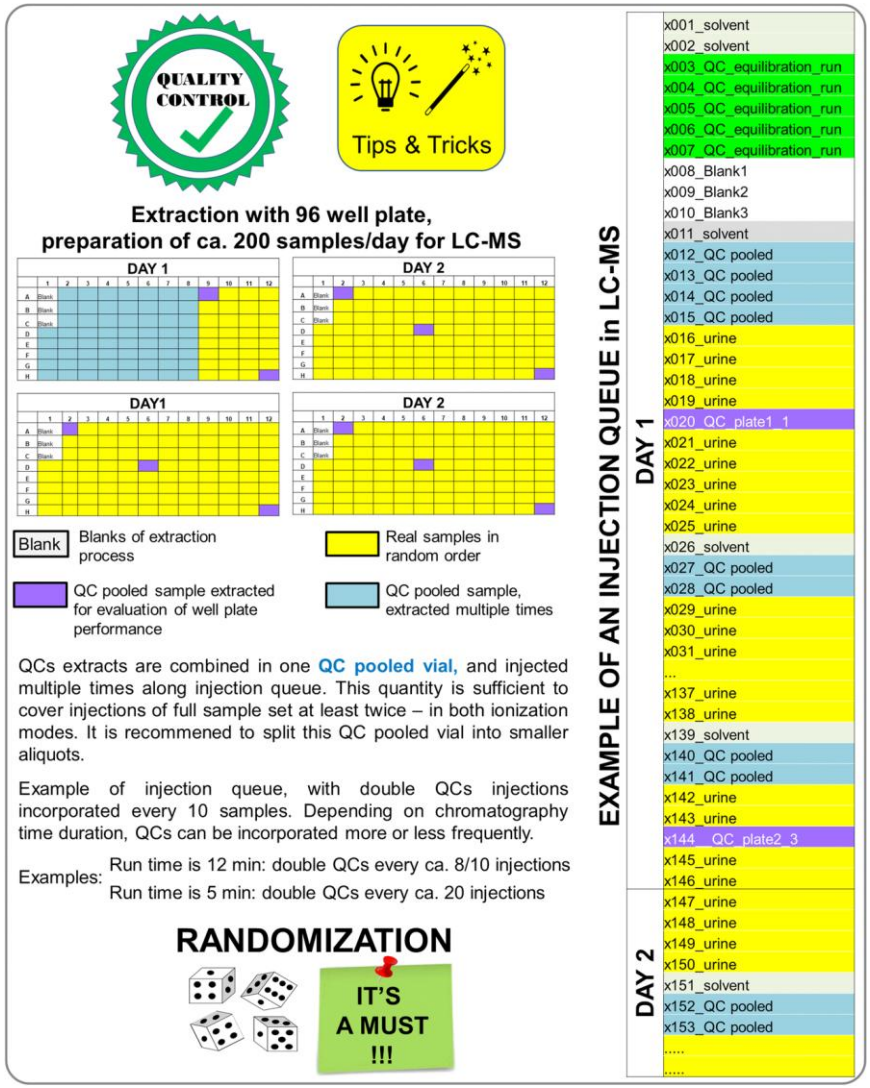

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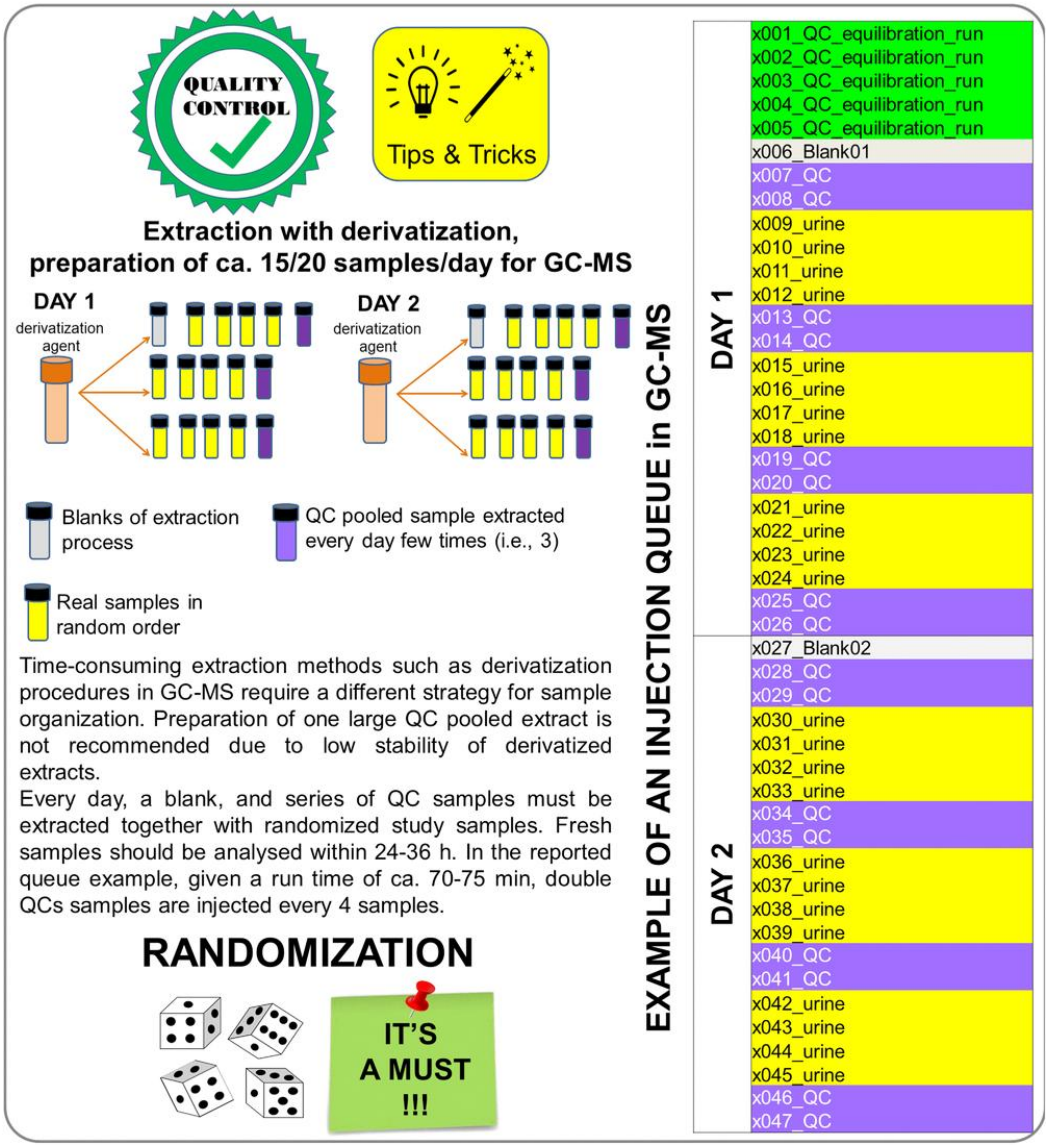

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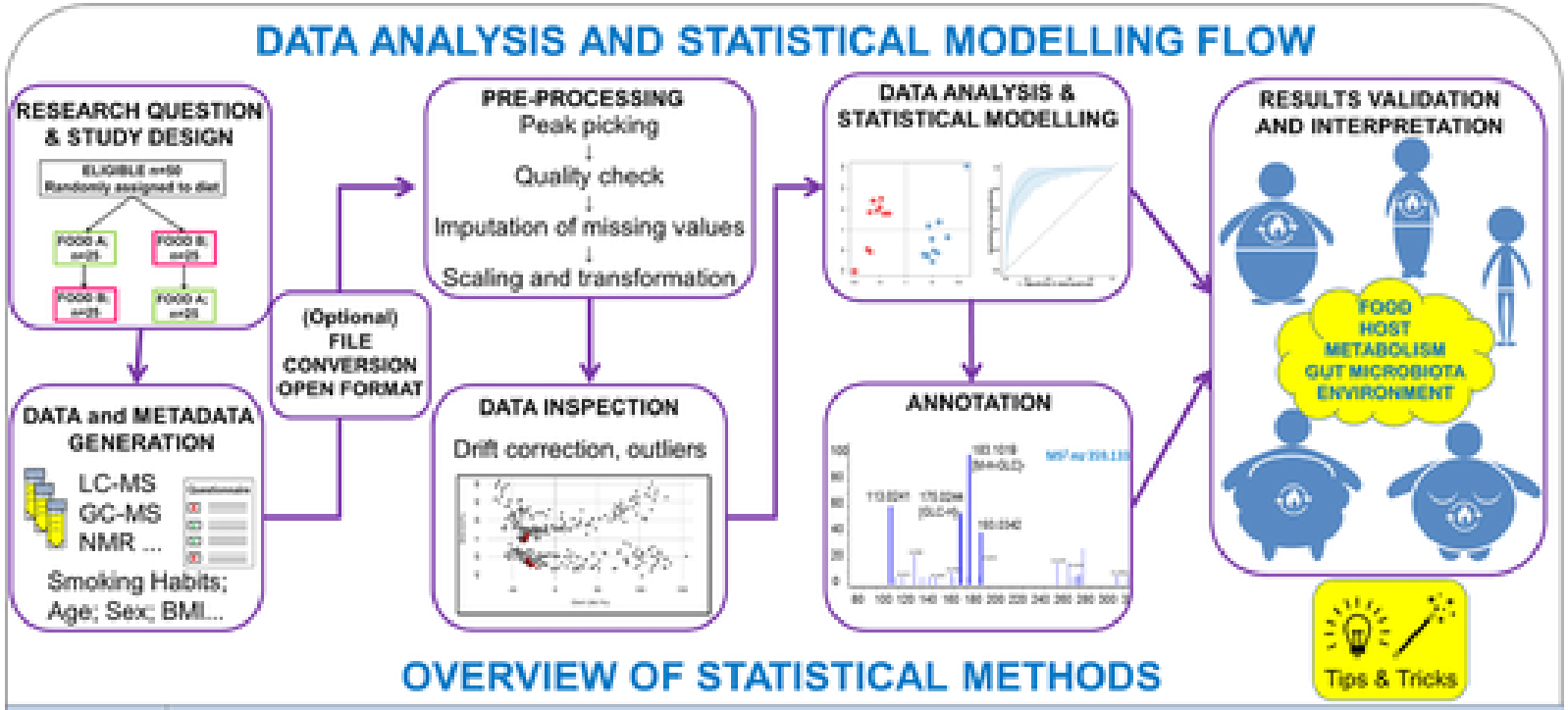
Qual análise estatística?

Nutrimetabolomics: An Integrative Action for Metabolomic Analyses in Human Nutritional Studies



OVERVIEW OF STATISTICAL METHODS

	Visualisation	Regression	Independent 2-group test	Dependent 2-group test	Multiple factors	Time series
Parametric	Plot	Linear models	t-test	Paired t-test	ANOVA Mixed models	Repeated measures
Non-parametric	Rank plot	Rank regr. Kernel LOESS	MW U-test	Wilcoxon signed-rank	Kruskal-Wallis Friedman rank ANOVA	Friedman rank-repeated
Multivariate	PCA	PLS; RF; SVM	PLS-DA; RF; SVM	ML-PLS; ML-RF; ML-SVM	ANOVA decomposition	ANOVA decomposition

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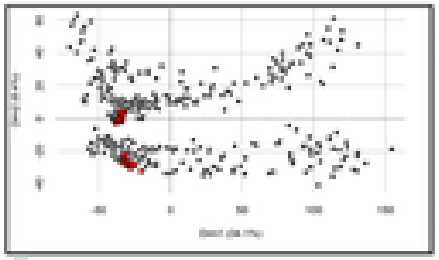
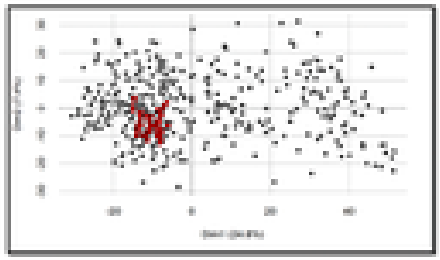
VERIFICATION OF QUALITY CONTROL SAMPLES AND OUTLIERS WITH PCA

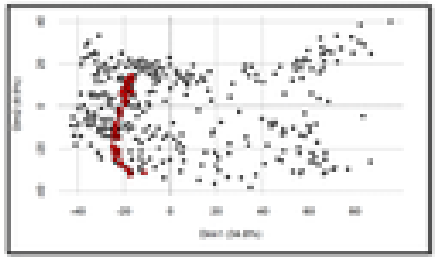
Example of Batch Effect – samples divided into two clouds due to a prompt problem during injections (RAW, not normalized data)



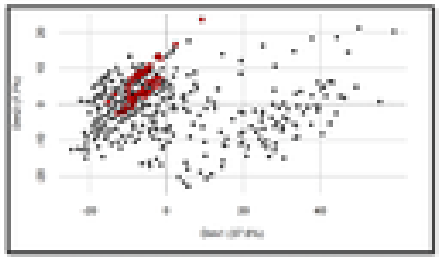
The same sample set normalized by:
1) median of each feature/plate = 1
2) Intensity of creatinine *m/z* feature



Example of Drift due to loss of signal intensity– Samples and QCs suffered from drop in the signal intensity during injections (RAW, not normalized data)

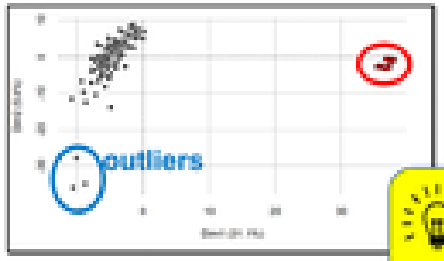


The same sample set normalized by:
1) median of each feature/plate = 1
2) Intensity of creatinine *m/z* feature



Example of QC prepared from study samples – a tight QCs cloud is located nearby a wide cloud of real samples (RAW, not normalized data)

Example of QC prepared from commercial biological fluid (i.e. plasma)– a tight QCs cloud is located far from a wider cloud of real samples (RAW, not normalized data). Three samples (outliers) are separated from the rest of samples (blue circle)



QC samples Study samples Outliers samples

Tips & Tricks

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Exemplos

Universidade Federal do Ceará
Departamento de Engenharia Química

UNIVERSITY OF COPENHAGEN

Conselho Nacional de Desenvolvimento Científico e Tecnológico

inct
institutos nacionais
de ciência e tecnologia

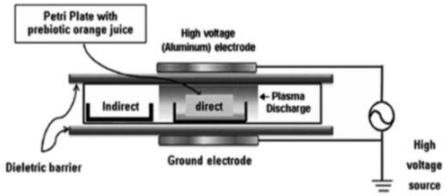
^{1}H NMR spectroscopy and chemometrics evaluation of non-thermal processing of orange juice

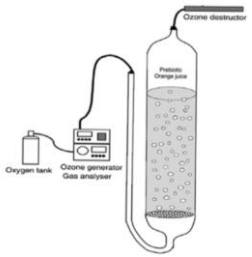
Amostra: Suco laranja

Processamento: Plasma e O_3

Análise: RMN ^{1}H

Quimiometria: PCA e quantificação (*q*NMR)

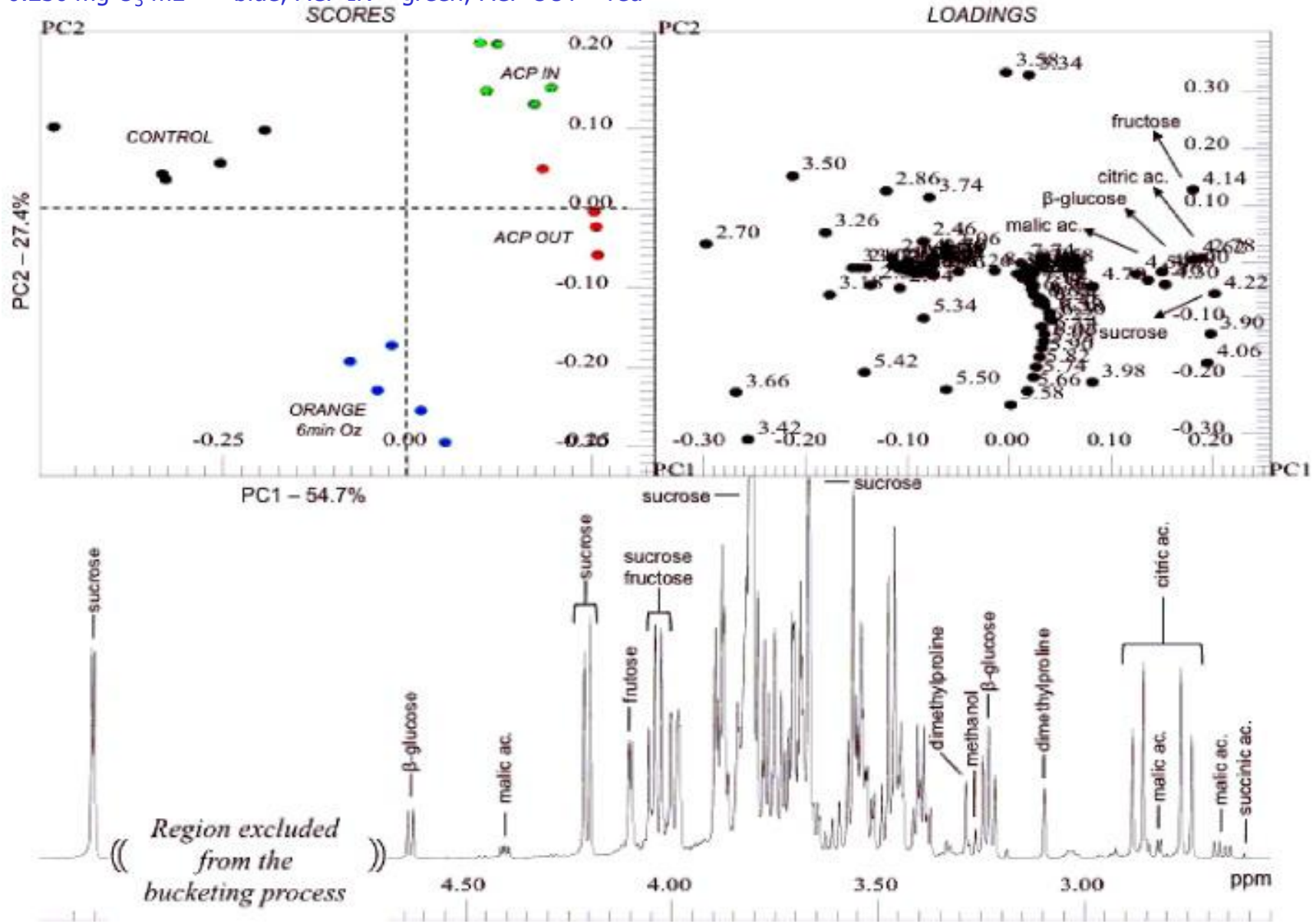




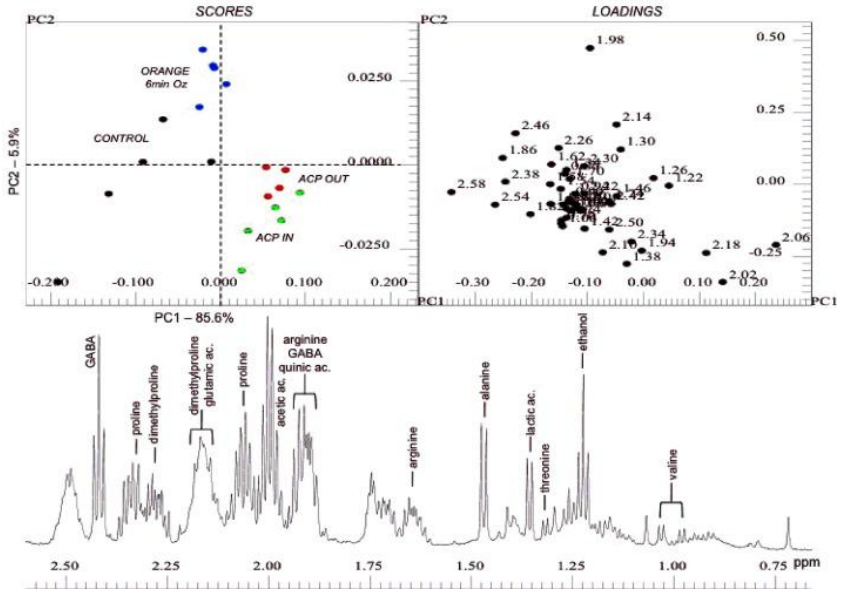
FOOD CHEMISTRY

Food Chemistry 204 (2016) 102–107

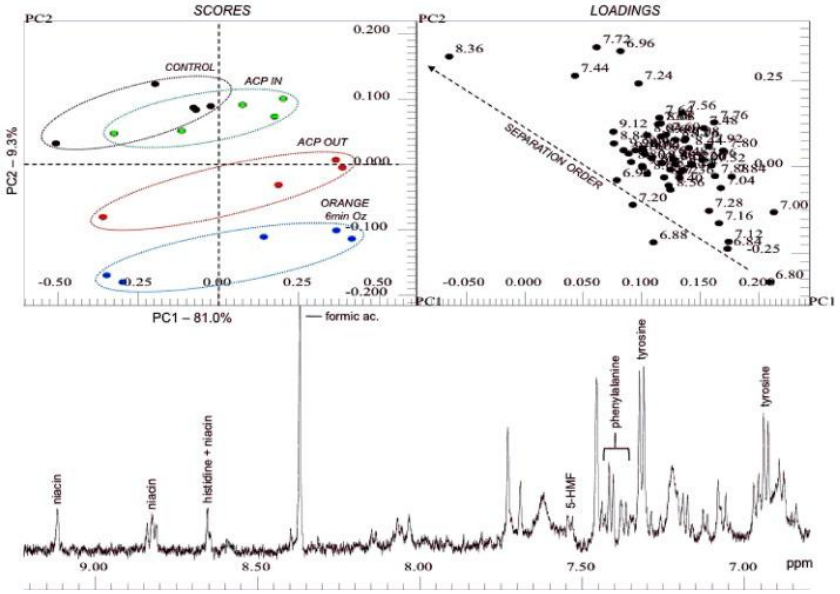
Fig. 1. ^{1}H NMR spectrum (C), PC1 *vs.* PC2 scores (left side – A) and loadings (right side – B) coordinate system for the orange juice submitted to different processing: control – black; 0.230 mg O_3 mL^{-1} – blue; ACP IN – green; ACP OUT – red



Região alifáticos



Região aromáticos



- The processes promoted slight variation in concentration of primary metabolites.

- The variations did not result in significant changes in orange juice composition.

- Plasma and ozone are suitable non-thermal alternatives for orange juice processing.

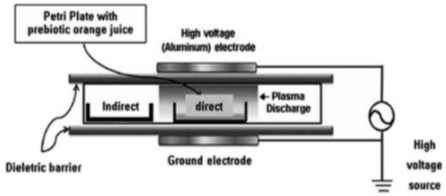
An untargeted chemometric evaluation of plasma and ozone processing effect on volatile compounds in orange juice

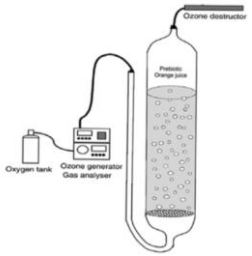
Amostra: Suco laranja

Processamento: Plasma e O_3

Análise: GC-MS

Quimiometria: análise hierárquica + PCA





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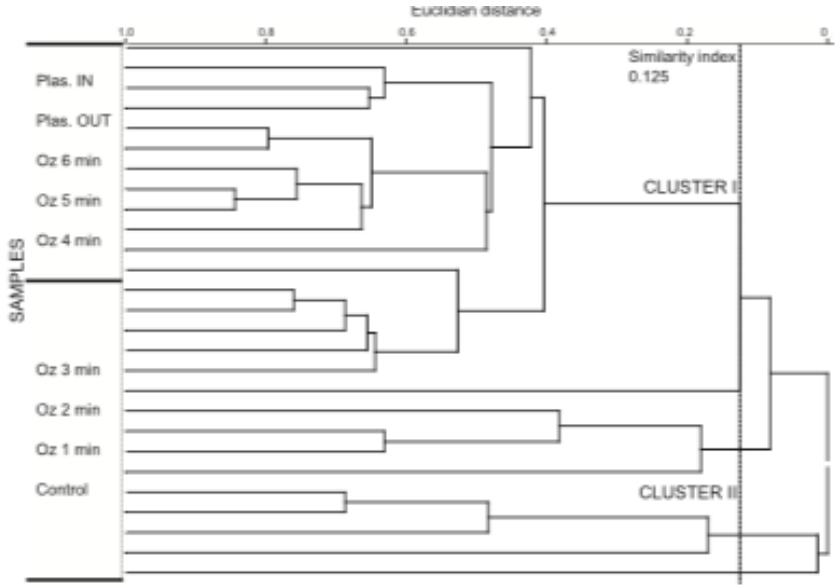
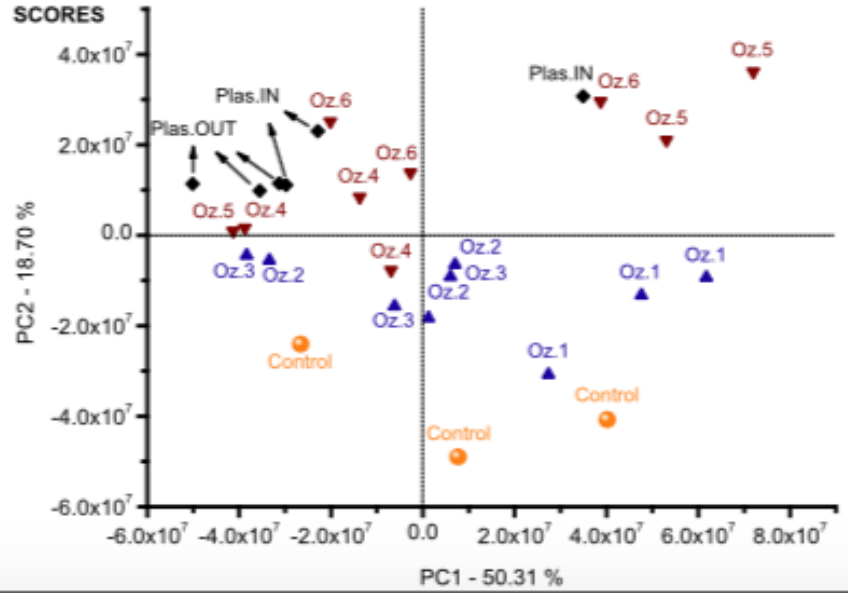
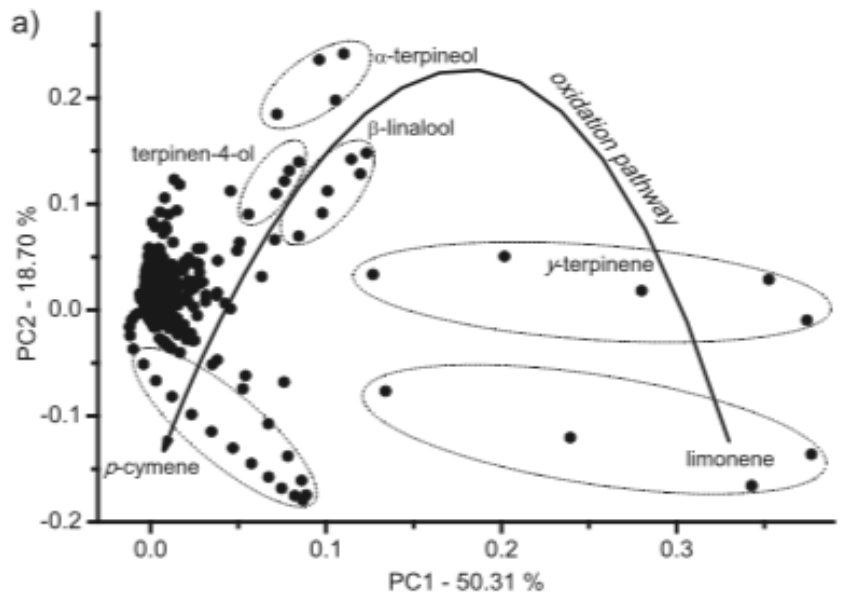


Fig. 2. Dendrogram representing chemical composition similarity relationships among orange juices to whose cluster it belongs: I) plasma IN, plasma OUT, and 4–6 min ozone; II) control, and 1–3 min ozone.





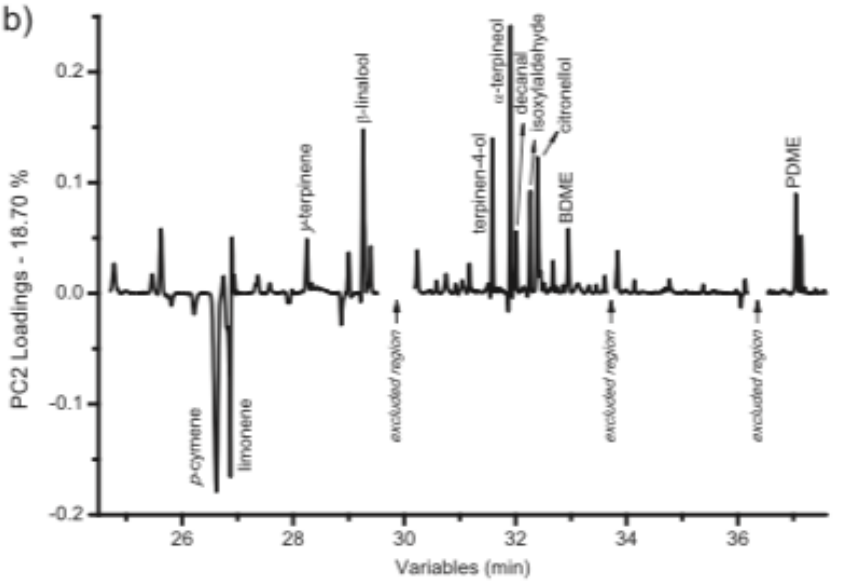


Fig. 4. PC1 vs PC2 loadings plotted in two dimensions (a); and PC2 plotted in line (b). The excluded regions reflect interfering signals removed for chemometrics.

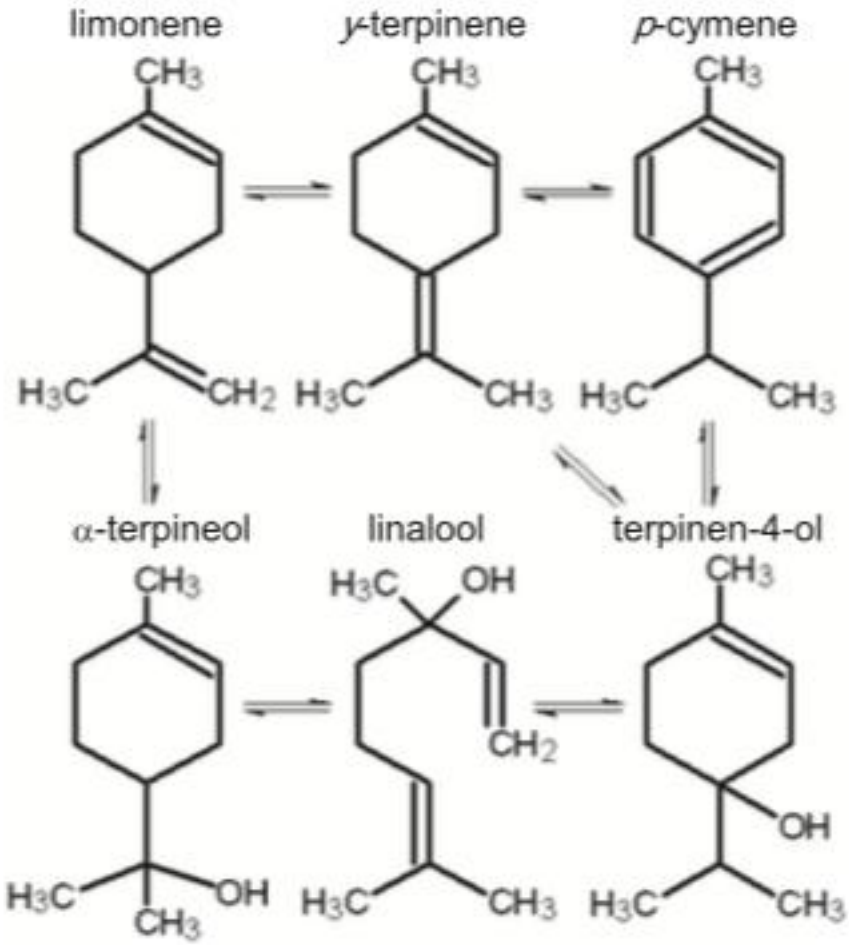


Fig. 5. Proposed oxidation, hydrolysis and reduction of terpenes under plasma and ozone processing of orange juice.

NMR spectroscopy and chemometrics to evaluate different processing of coconut water

Amostra: água de coco

Processamento: térmico (UHT) + com/sem SO_2

Análise: RMN ^1H

Quimiometria: PCA e quantificação (*q*NMR)

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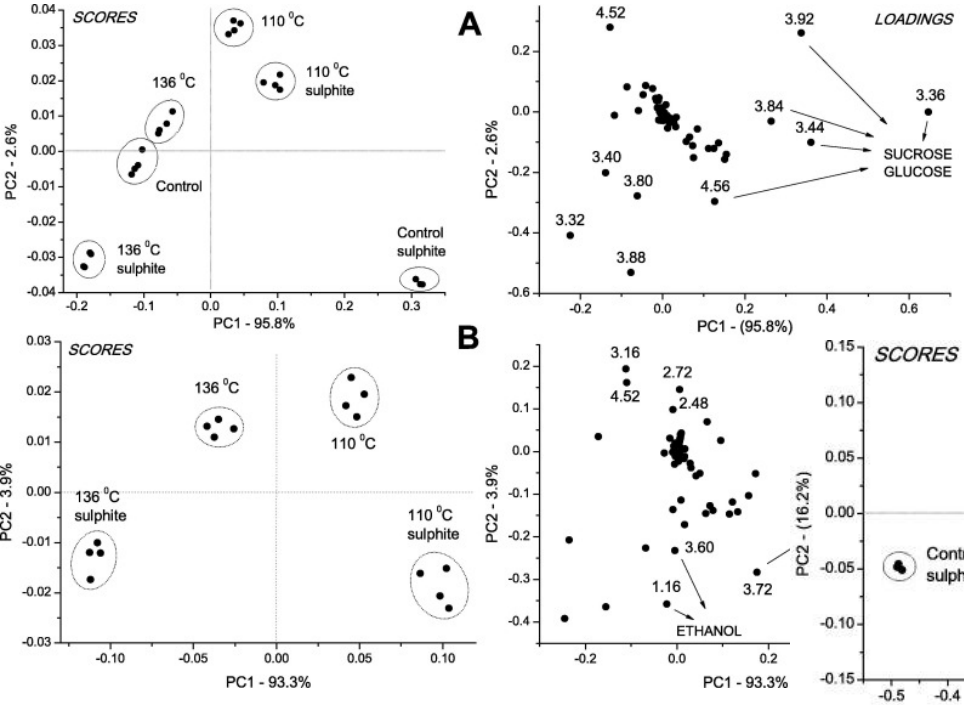
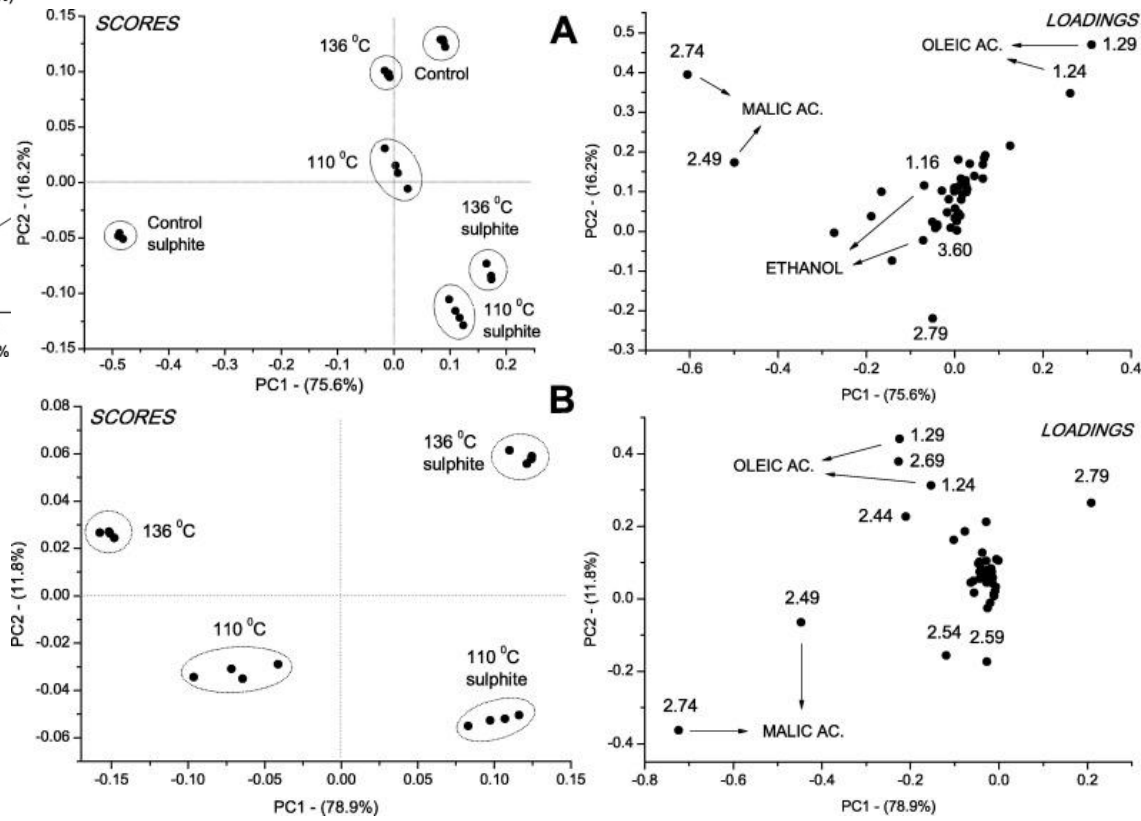
Completo



Alifático



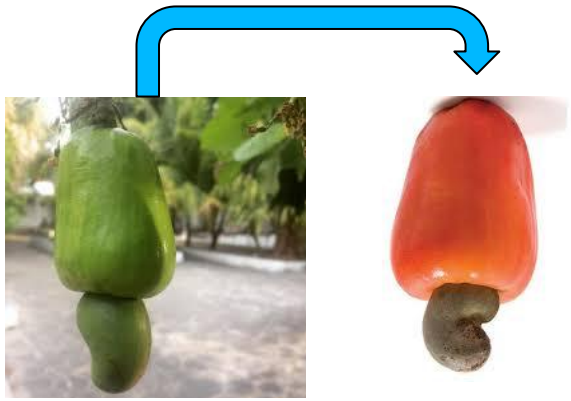
UPLC–qTOF-MS/MS-based phenolic profile and their biosynthetic enzyme activity used to discriminate between cashew apple (*Anacardium occidentale* L.) maturation stages

Amostra: caju

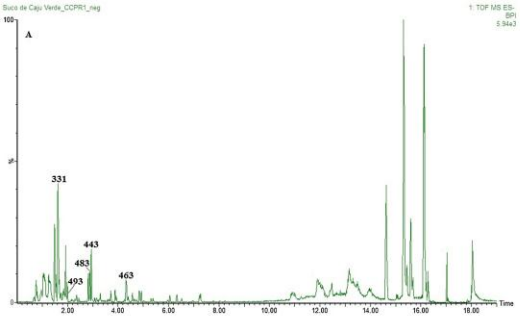
Processamento: diferentes estádios de maturação

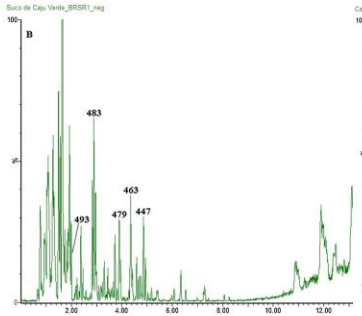
Análise: LC-MS

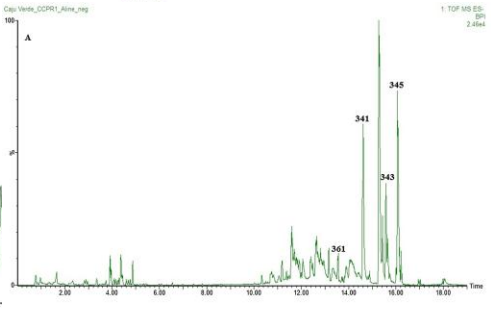
Quimiometria: PCA e OPLS-DA

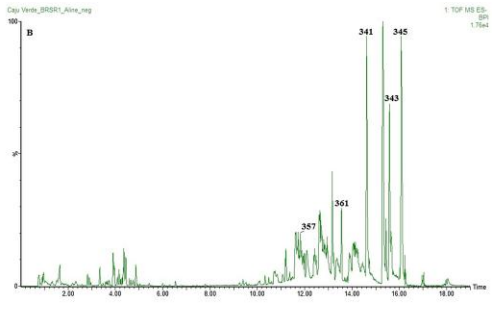


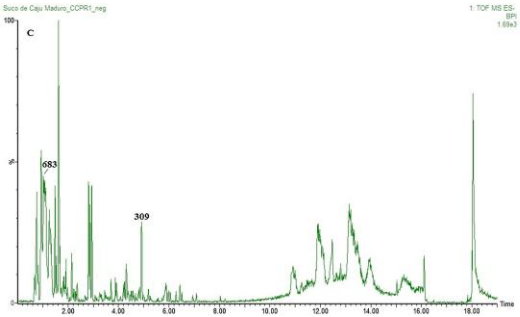
Journal of Chromatography B, 1051 (2017) 24–32

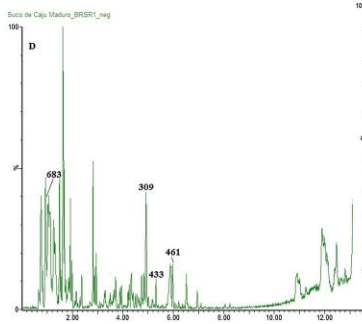

A
331
443
483
493
463

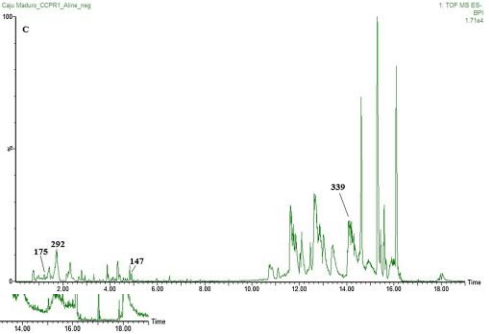

B
483
493
479
463
447

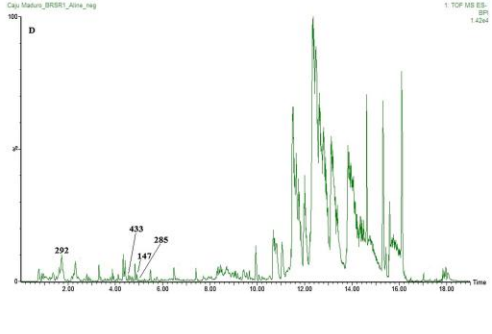

A
345
341
343
361

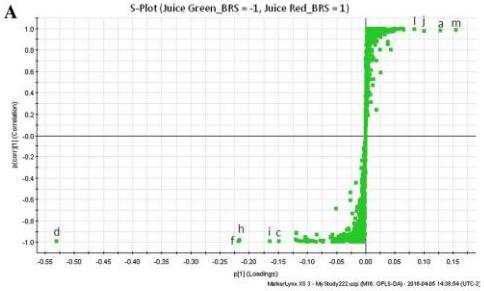

B
341
345
343
361
357

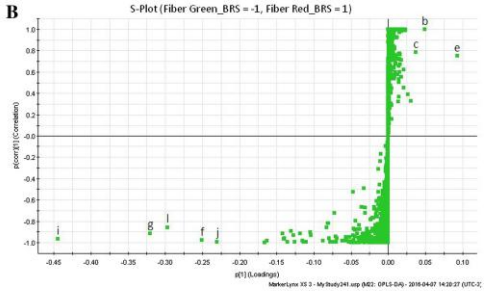

C
683
309

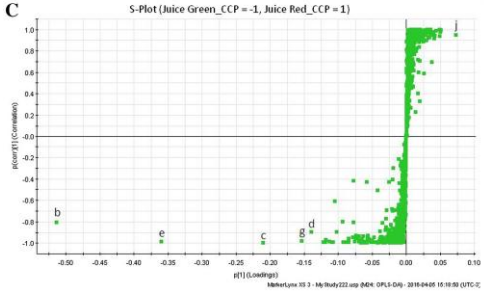

D
683
309
461
433

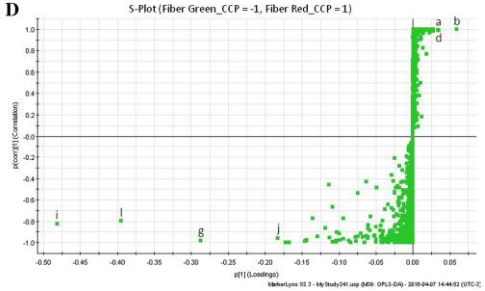

C
339
292
175
147


D
433
285
147
292


A
S-Plot (Juice Green_BRS = -1, Juice Red_BRS = 1)
p(corr)[1] (Correlation)
p[1] (Loadings)


B
S-Plot (Fiber Green_BRS = -1, Fiber Red_BRS = 1)
p(corr)[1] (Correlation)
p[1] (Loadings)


C
S-Plot (Juice Green_CCP = -1, Juice Red_CCP = 1)
p(corr)[1] (Correlation)
p[1] (Loadings)


D
S-Plot (Fiber Green_CCP = -1, Fiber Red_CCP = 1)
p(corr)[1] (Correlation)
p[1] (Loadings)

cinnamoyl glucoside may be used as a chemical marker stage 7-ripe juice samples, while monogalloyl diglucoside (c) and digalloyl glucoside (d) as chemical markers of stage 2-green juice samples from both cashew clones. Meanwhile, four compounds could be used as markers for stage 2-green fiber of both clones, anacardic acids (i, j and l) and GA_{19} (g) which have low solubilityin aqueous solutions due to their chemical natures as phenolic lipidsand a diterpenoid acid, respectively.

The evaluation of enzymes of phenolic biosynthetic pathway in juice samples showed that PAL activity decreased significantly during the development of cashew apple clones, despite of which, it was much higher in ripe CCP 76 cashew apple. UGT activity differed between clones, however its main product, cinnamoyl glucoside was the most characteristic compound, thus a chemical marker of ripe juice samples from both clones. FLS showed the highest specific activity in both cashew clones and its product, flavonols (glycosylated probably due to UGT action), were identified in cashew apple at immature and ripe stages. LAR activity was not detected during the development of cashew apple from clones BRS 189 and CCP 76 agreeing with previous publications [12], [13] which found no LAR substrates, flavan-3-ols or proanthocyanindins in cashew apples.

Chemical profiling of guarana seeds (*Paullinia cupana*) from different geographical origins using UPLC-QTOF-MS combined with chemometrics

^{1}H quantitative nuclear magnetic resonance and principal component analysis as tool for discrimination of guarana seeds from different geographic regions of Brazil

Amostra: guaraná

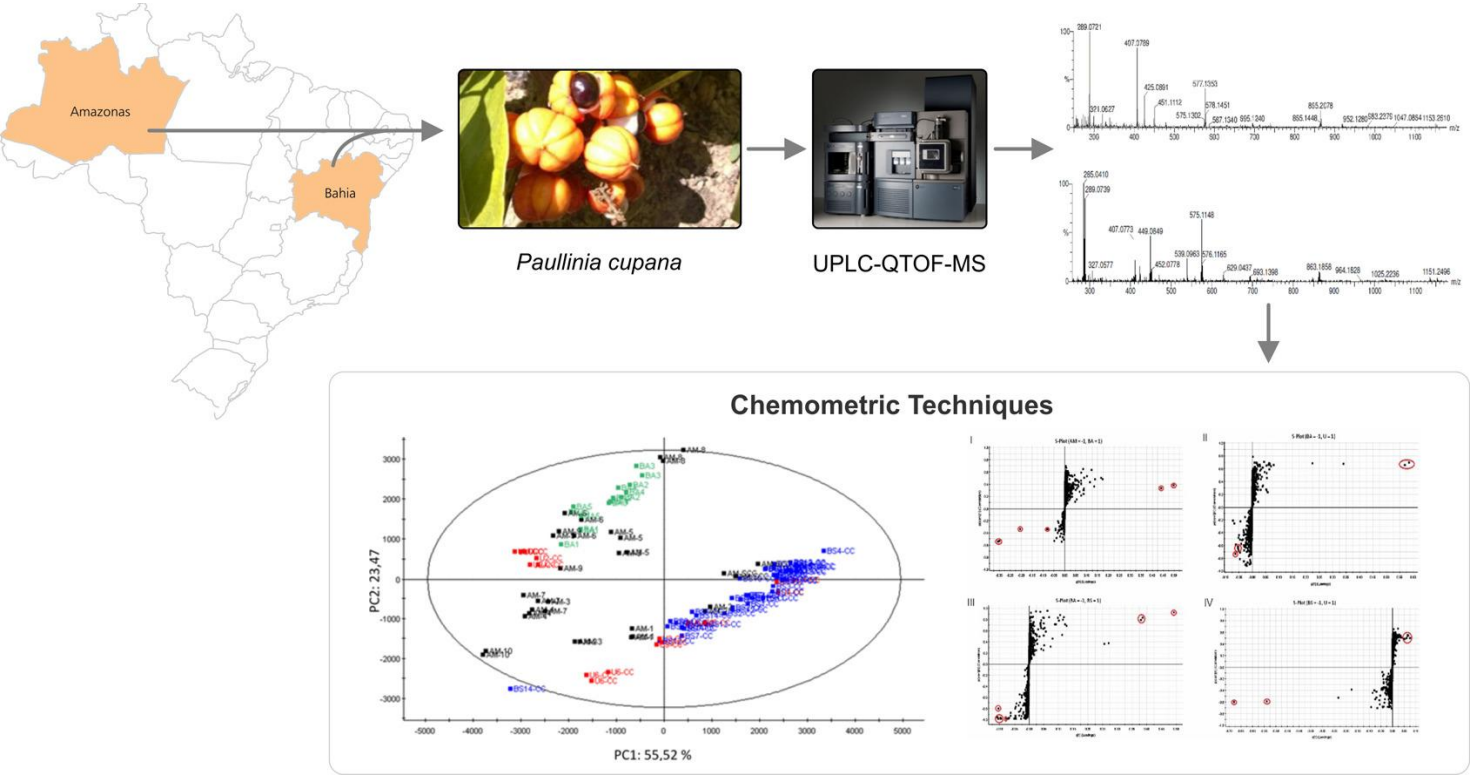
Processamento: diferentes origens

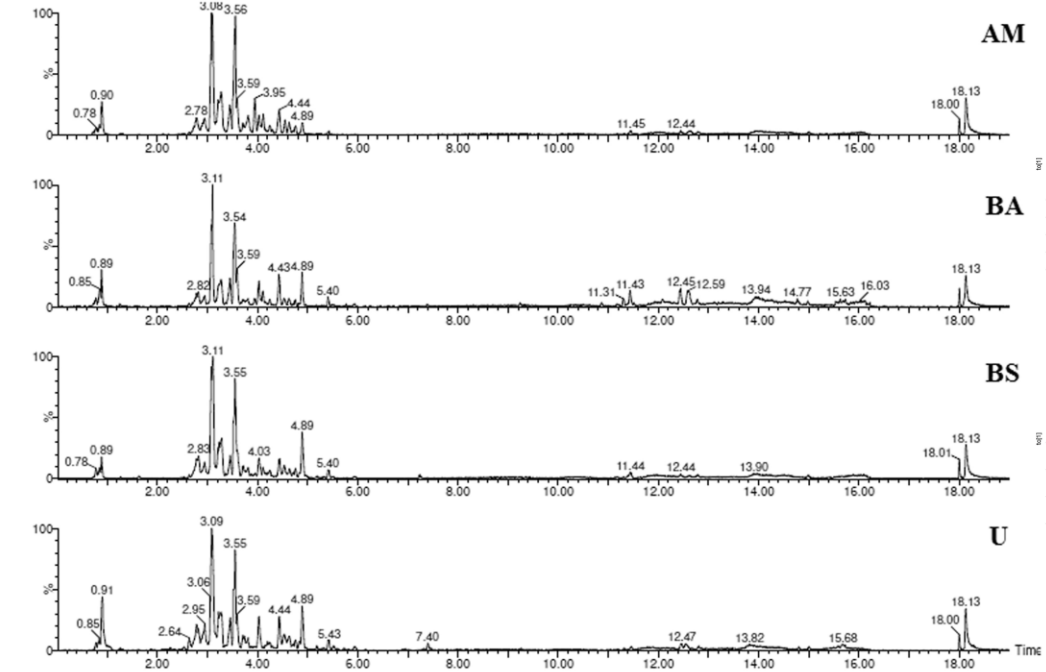
Análise: LC-MS e RMN ^{1}H

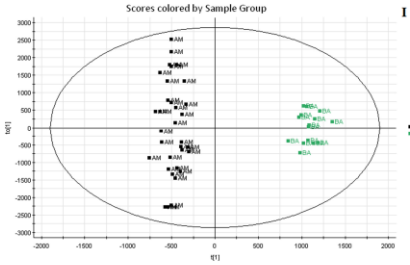
Quimiometria: PCA e quantificação

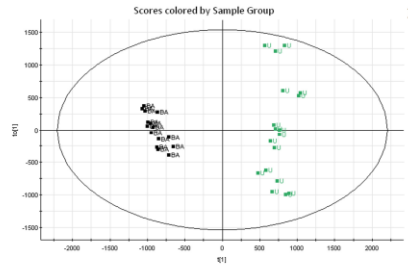
Food Research International

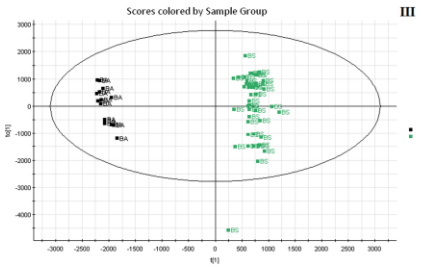
Magnetic Resonance in Food Science 2016 Proceedings

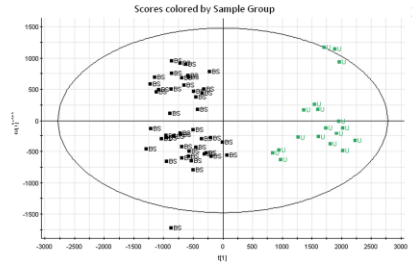

Amazonas
Bahia
Paullinia cupana
UPLC-QTOF-MS
Chemometric Techniques
PC2: 23,47
PC1: 55,52 %

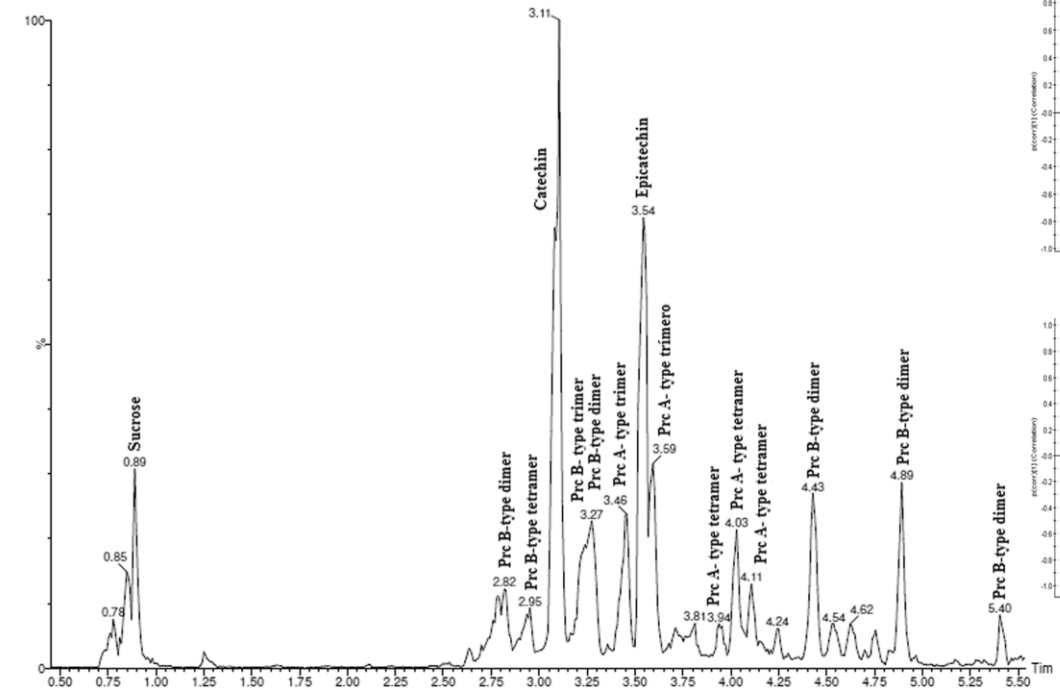

AM
BA
BS
U
Time

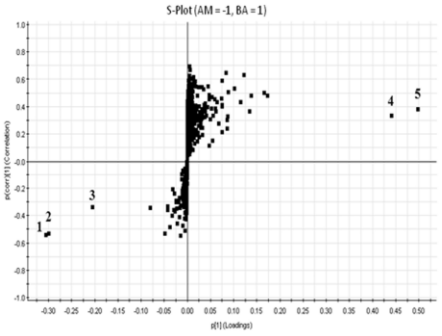

Scores colored by Sample Group
I
AM
BA

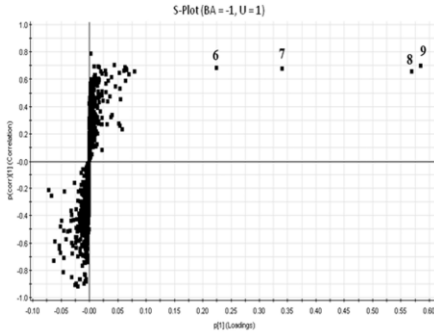

Scores colored by Sample Group
II
BA
U

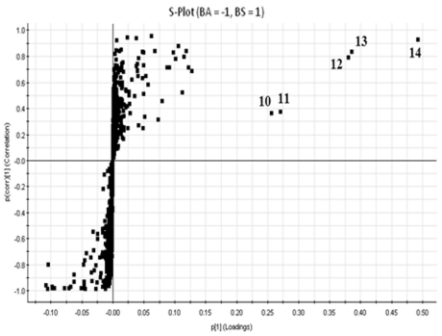

Scores colored by Sample Group
III
BA
BS

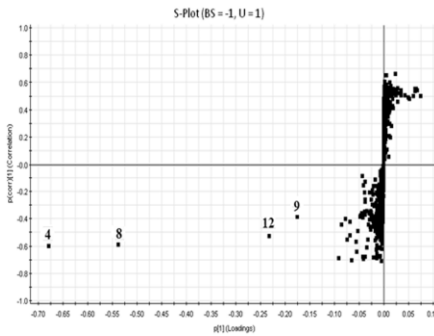

Scores colored by Sample Group
IV
BS
U

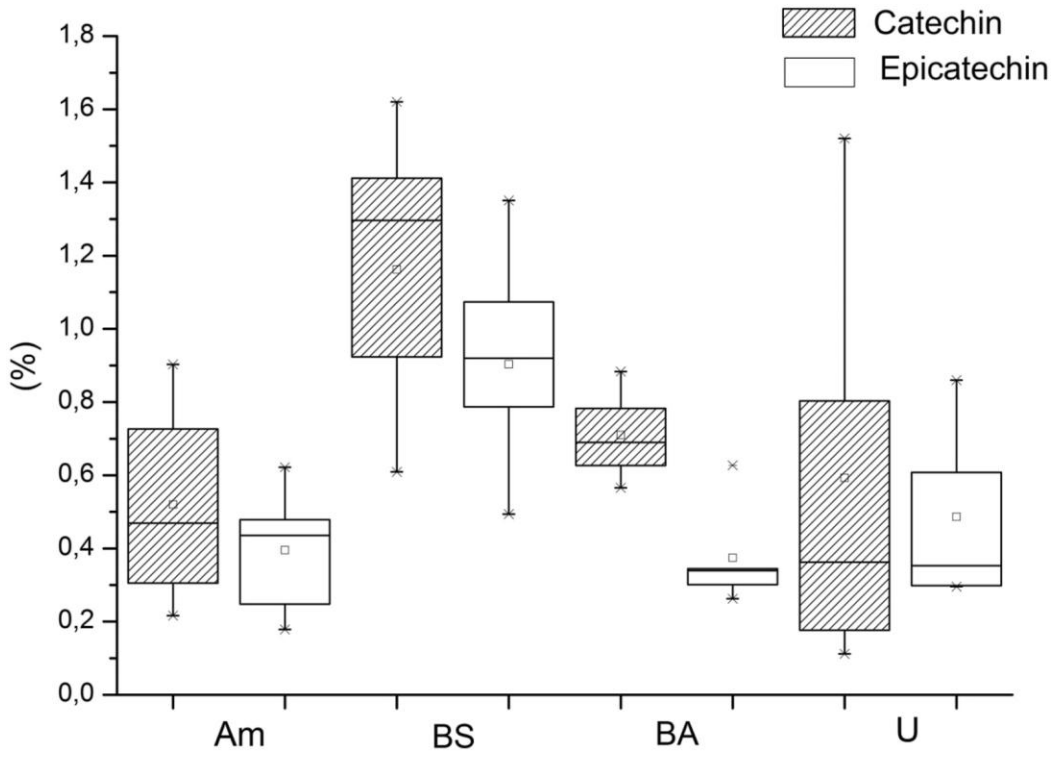

Sucrose
Catechin
Epicatechin
Prc B-type dimer
Prc B-type tetramer
Prc B- type trimer
Prc B-type dimer
Prc A- type trimer
Prc A- type trimero
Prc A- type tetramer
Prc A- type tetramer
Prc A- type tetramer
Prc B-type dimer
Prc B-type dimer
Prc B-type dimer
Time


S-Plot (AM = -1, BA = 1)


S-Plot (BA = -1, U = 1)


S-Plot (BA = -1, BS = 1)


S-Plot (BS = -1, U = 1)


Catechin
Epicatechin
(%)
1,8
1,6
1,4
1,2
1,0
0,8
0,6
0,4
0,2
0,0
Am
BS
BA
U

Genotype evaluation of cowpea seeds (*Vigna unguiculata*) using ^{1}H *q*NMR combined with exploratory tools and solid-state NMR

Amostra: feijão caupi

Processamento: (9 genótipos)

Análise: RMN ^{1}H

Quimiometria: PCA e quantificação (*q*NMR)

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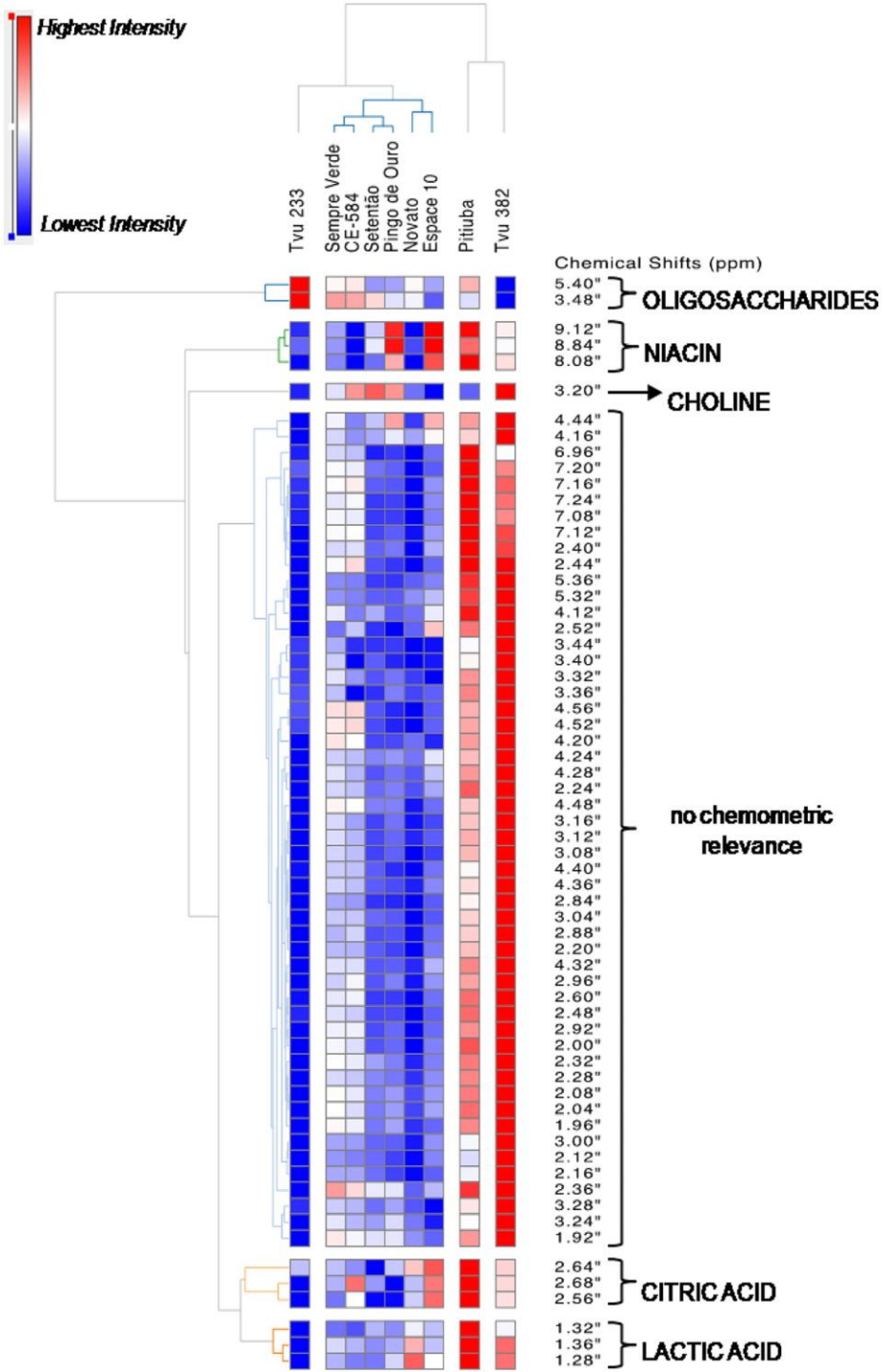
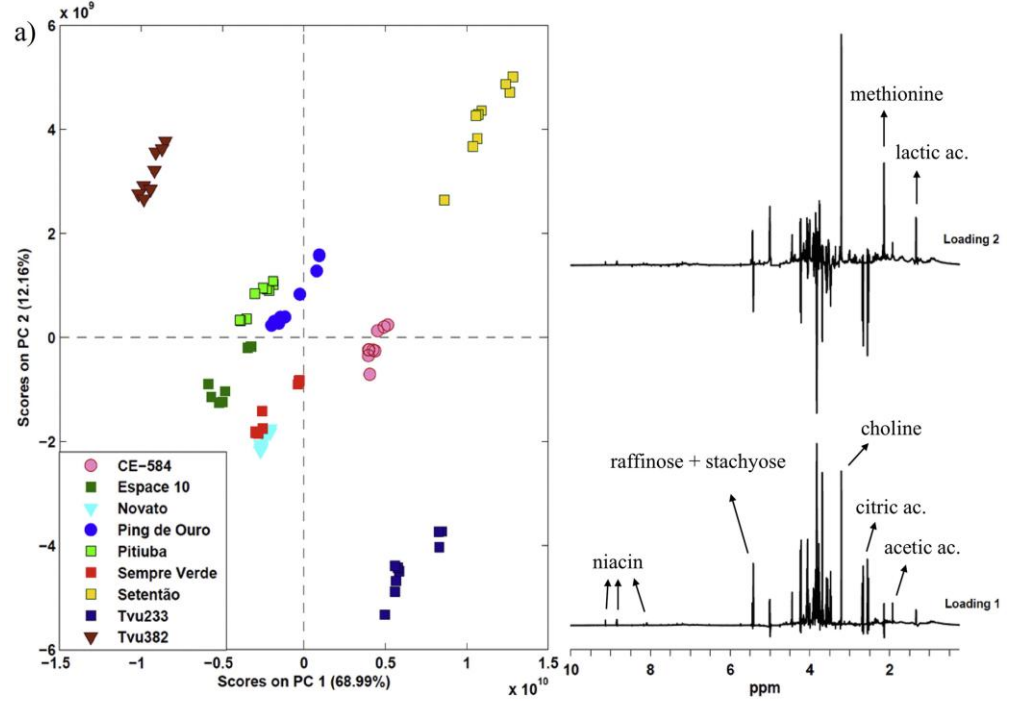
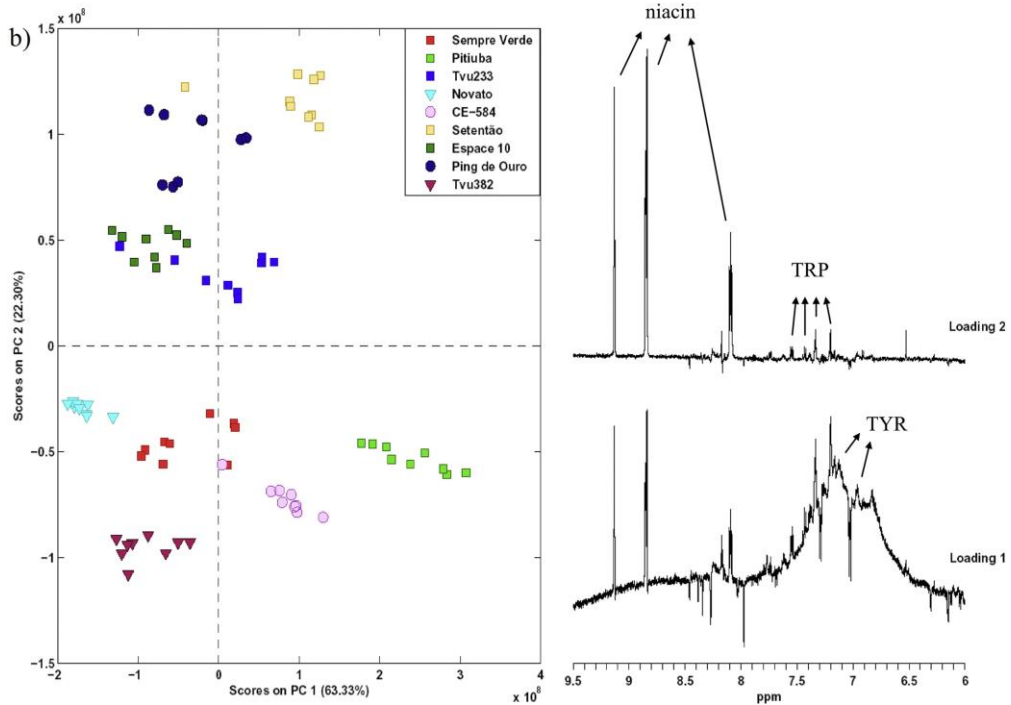
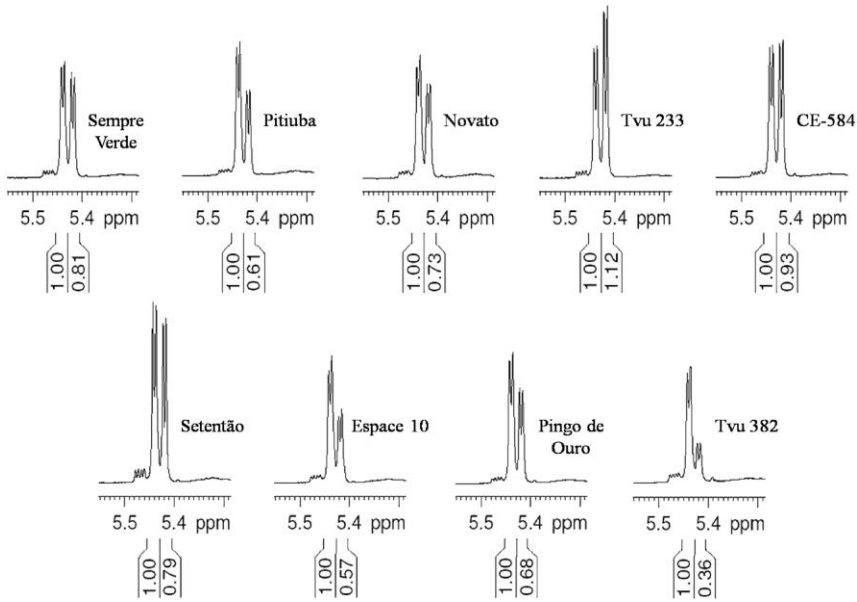

Highest Intensity
Lowest Intensity
Tvu 233
Sempre Verde
CE-584
Setentão
Pingo de Ouro
Novato
Espace 10
Pitiuba
Tvu 382
Chemical Shifts (ppm)
5.40"
3.48"
OLIGOSACCHARIDES
9.12"
8.84"
8.08"
NIACIN
3.20"
CHOLINE
4.44"
4.16"
6.96"
7.20"
7.16"
7.24"
7.08"
7.12"
2.40"
2.44"
5.36"
5.32"
4.12"
2.52"
3.44"
3.40"
3.32"
3.36"
4.56"
4.52"
4.20"
4.24"
4.28"
2.24"
4.48"
3.16"
3.12"
3.08"
4.40"
4.36"
2.84"
3.04"
2.88"
2.20"
4.32"
2.96"
2.60"
2.48"
2.92"
2.00"
2.32"
2.28"
2.08"
2.04"
1.96"
3.00"
2.12"
2.16"
2.36"
3.28"
3.24"
1.92"
no chemometric relevance
2.64"
2.68"
2.56"
CITRIC ACID
1.32"
1.36"
1.28"
LACTIC ACID


a)
x 10^9
Scores on PC 2 (12.16%)
Scores on PC 1 (68.99%)
x 10^10
CE−584
Espace 10
Novato
Ping de Ouro
Pitiuba
Sempre Verde
Setentão
Tvu233
Tvu382
methionine
lactic ac.
Loading 2
choline
raffinose + stachyose
citric ac.
acetic ac.
niacin
Loading 1
ppm


b)
x 10^8
Scores on PC 2 (22.30%)
Scores on PC 1 (63.33%)
x 10^8
Sempre Verde
Pitiuba
Tvu233
Novato
CE−584
Setentão
Espace 10
Ping de Ouro
Tvu382
niacin
TRP
Loading 2
TYR
Loading 1
ppm


Sempre Verde
Pitiuba
Novato
Tvu 233
CE-584
Setentão
Espace 10
Pingo de Ouro
Tvu 382
5.5 5.4 ppm
1.00 0.81
1.00 0.61
1.00 0.73
1.00 1.12
1.00 0.93
1.00 0.79
1.00 0.57
1.00 0.68
1.00 0.36

Tracking thermal degradation on passion fruit juice through Nuclear Magnetic Resonance and chemometrics

Amostra: suco maracujá

Processamento: térmico UHT e HTST

Análise: RMN ^{1}H

Quimiometria: PCA e quantificação (*q*NMR)

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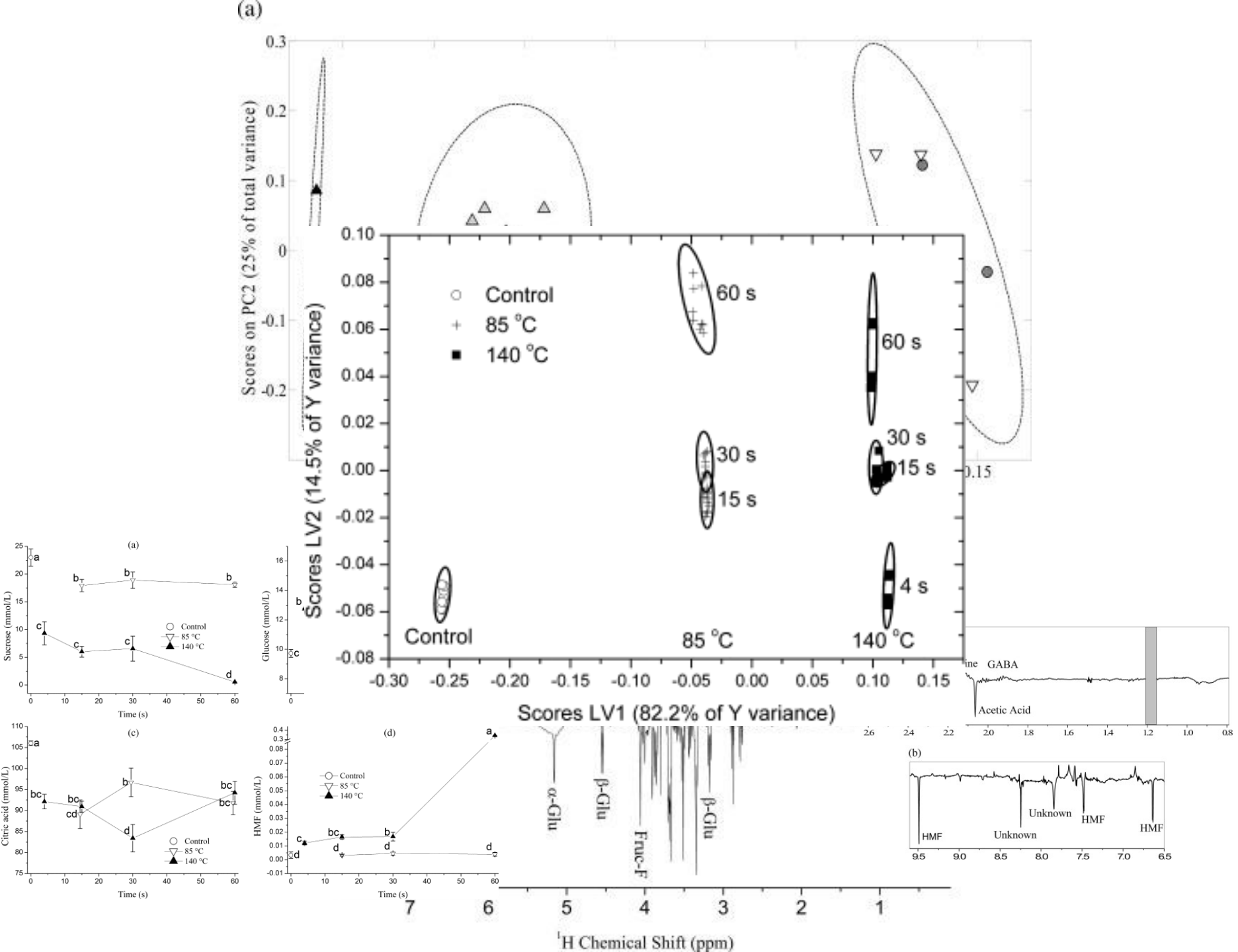

(a)
Scores on PC2 (25% of total variance)
Scores LV2 (14.5% of Y variance)
Scores LV1 (82.2% of Y variance)
Control
85 °C
140 °C
60 s
30 s
15 s
4 s
(a)
Sucrose (mmol/L)
Time (s)
(b)
Glucose (mmol/L)
(c)
Citric acid (mmol/L)
(d)
HMF (mmol/L)
α-Glu
β-Glu
Fruc-F
β-Glu
GABA
Acetic Acid
Unknown
HMF
Unknown
HMF
HMF
¹H Chemical Shift (ppm)

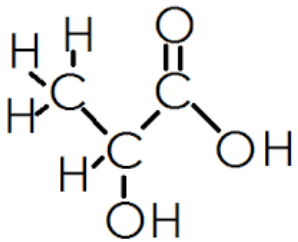
Chemometric evaluation of the volatile profile of probiotic melon and probiotic cashew juice

Amostra: suco melão e caju

Processamento: probiotico + térmico UHT e HTST

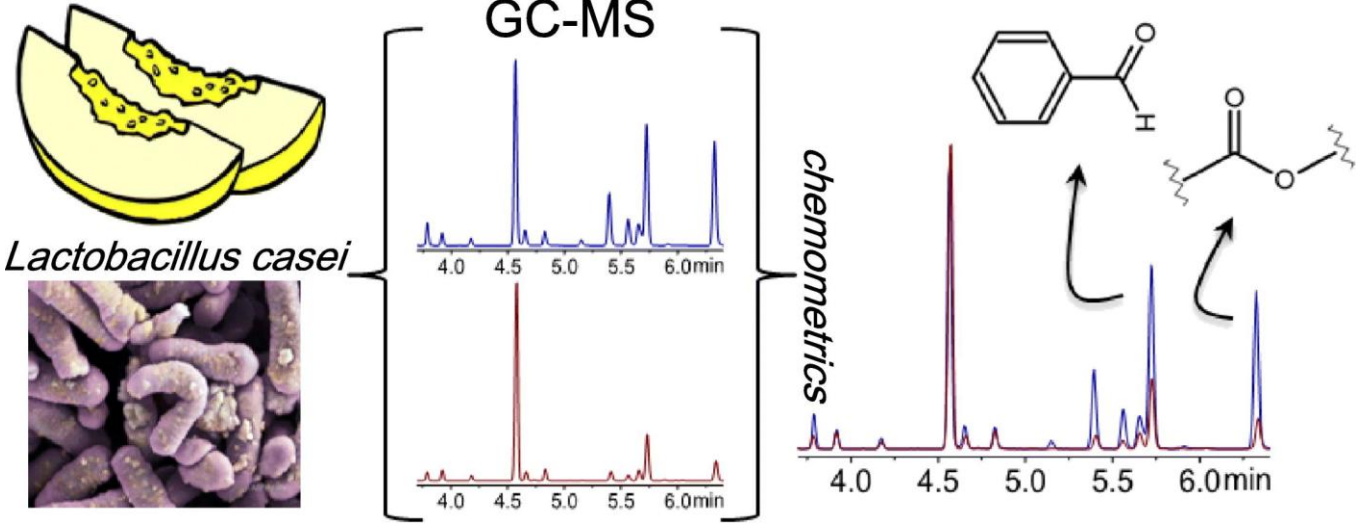
Análise: GC-MS

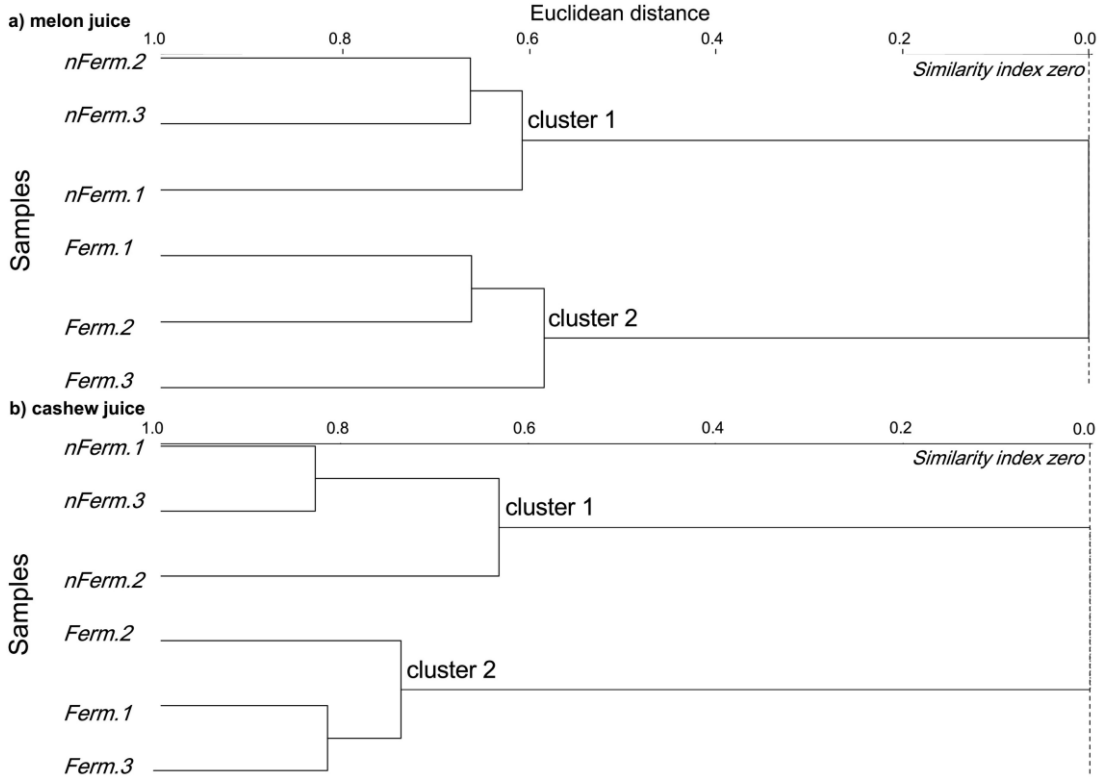
Quimiometria: PCA e quantificação (*q*NMR)

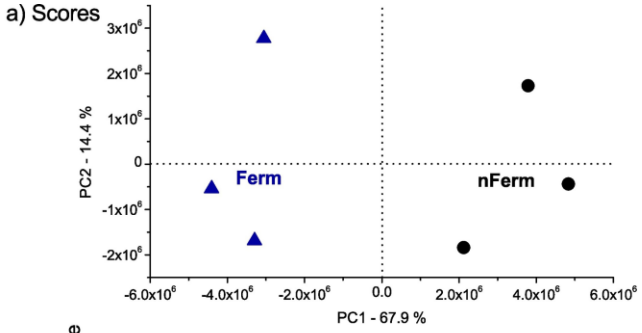


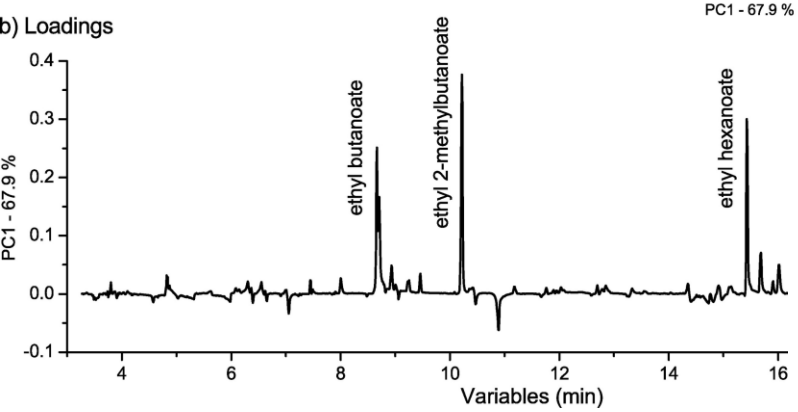
Lactic Acid

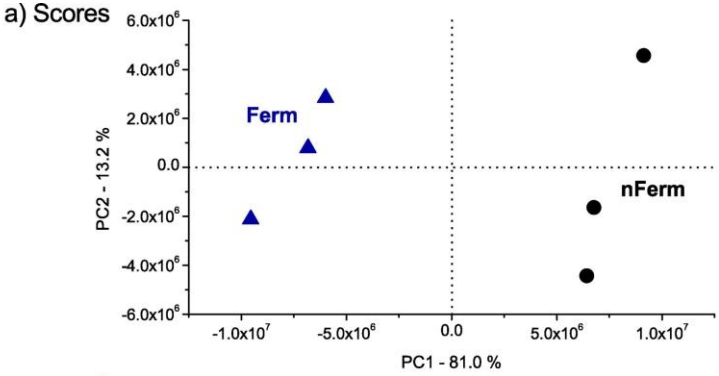
Food Research International 99 (2017) 461–468

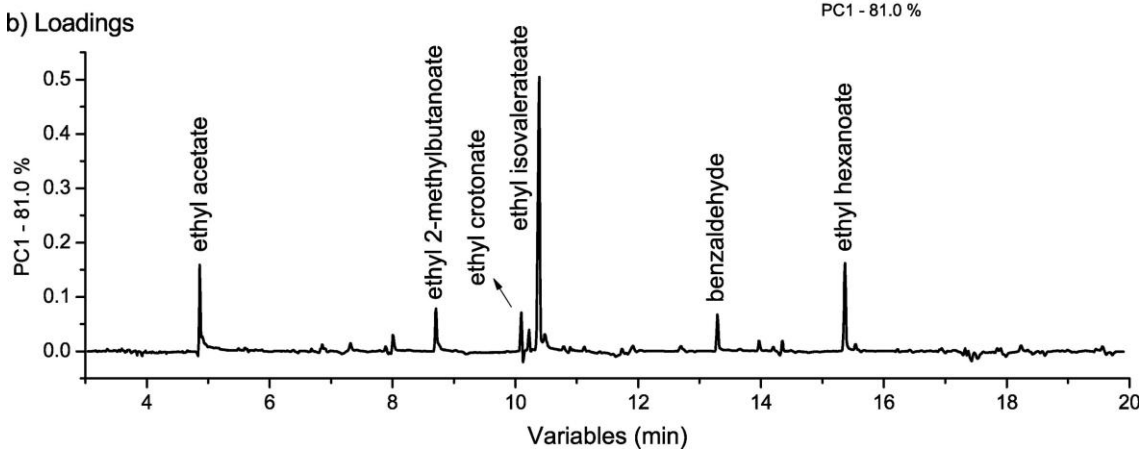

GC-MS
Lactobacillus casei
4.0 4.5 5.0 5.5 6.0min
4.0 4.5 5.0 5.5 6.0min
chemometrics
4.0 4.5 5.0 5.5 6.0min

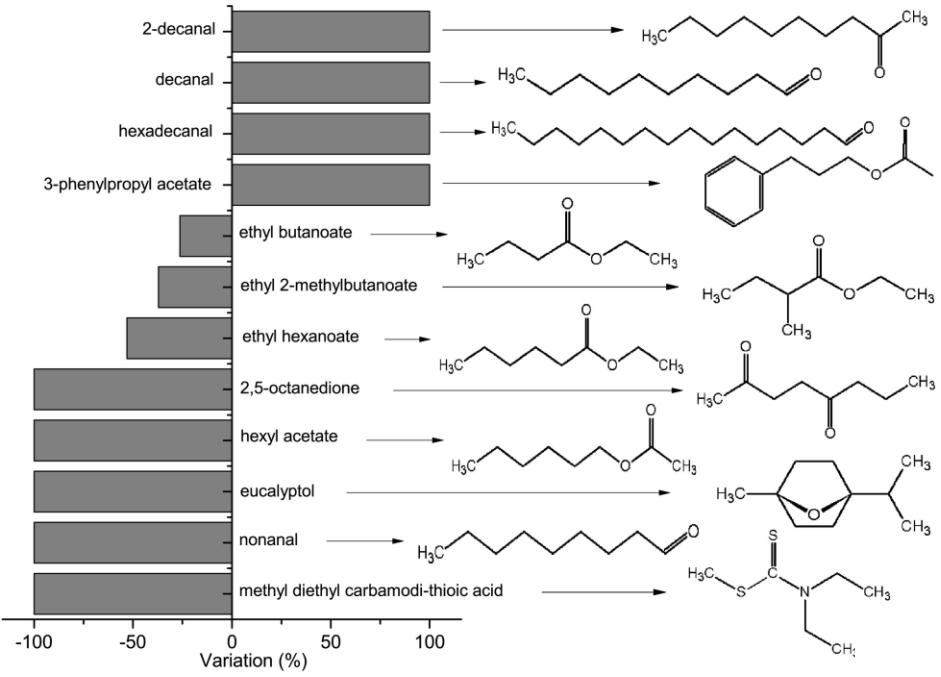

a) melon juice
Euclidean distance
1.0 0.8 0.6 0.4 0.2 0.0
Similarity index zero
nFerm.2
nFerm.3
nFerm.1
Ferm.1
Ferm.2
Ferm.3
Samples
cluster 1
cluster 2
b) cashew juice
1.0 0.8 0.6 0.4 0.2 0.0
Similarity index zero
nFerm.1
nFerm.3
nFerm.2
Ferm.2
Ferm.1
Ferm.3
Samples
cluster 1
cluster 2

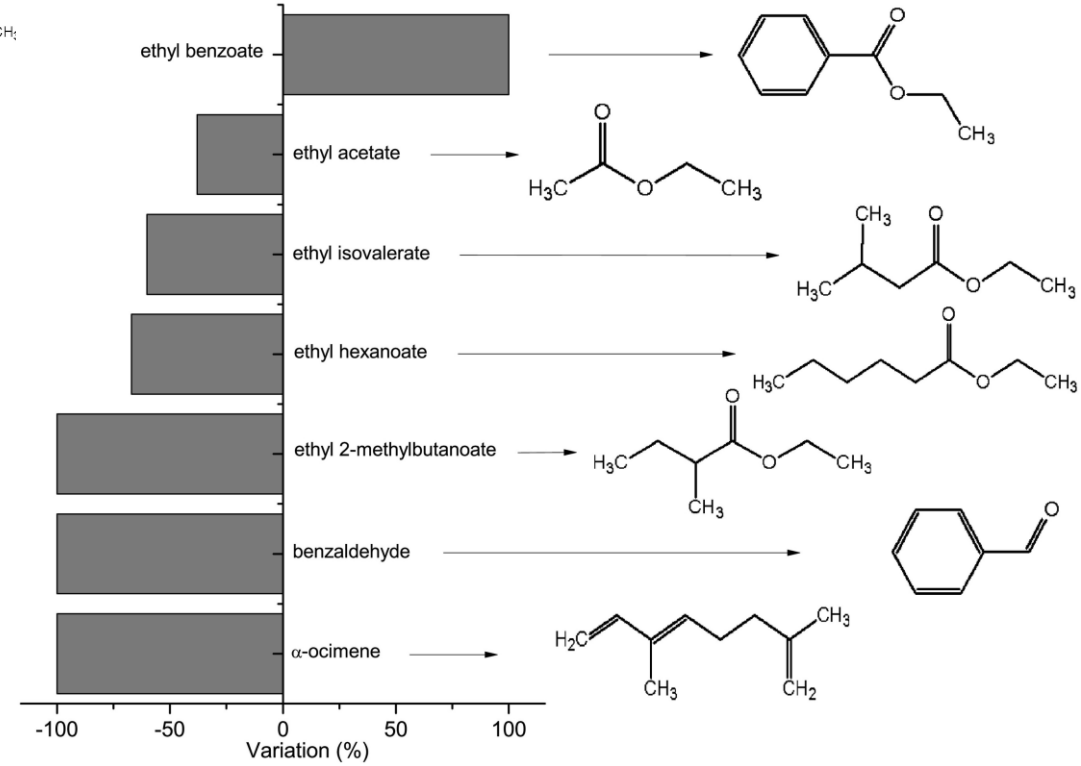

a) Scores
PC2 - 14.4 %
PC1 - 67.9 %
Ferm
nFerm


b) Loadings
PC1 - 67.9 %
ethyl butanoate
ethyl 2-methylbutanoate
ethyl hexanoate
Variables (min)


a) Scores
PC2 - 13.2 %
PC1 - 81.0 %
Ferm
nFerm


b) Loadings
PC1 - 81.0 %
ethyl acetate
ethyl 2-methylbutanoate
ethyl crotonate
ethyl isovalerateate
benzaldehyde
ethyl hexanoate
Variables (min)


2-decanal
decanal
hexadecanal
3-phenylpropyl acetate
ethyl butanoate
ethyl 2-methylbutanoate
ethyl hexanoate
2,5-octanedione
hexyl acetate
eucalyptol
nonanal
methyl diethyl carbamodi-thioic acid
-100
-50
0
50
100
Variation (%)


ethyl benzoate
ethyl acetate
ethyl isovalerate
ethyl hexanoate
ethyl 2-methylbutanoate
benzaldehyde
α-ocimene
-100
-50
0
50
100
Variation (%)

Evaluation of thermal and non-thermal processing effect on non-prebiotic and prebiotic acerola juices using ^{1}H *q*NMR and GC–MS coupled to chemometrics

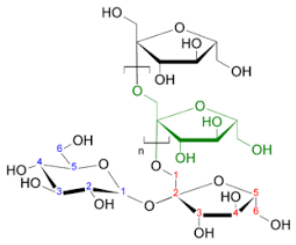
Amostra: suco acerola

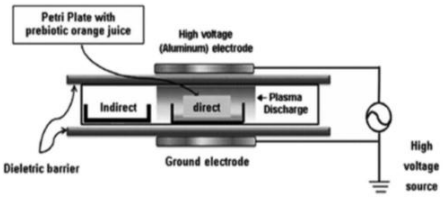
Processamento: prébiotico + térmico UHT e HTST + US+ Plasma

Análise: RMN ^{1}H + GC-MS

Quimiometria: PCA e quantificação (*q*NMR)

+





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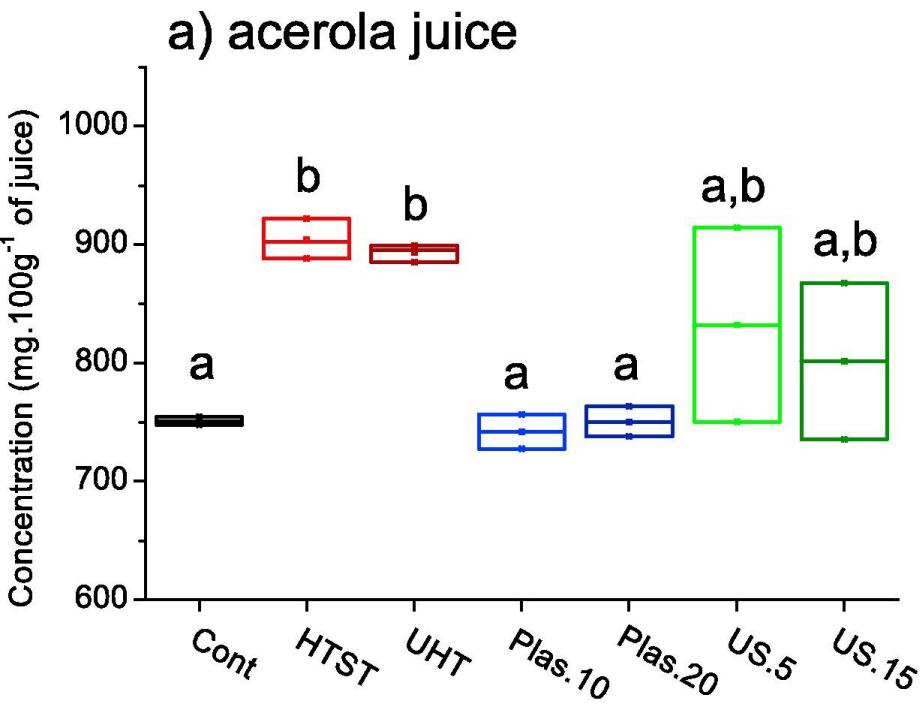
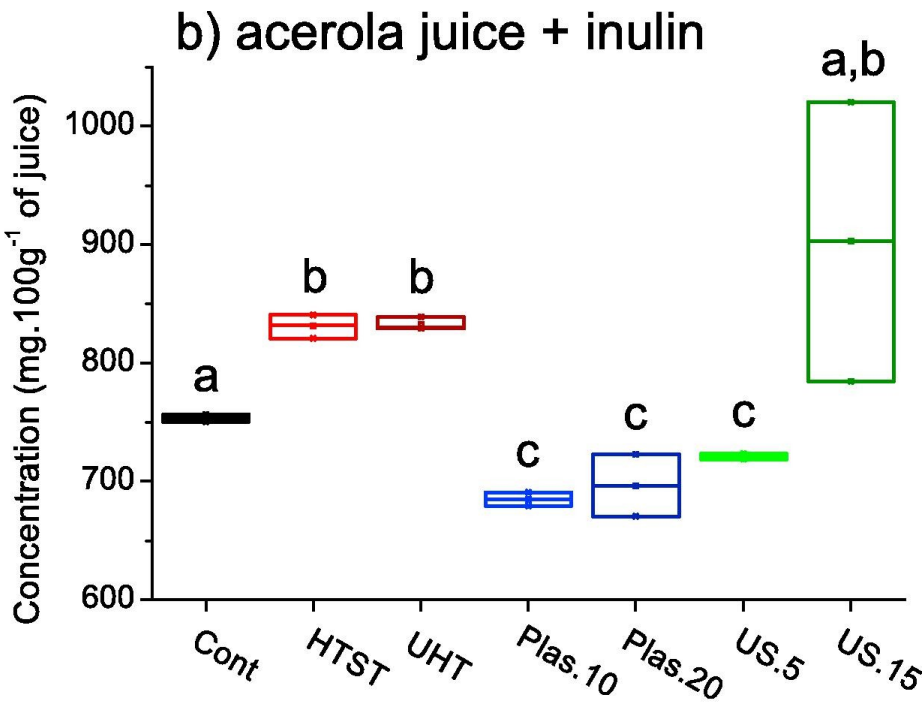
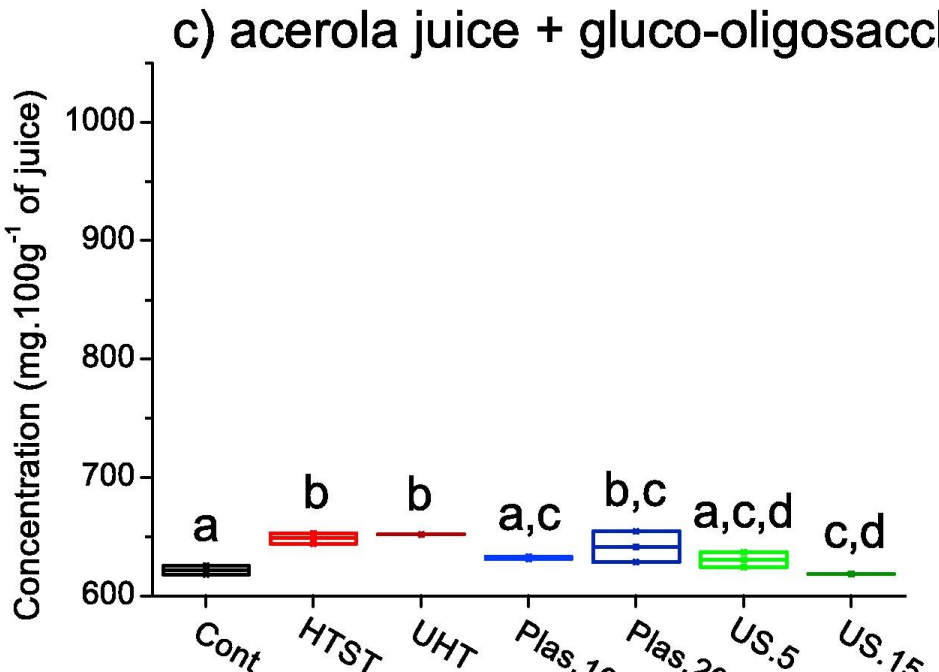
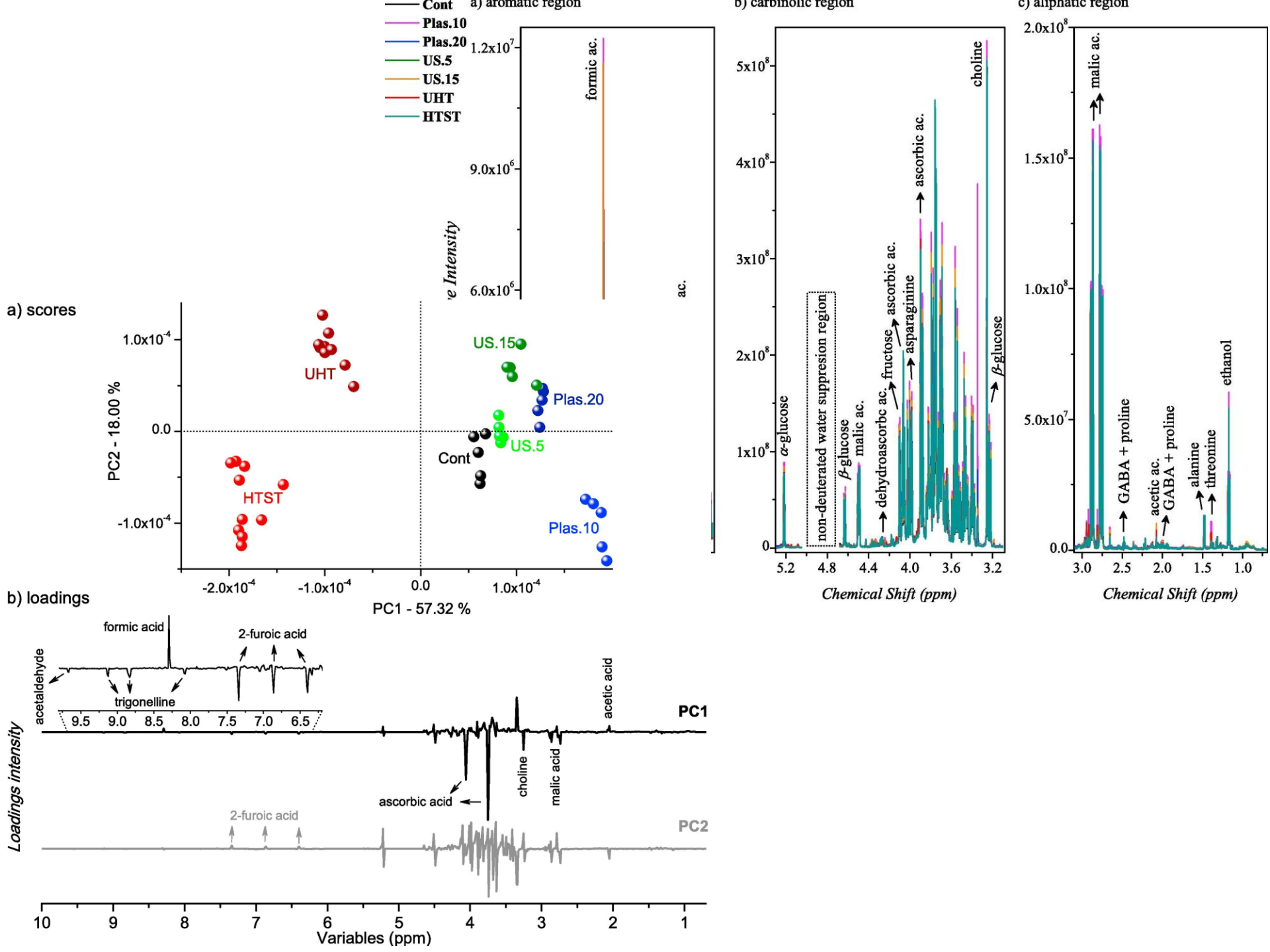
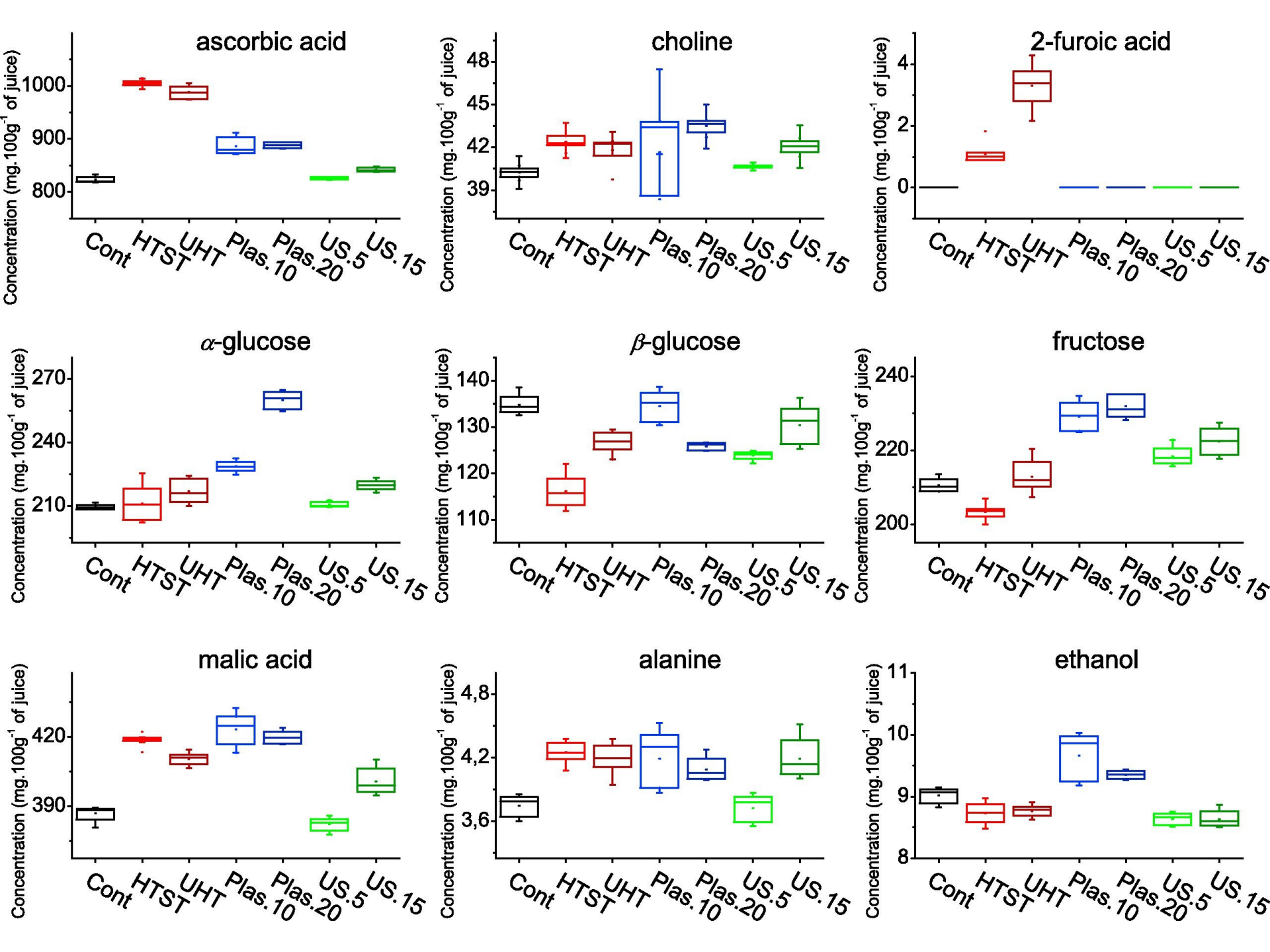
Vitamina C


a) acerola juice
Concentration (mg.100g-1 of juice)
1000
900
800
700
600
a
b
b
a
a
a,b
a,b
Cont
HTST
UHT
Plas.10
Plas.20
US.5
US.15


b) acerola juice + inulin
Concentration (mg.100g-1 of juice)
1000
900
800
700
600
a
b
b
c
c
c
a,b
Cont
HTST
UHT
Plas.10
Plas.20
US.5
US.15


c) acerola juice + gluco-oligosaccharides
Concentration (mg.100g-1 of juice)
1000
900
800
700
600
a
b
b
a,c
b,c
a,c,d
c,d
Cont
HTST
UHT
Plas.10
Plas.20
US.5
US.15


Cont
Plas.10
Plas.20
US.5
US.15
UHT
HTST
a) aromatic region
b) carbinolic region
c) aliphatic region
formic ac.
ac.
a) scores
PC2 - 18.00 %
PC1 - 57.32 %
UHT
US.15
Plas.20
US.5
Cont
HTST
Plas.10
b) loadings
formic acid
2-furoic acid
acetaldehyde
trigonelline
acetic acid
PC1
ascorbic acid
choline
malic acid
2-furoic acid
PC2
Loadings intensity
Variables (ppm)
α-glucose
non-deuterated water suppresion region
β-glucose
malic ac.
dehydroascorbc ac.
fructose
ascorbic ac.
asparaginine
ascorbic ac.
choline
β-glucose
Chemical Shift (ppm)
malic ac.
GABA + proline
acetic ac.
GABA + proline
alanine
threonine
ethanol
Chemical Shift (ppm)


ascorbic acid
choline
2-furoic acid
α-glucose
β-glucose
fructose
malic acid
alanine
ethanol
Concentration (mg.100g-1 of juice)
Cont
HTST
UHT
Plas.10
Plas.20
US.5
US.15

voláteis

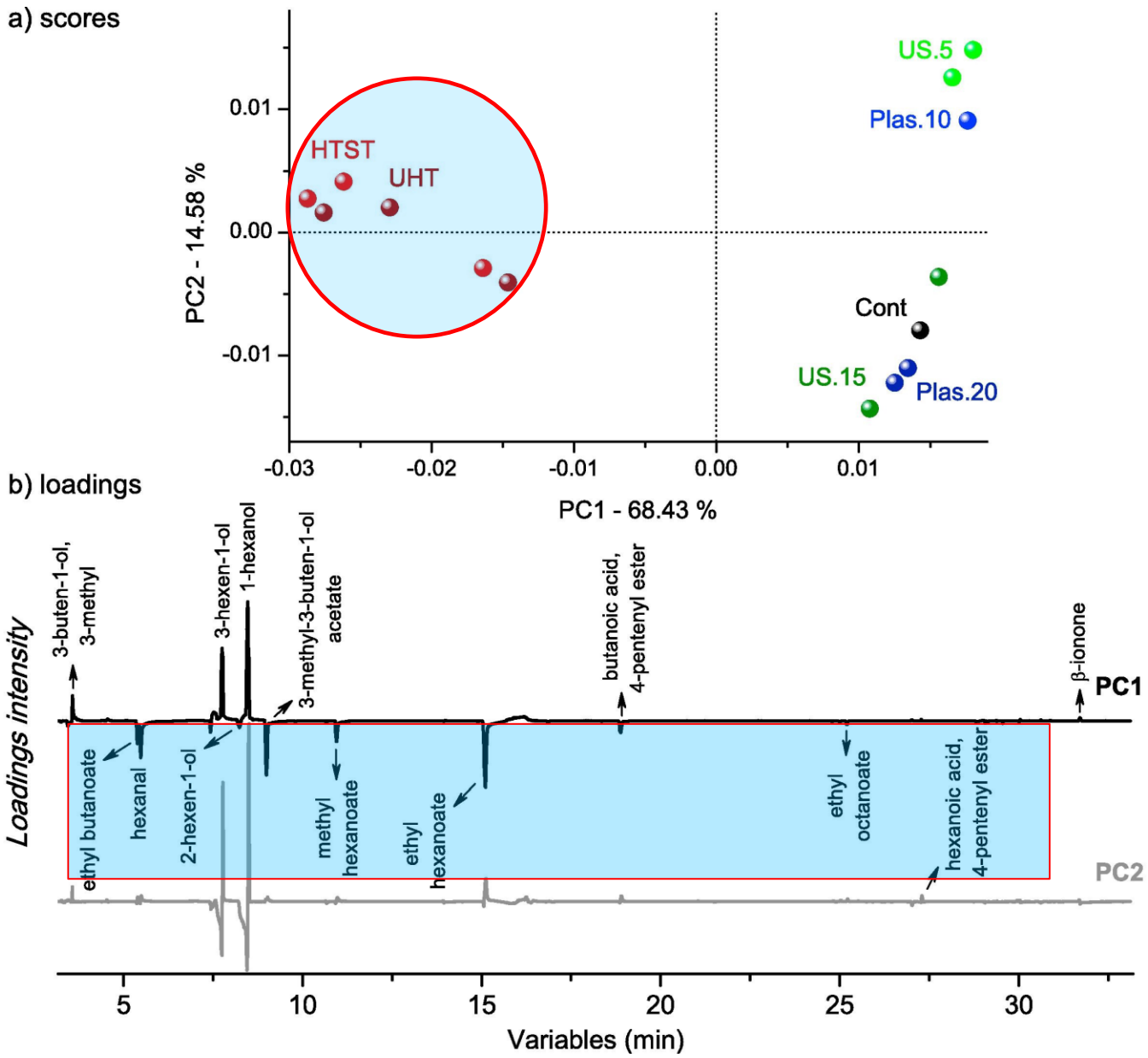
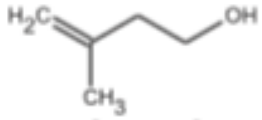
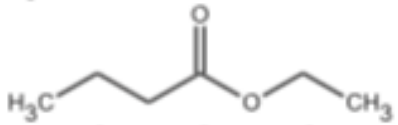
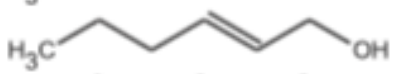
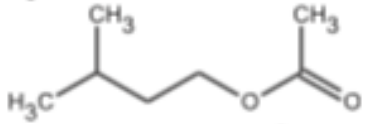
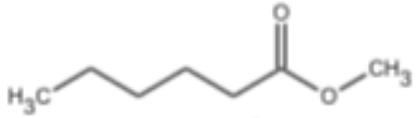
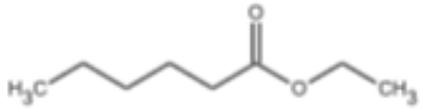
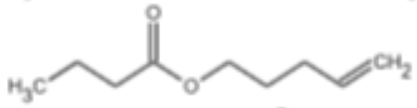
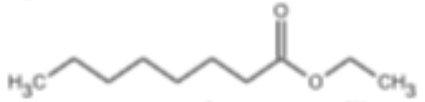
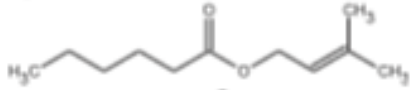
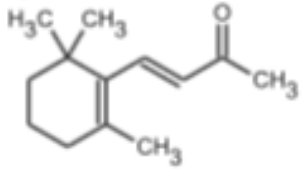

a) scores
US.5
Plas.10
HTST
UHT
Cont
US.15
Plas.20
PC2 - 14.58 %
PC1 - 68.43 %
0.01
0.00
-0.01
-0.03
-0.02
-0.01
0.00
0.01
b) loadings
Loadings intensity
3-buten-1-ol, 3-methyl
3-hexen-1-ol
1-hexanol
3-methyl-3-buten-1-ol
acetate
butanoic acid, 4-pentenyl ester
β-ionone
PC1
ethyl butanoate
hexanal
2-hexen-1-ol
methyl hexanoate
ethyl hexanoate
ethyl octanoate
hexanoic acid, 4-pentenyl ester
PC2
5
10
15
20
25
30
Variables (min)

Table 1

Structures of the relevant volatile flavor components detected in acerola juices according to PCA, with respective retention times (RT), experimental and reference retention index (RI), major *m/z* peak, and percentage (%) of match.

RT (min)	Compound name	Structure	RI[a] exp.	RI[a] refer.	Major *m/z*	Match (%)
3.577	3-methyl 3-buten-1-ol		727	723[b]	41	94
5.384	hexanal		801	801[b]	44	86
5.479	ethyl butanoate		804	802[b]	43	92
7.706	3-hexen-1-ol		856	850[b]	41	97
8.021	2-hexen-1-ol		864	859[b]	57	95
8.431	1-hexanol		873	871[b]	56	94
8.972	3-methyl-3-buten-1-ol, acetate		886	880[c]	43	95
10.957	methyl hexanoate		926	921[b]	74	94
15.086	ethyl hexanoate		1001	997[b]	88	95
18.897	butanoic acid, 4-pentenyl ester		1067	1064[b]	68	84
25.208	ethyl octanoate		1199	1196[b]	94	88
27.310	hexanoic acid, 3-methyl-2-butenyl ester		1268	1267[d]	88	68
31.670	β-ionone		1490	1487[b]	177	92

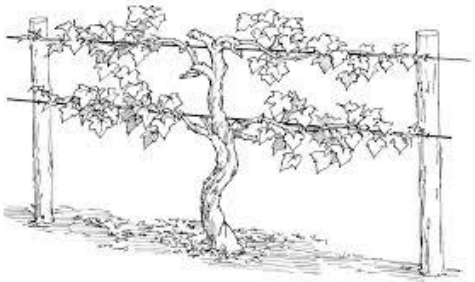
^{1}H NMR and LC-MS-based metabolomic approach for evaluation of the seasonality and viticultural practices in wines from São Francisco River Valley, a Brazilian semi-arid region

Amostra: vinho

Processamento: prébiotico + térmico UHT e HTST + US+ Plasma

Análise: RMN ^{1}H + LC-MS

Quimiometria: PCA e quantificação (*q*NMR)



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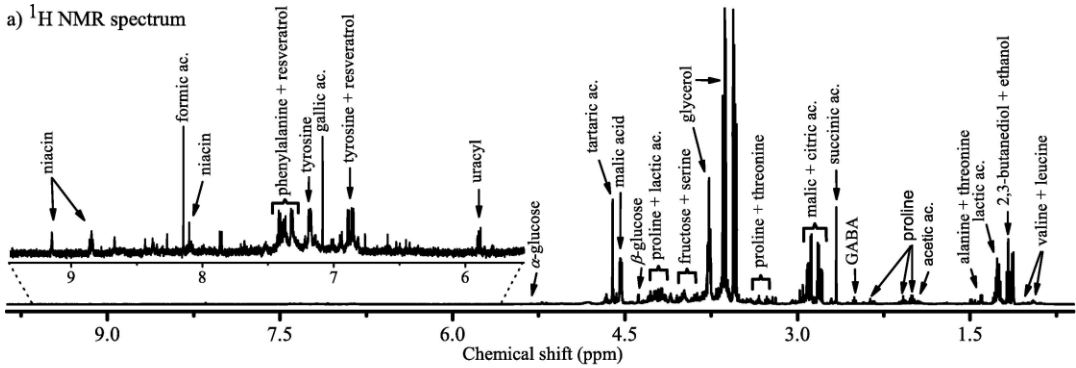
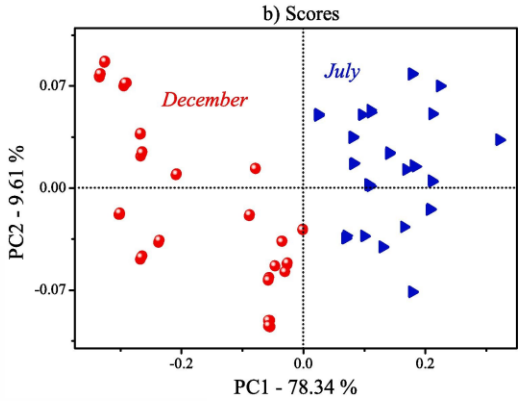
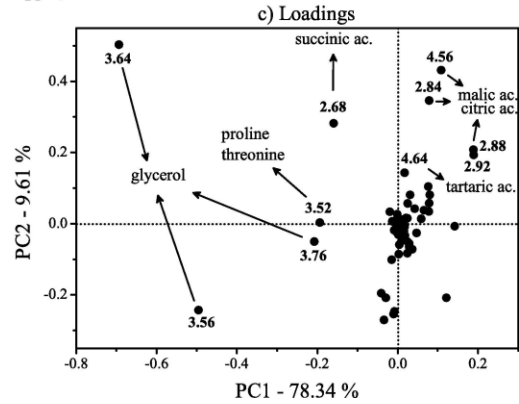
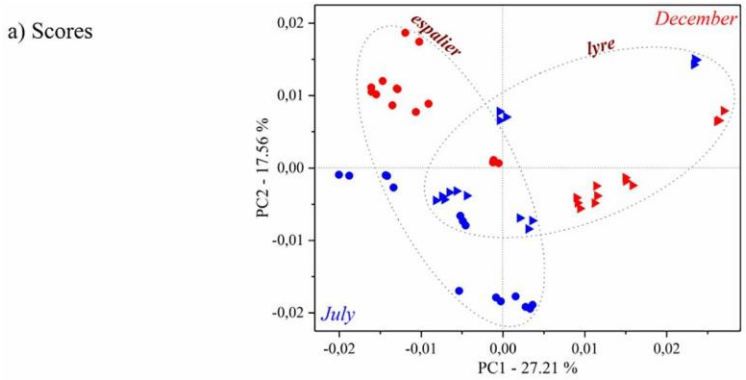
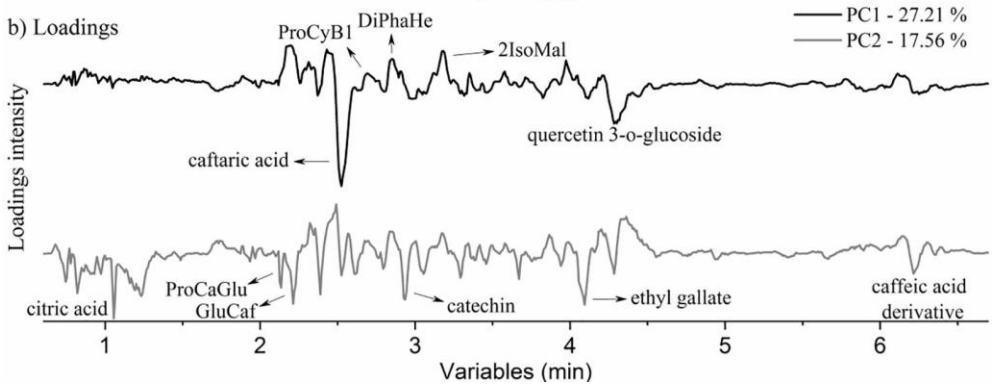
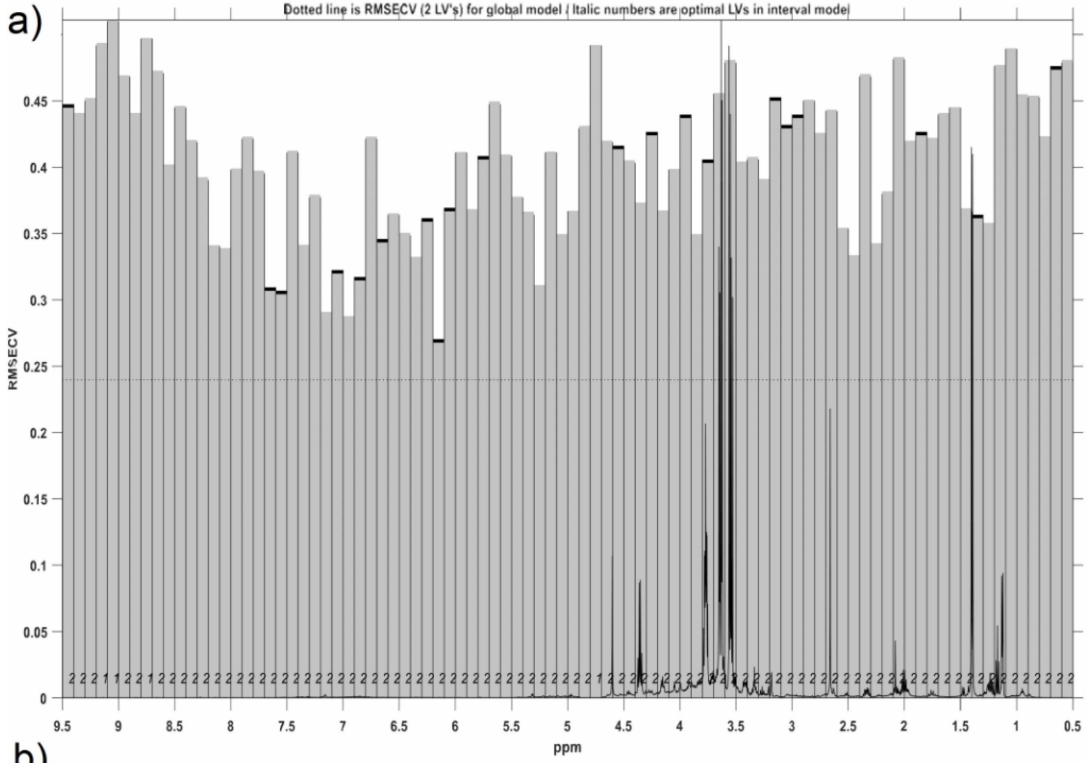

a) ^{1}H NMR spectrum
niacin
formic ac.
niacin
phenylalanine + resveratrol
tyrosine
gallic ac.
tyrosine + resveratrol
uracyl
α-glucose
tartaric ac.
malic acid
β-glucose
proline + lactic ac.
fructose + serine
glycerol
proline + threonine
malic + citric ac.
succinic ac.
GABA
proline
acetic ac.
alanine + threonine
lactic ac.
2,3-butanediol + ethanol
valine + leucine
9.0
7.5
6.0
4.5
3.0
1.5
Chemical shift (ppm)

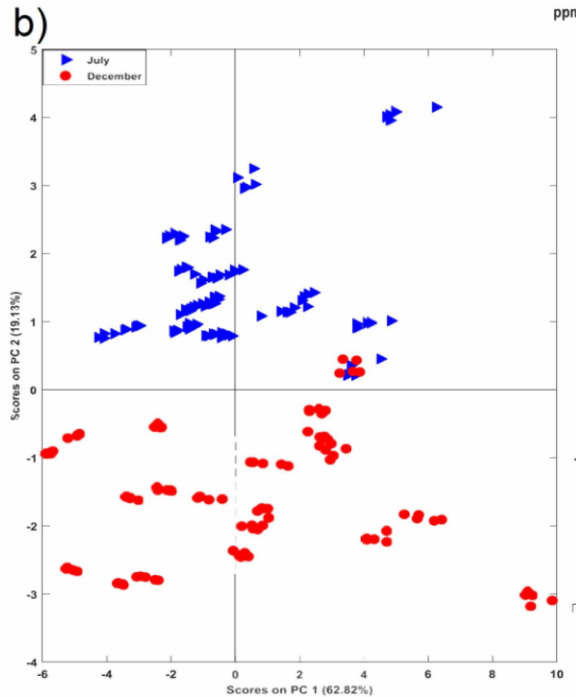

b) Scores
December
July
PC2 - 9.61 %
PC1 - 78.34 %

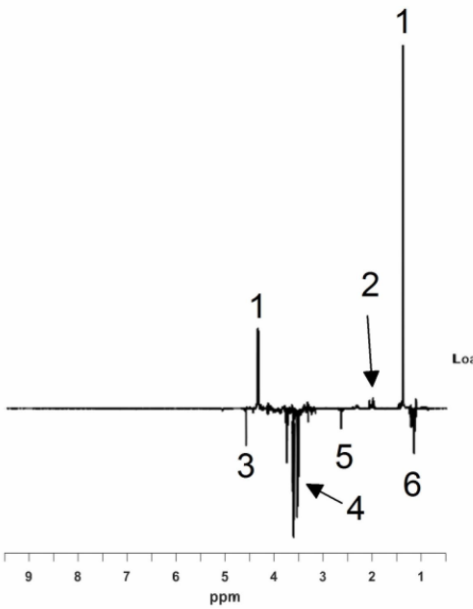

c) Loadings
succinic ac.
malic ac.
citric ac.
proline
threonine
glycerol
tartaric ac.
3.64
4.56
2.84
2.68
2.88
2.92
4.64
3.52
3.76
3.56
PC2 - 9.61 %
PC1 - 78.34 %

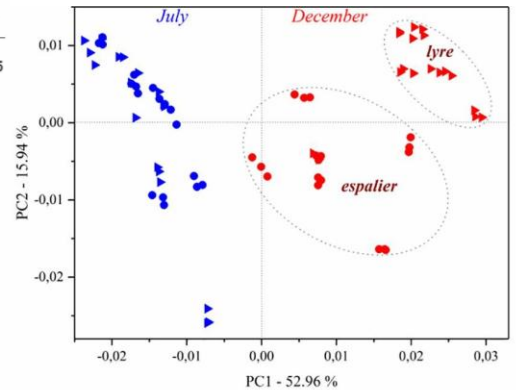

a) Scores
espalier
lyre
December
July
PC2 - 17.56 %
PC1 - 27.21 %

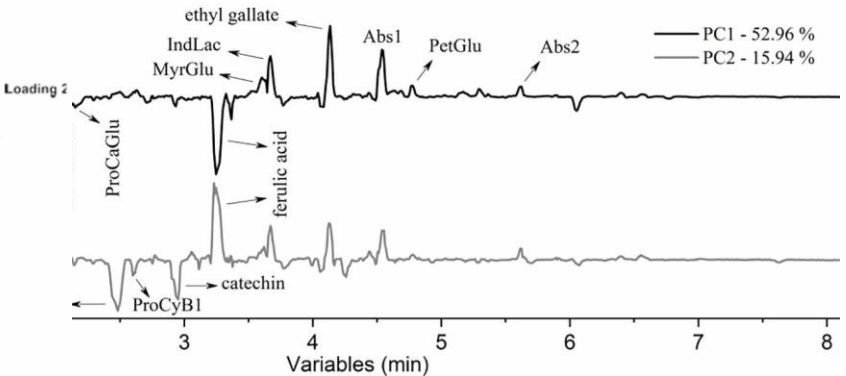

b) Loadings
PC1 - 27.21 %
PC2 - 17.56 %
ProCyB1
DiPhaHe
2IsoMal
quercetin 3-o-glucoside
caftaric acid
Loadings intensity
citric acid
ProCaGlu
GluCaf
catechin
ethyl gallate
caffeic acid derivative
Variables (min)


a)
Dotted line is RMSECV (2 LV's) for global model (Italic numbers are optimal LVs in interval mode)
RMSECV
ppm


b)
July
December
Scores on PC 2 (19.13%)
Scores on PC 1 (62.82%)


1
2
3
4
5
6
ppm


July
December
lyre
espalier
PC2 - 15.94 %
PC1 - 52.96 %


ethyl gallate
IndLac
MyrGlu
Abs1
PetGlu
Abs2
PC1 - 52.96 %
PC2 - 15.94 %
ProCaGlu
ferulic acid
catechin
ProCyB1
Variables (min)

Food & Function

PAPER

Cite this: *Food Funct.*, 2019, **10**, 1671

Cashew apple fiber prevents high fat diet-induced obesity in mice: an NMR metabolomic evaluation†

Diana Valesca Carvalho,[a] Lorena Mara Alexandre Silva,[b] Elenilson Godoy Alves Filho,[b] Flávia Almeida Santos,[a] Renan Pereira de Lima,[a] Ana Flávia Seraine Custódio Viana,[a] Paulo Iury Gomes Nunes,[a] Said Gonçalves da Cruz Fonseca,[a] Tiago Sousa de Melo,[c] Daniel de Araújo Viana,[d] Maria Izabel Gallão[a] and Edy Sousa de Brito *[b]

Contents lists available at ScienceDirect

Bioactive Carbohydrates and Dietary Fibre

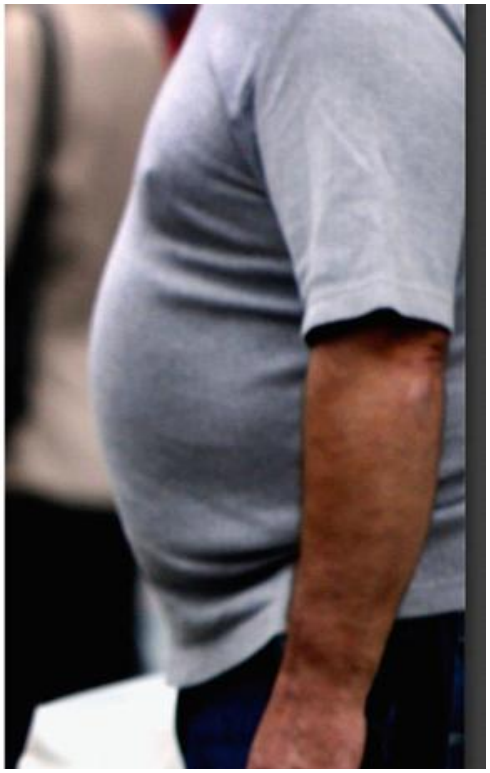
journal homepage: www.elsevier.com/locate/bcdf

Influence of low molecular weight compounds associated to cashew (*Anacardium occidentale* L.) fiber on lipid metabolism, glycemia and insulinemia of normal mice

Diana Valesca Carvalho[a], Flávia Almeida Santos[b], Renan Pereira de Lima[c], Ana Flávia Seraine Custódio Viana[b], Said Gonçalves Cruz Fonseca[d], Paulo Iury Gomes Nunes[b], Tiago Sousa de Melo[e], Maria Izabel Gallão[f], Edy Sousa de Brito[g,*]

Brasil

53,8%



EXCESSO DE PESO

* IMC ≥ 25kg/m²

18,9%

* IMC ≥ 30kg/m²

(Vigitel, 2017)

Bagaço de frutas – fonte alternativa de fibra alimentar

- Subproduto produção de sucos
- Geralmente descartado no meio ambiente
- Fibra e compostos fenólicos ligados à matriz da fibra
- Caju: Importância sócio-econômica/riqueza em nutrientes

Fonte: Siqueira, de Brito. Foto: Cláudio de Norões Rocha

JURGOŃSKI et al., 2016; OLIVEIRA; IPIRANGA, 2009

Preparação da ração dos camundongos

Metabolismo normal

FcI (10%)

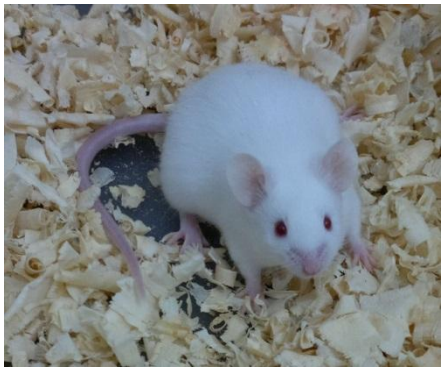
FcSM (10%)

Dieta Normal (ND) – Ração comercial

Dieta Normal (ND) – Ração comercial

ND-FcI

ND-FcSM



(p:19-25g)

(CEPA/UFC 21/15)

Dietas

- Dieta Normal (ND) n=10
- ND-FcI (10%) n=10
- ND-FcSM (10%) n=10
- Dieta Normal (ND) n=10
- HFD n=10
- HFD-FcSM (10%) n=10

Metabolismo normal

Obesidade

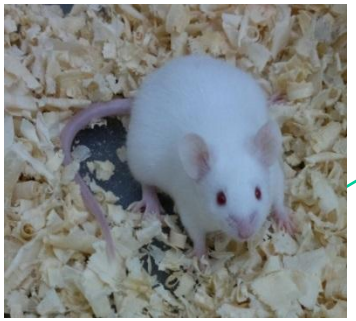
Alimentados durante 15 semanas; ração e água *ad libidum*

Peso animal

Consumo de ração e água 2x/semana

Coleta de amostras

14ª semana



Fezes

Obesidade

15ª semana

Jejum de 6h

Sangue

Eutanásia

Metabolismo normal e Obesidade

Fígado

Tecido adiposo branco abdominal

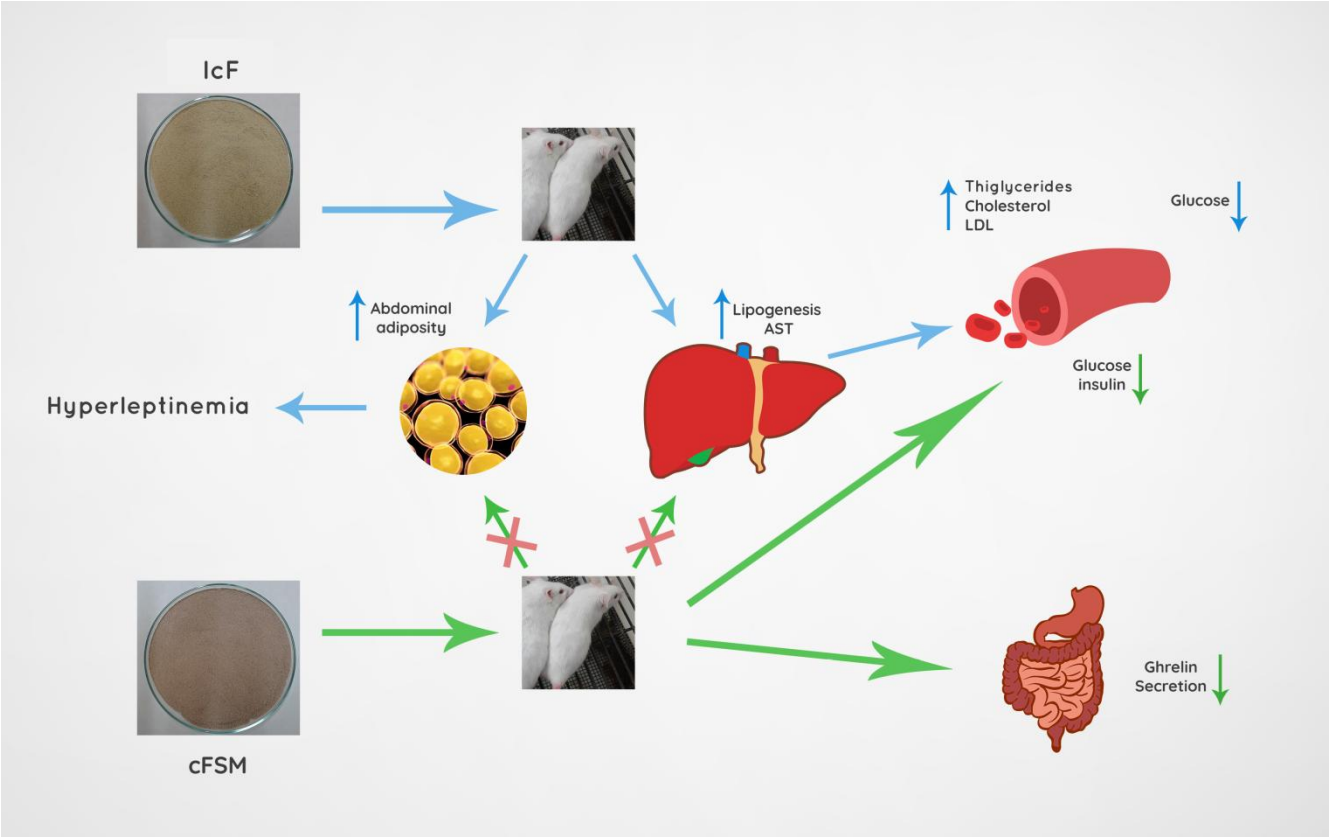
Soro

- **Glicose**
- **Colesterol total, HDL, LDL, triglicerídeos**
- **Enzimas hepáticas (AST, ALT)**
- **Enzimas digestivas (amilase, lipase)**
- **Hormônios (Insulina, leptina, grelina)**
- **Citocinas (IL-6 e TNF-α, adiponectina)**

- **Peso**
- **MDA**
- **NP-SH**
- **Colesterol**
- **Histologia**

Peso

Metabolismo normal

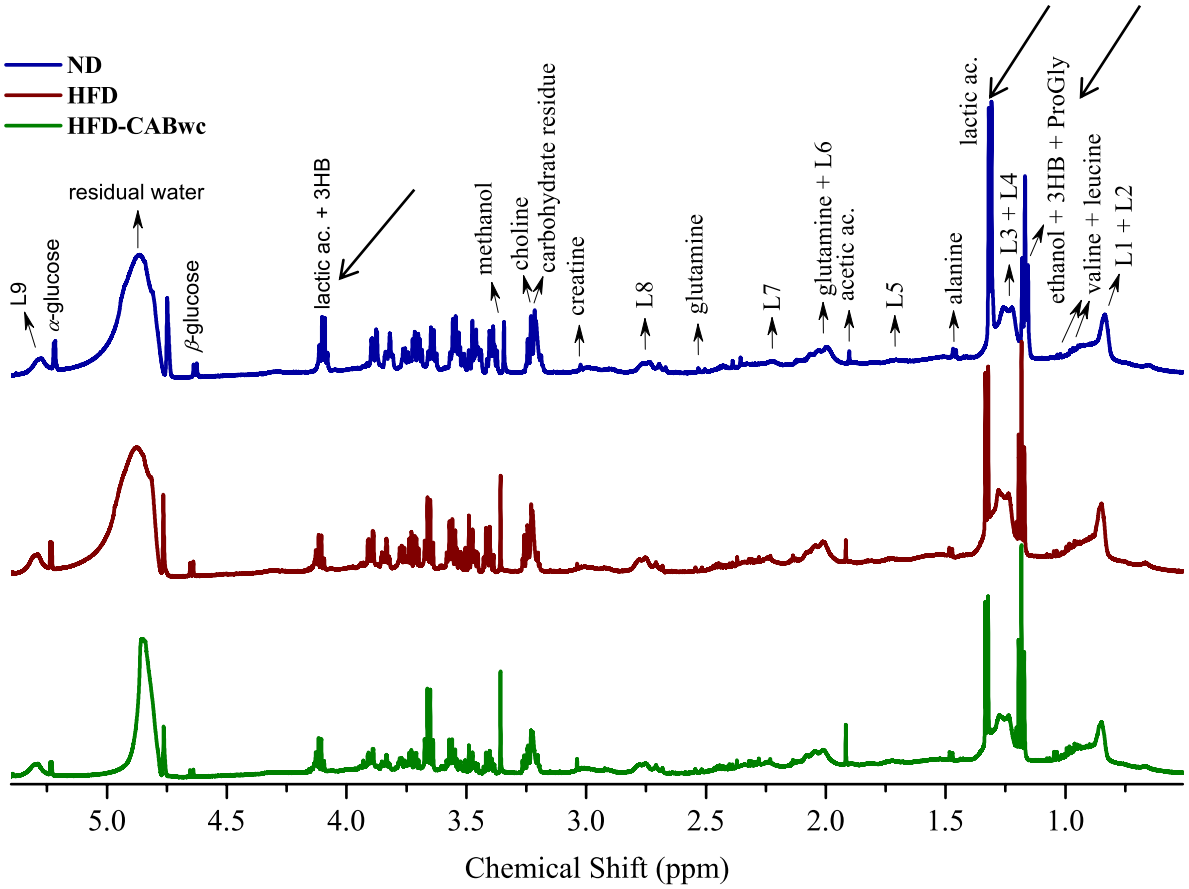


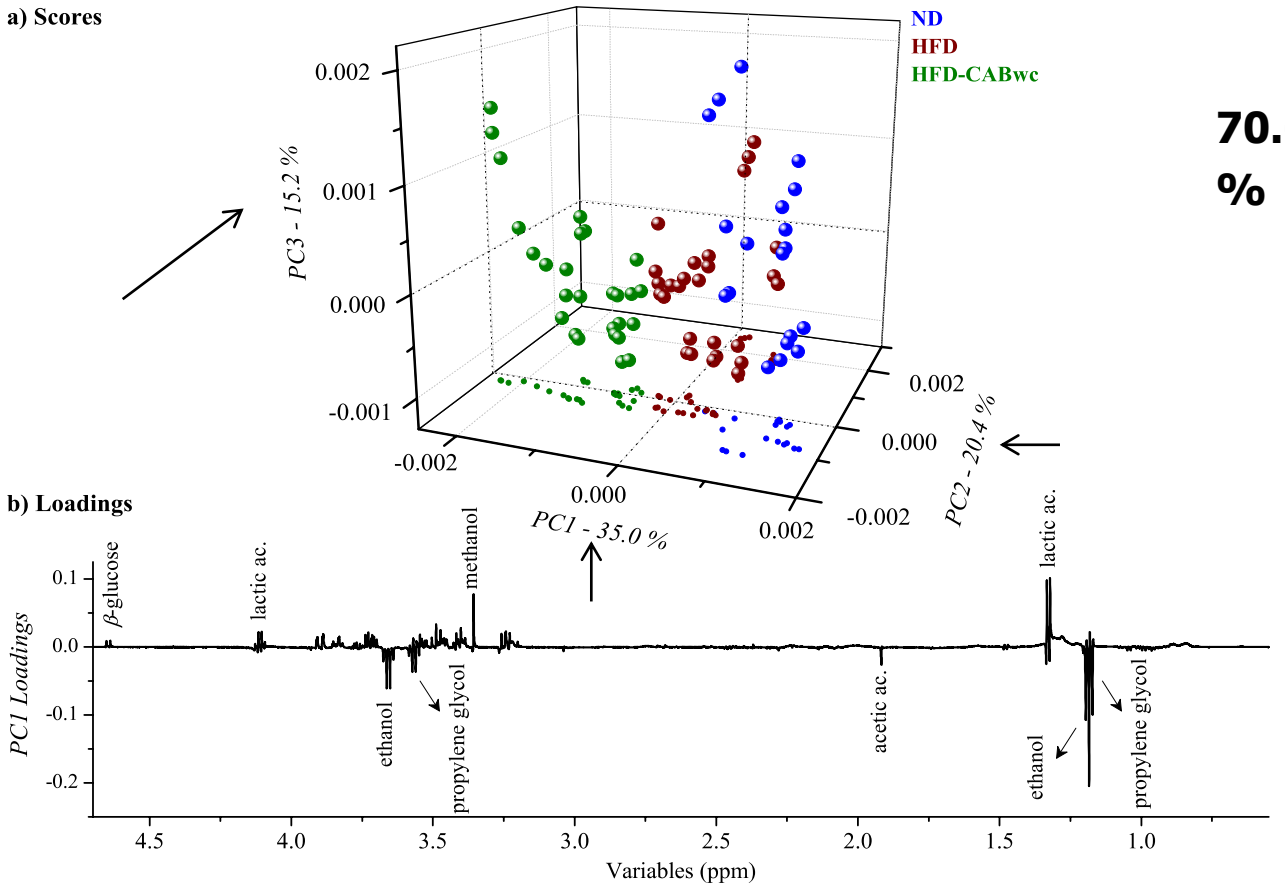
Ingestão alimentar e energética: sem diferença

Ingestão de água: elevada FcSM

Peso corporal: sem diferença

Perfil do soro dos camundongos através de RMN acoplado a quimiometria

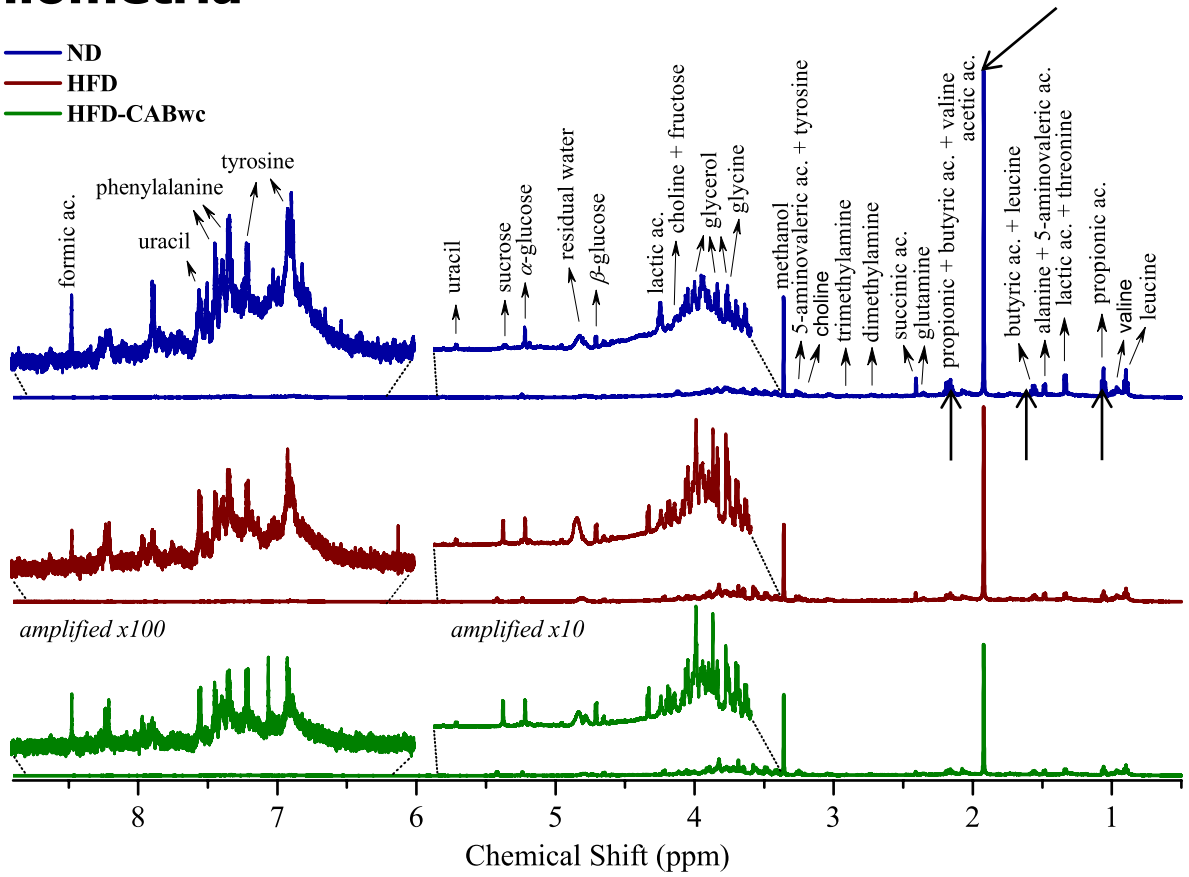




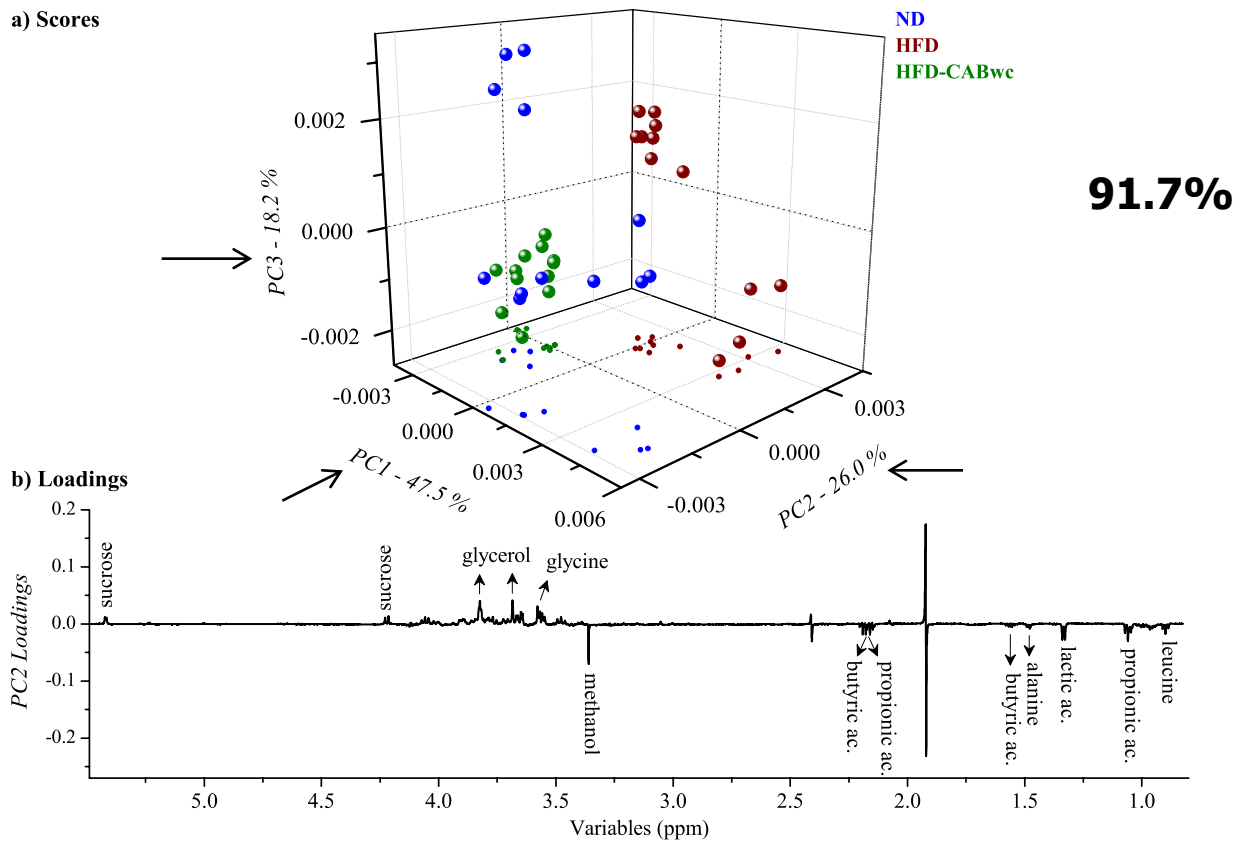
70.6 %

» **etanol pode ser produzido por microrganismos intestinais sob diferentes condições nutricionais (Elshaghabee et. al, 2016).**

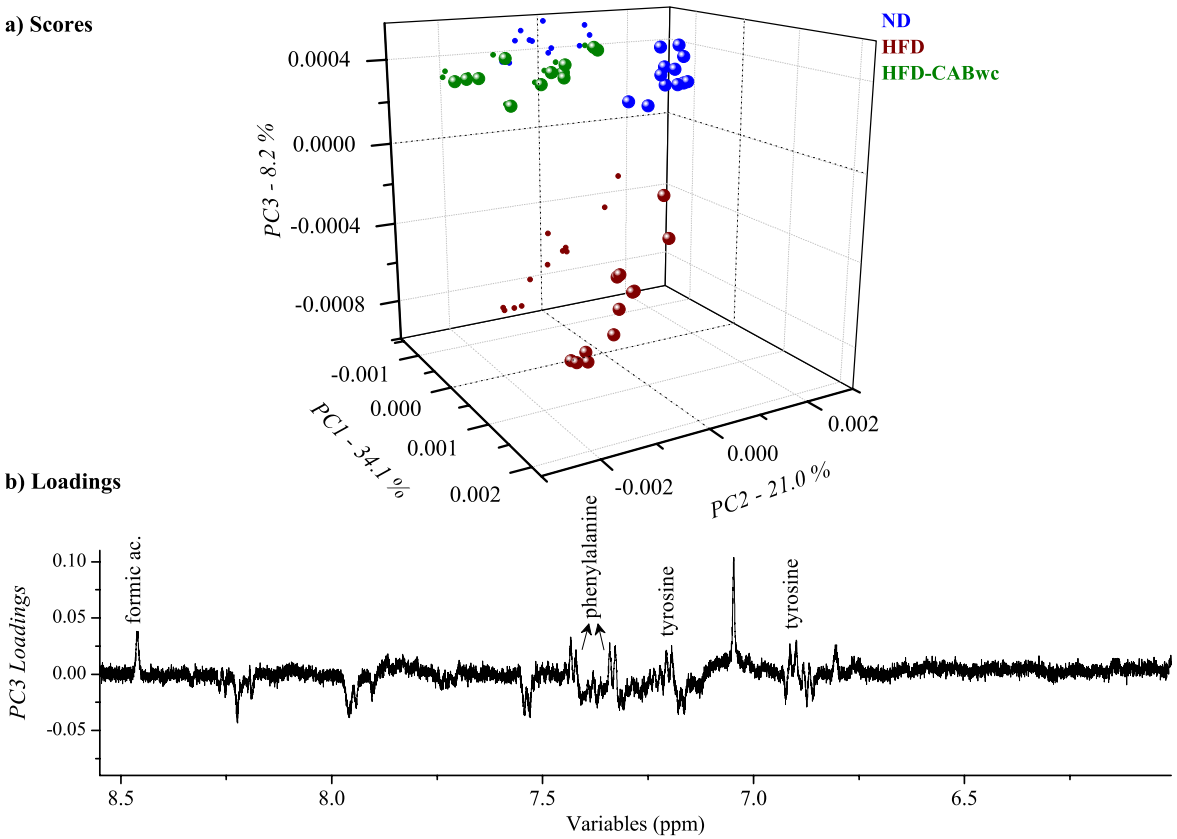
Perfil das fezes dos camundongos através de RMN acoplado a quimiometria



A comparação entre os espectros mostrou que as fezes são principalmente compostos de ácidos orgânicos de cadeia curta (acético, propiônico, butírico, láctico, succínico e fórmico), aminoácidos (leucina, alanina, uracila, tirosina e fenilalanina), açúcares (sacarose, glicose e frutose) e metanol.


a) Scores
ND
HFD
HFD-CABwc
0.002
0.000
-0.002
PC3 - 18.2 %
-0.003
0.000
0.003
0.006
PC1 - 47.5 %
0.003
0.000
-0.003
PC2 - 26.0 %
b) Loadings
PC2 Loadings
0.2
0.1
0.0
-0.1
-0.2
sucrose
sucrose
glycerol
glycine
methanol
butyric ac.
propionic ac.
alanine
butyric ac.
lactic ac.
propionic ac.
leucine
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
Variables (ppm)

91.7%



A presença desses aminoácidos está possivelmente relacionada às atividades metabólicas das bactérias intestinais do gênero *Adlercreutzia, Anaerostipes, Coprococcus* da família Lachnospiraceae (Lin et. al, 2016).

Agradecimentos

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Departamento de Engenharia Química

Embrapa

MINISTÉRIO DA
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