Munich
GERMANY 4-6 May 2022
IAFP'S EUROPEAN SYMPOSIUM ON FOOD SAFETY

Symposium S3: Process Analytical Technology at the Service of Food Protection; The "DiTECT" Approach

Wednesday May 4, 2022

Holiday Inn Munich, Germany

Process Analytical Technology in the Food Industry: Principles, Methods and Applications

Alexandra Lianou[1] & George-John Nychas[2]

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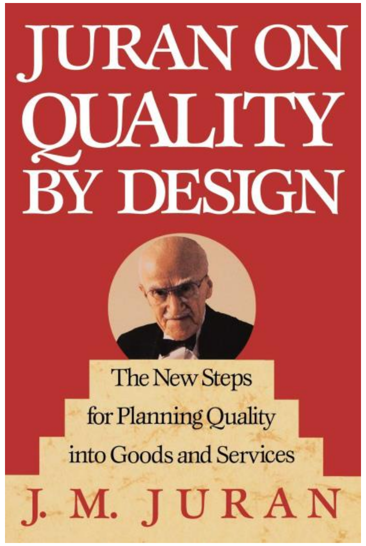
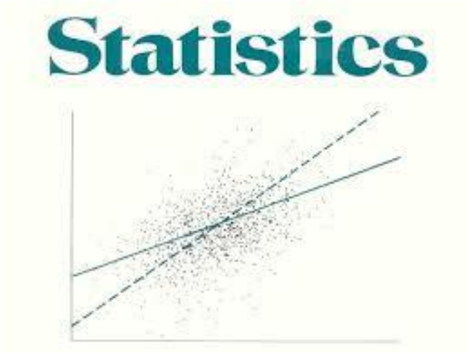
Munich GERMANY 4-6 May 2022 IAFP'S EUROPEAN SYMPOSIUM ON FOOD SAFETY

Process Analytical Technology (PAT)

- Basis for the fulfillment of "Quality by Design" (QbD)
- Processing: focus on product quality
- From post-manufacturing controls to integration of quality into processes
- Main objective: compliance with quality standards
- Joseph M. Juran: "…***quality can be designed***…"
- Application of QbD in manufacture:
  1. Identification of critical quality parameters
  2. Process design
  3. Control interventions
  4. Validation and record-keeping
  5. Continuous monitoring



Basic principles of PAT



PAT

Basic principles of PAT

U.S. FOOD & DRUG ADMINISTRATION

“Systems for analysis and control of manufacturing processes based on ***real-time process monitoring of critical quality parameters and performance attributes of raw materials and in-process products****, to assure acceptable end-product quality at the completion of the process”*

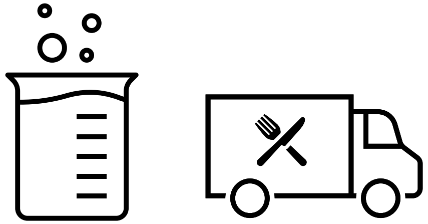
Ways of monitoring

Connection to final quality

Systematic framework

Basic principles of PAT

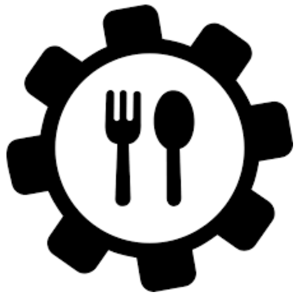
- Important differences among industries
- Key points in industrial applications:
  - ✓ Interpretation of PAT among distinct production units
  - ✓ Analytical equipment and data processing
  - ✓ Deviation from initial objective: real-time quality assurance via a holistic and systematic approach



- Transition from Process Analytical Chemistry (**PAC**) to **PAT**: understanding of the chemical, physical and biological parameters

Application of PAT in the food industry

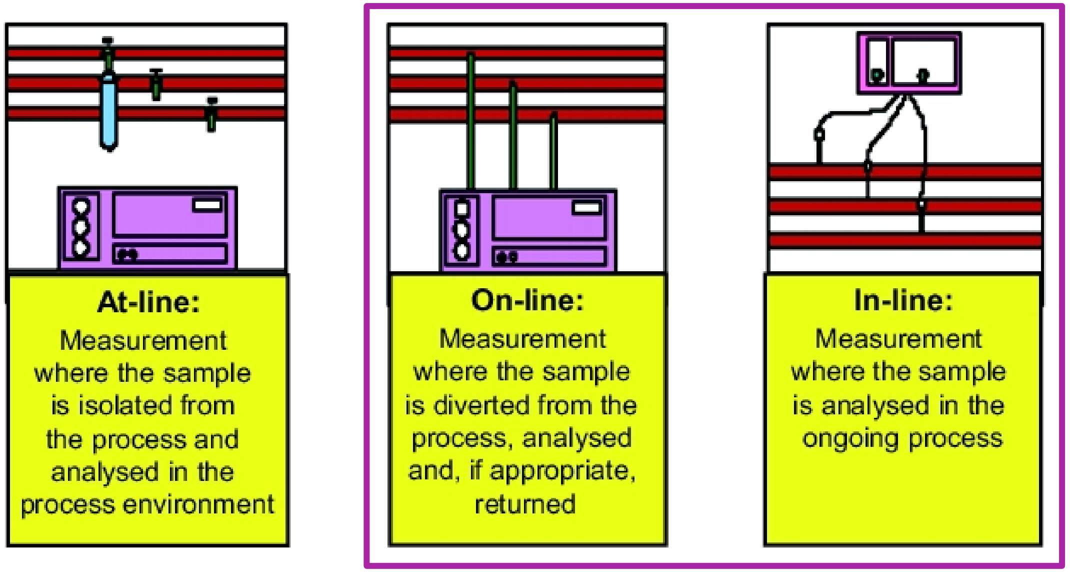
- F. van den Berg et al. (2013)*:
  "PAT represents a silent revolution in industrial quality control in food processing"
- Strict regulatory requirements in safety, quality and traceability issues
- Important limitations and challenges:
  - ✓ Food products: complex and multifactorial systems
  - ✓ Food systems: extensive "natural" variability (biological)
- Incompetency of "traditional" technologies (based on off-line laboratory measurements)



*https://doi.org/10.1016/j.tifs.2012.04.007

Application of PAT in the food industry

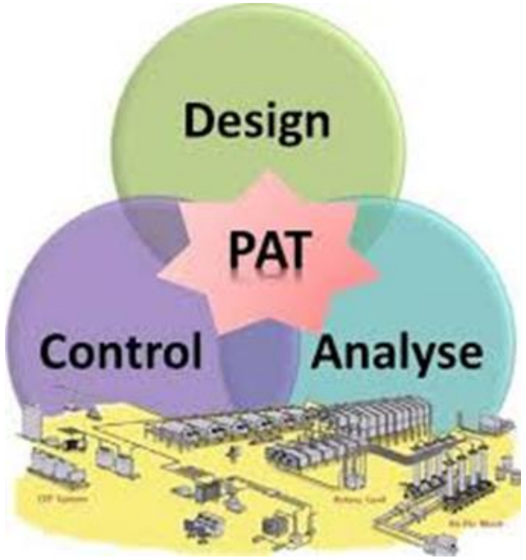
- Goal: end-product quality assurance in an efficient, traceable and environmentally responsible manner
- Development of novel sampling methods/analytical tools allowing for timely measurements of critical parameters



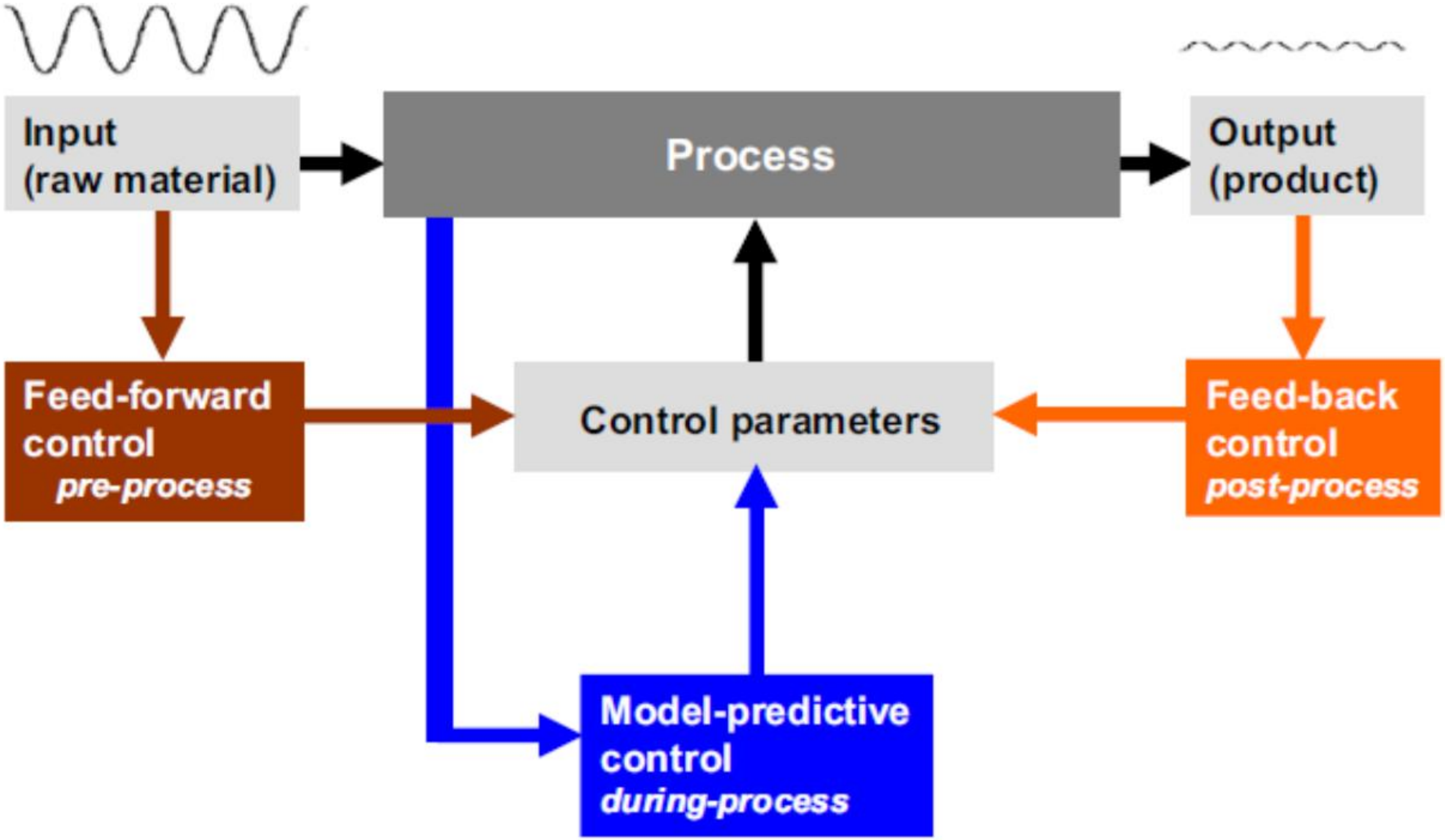
https://gmpua.com/

Benefits of PAT in the food industry

- Improved utilization and control of raw materials
- Reduction of the variability in end-product quality
- Reduction/elimination of food loss
- Reduction of the duration of process cycles
- Time efficiency
- Cost reduction
- Ongoing knowledge update
- Process and product innovation
- Assisting HACCP strategies

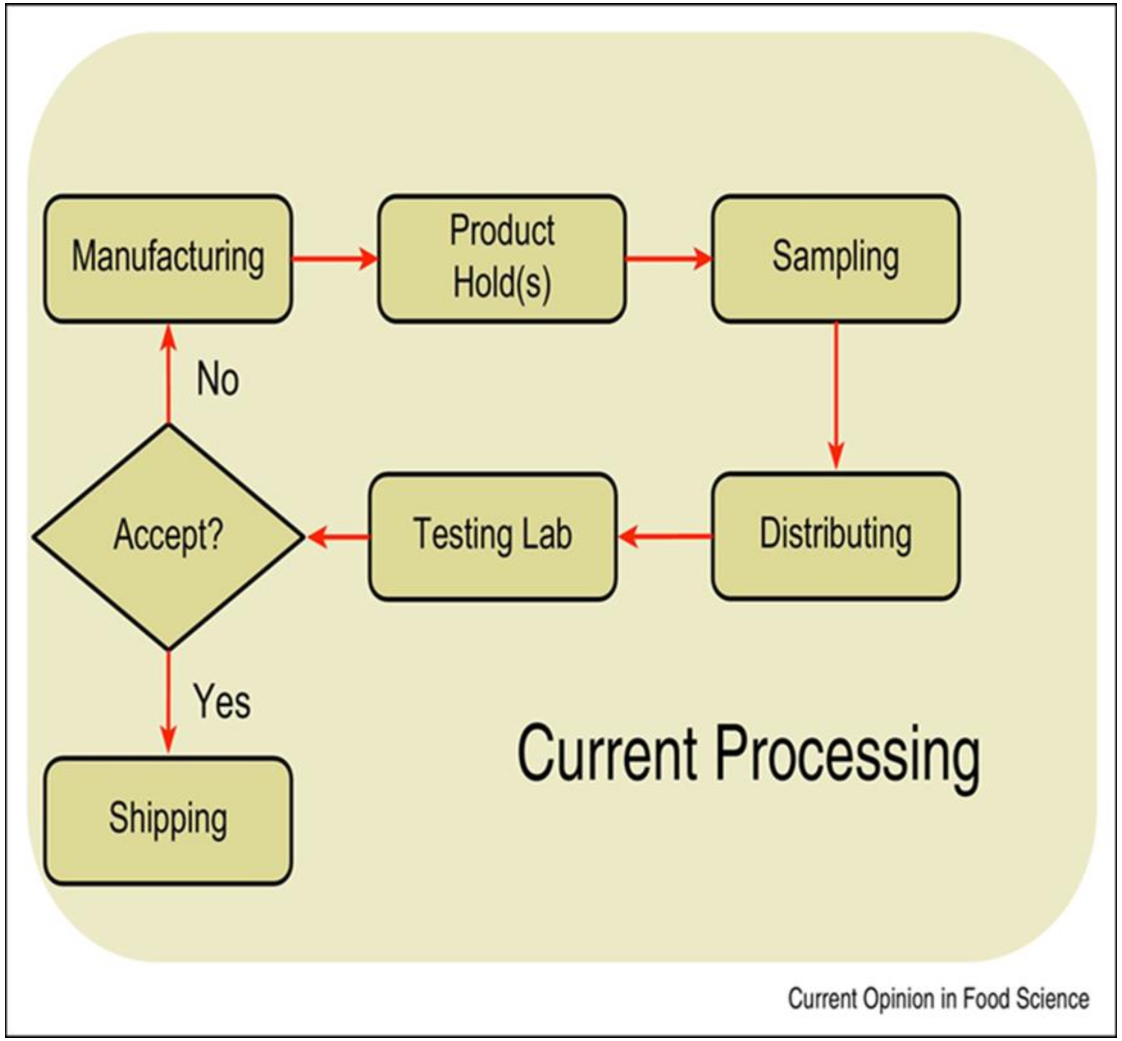


Application of PAT in the food industry



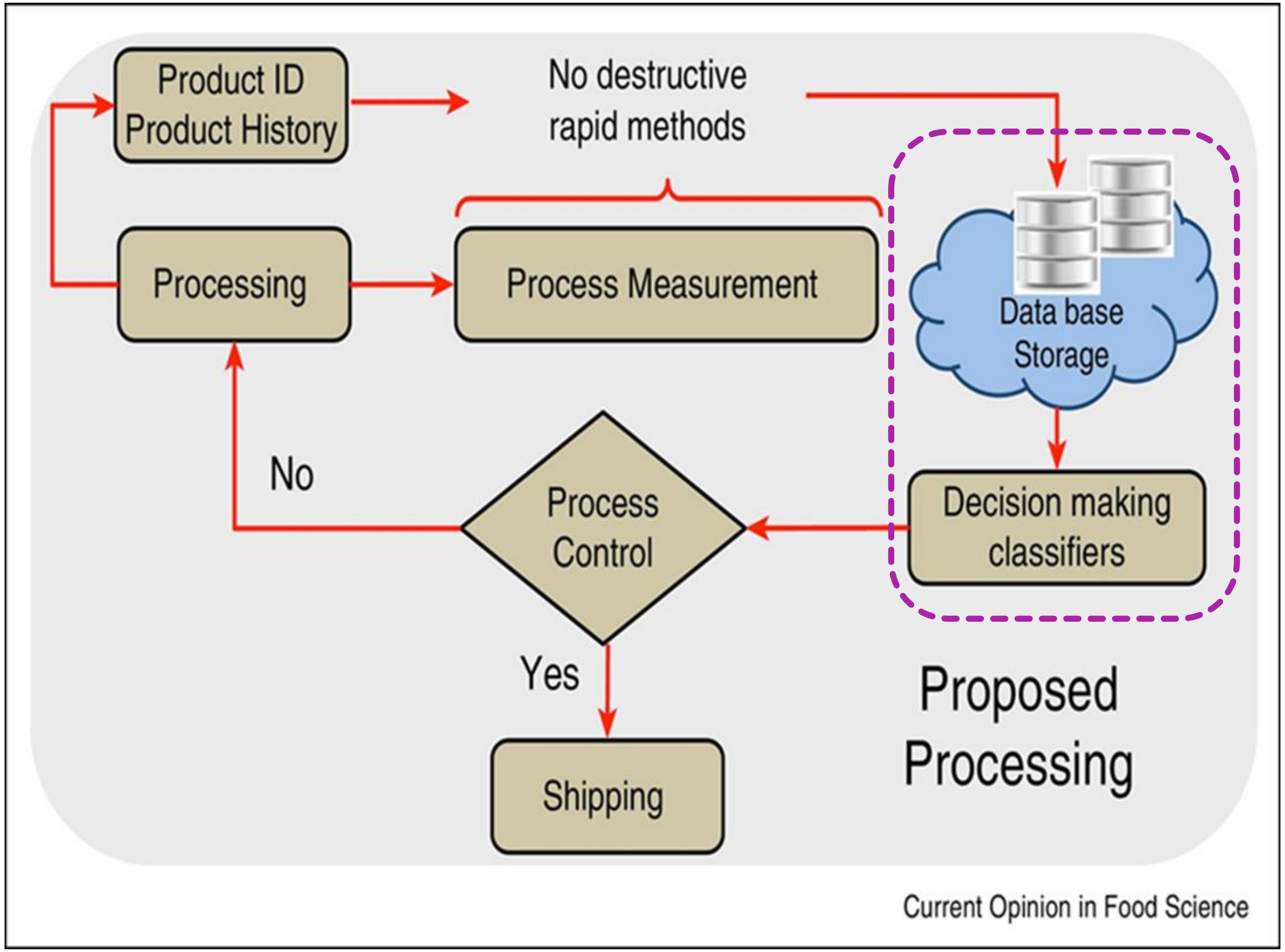
Van den Berg et al., 2013; https://doi.org/10.1016/j.tifs.2012.04.007

“Traditional” food industry processes…



Nychas et al., 2016; http://dx.doi.org/10.1016/j.cofs.2016.06.005

Proposed food industry processes…



Nychas et al., 2016; http://dx.doi.org/10.1016/j.cofs.2016.06.005

Application of PAT in the food industry

- **Basic components of PAT application:**
  - ✓ Understanding of critical quality parameters of products
  - ✓ Understanding of process dynamics in relation to sampling
  - ✓ Analytical instruments for in-line/on-line measurements
  - ✓ Massive flows of process (multivariate) data
- **Tools for PAT application:**
  - (I) Process monitoring using spectroscopy-based methods
  - (II) Statistical analysis of multivariate data
  - (III) Information/data management and ongoing improvement

PAT application tools

(I) Non-invasive analytical technologies based on spectroscopy and/or image analysis

⚙ PAT application tools

(I) Non-invasive analytical technologies based on spectroscopy and/or image analysis

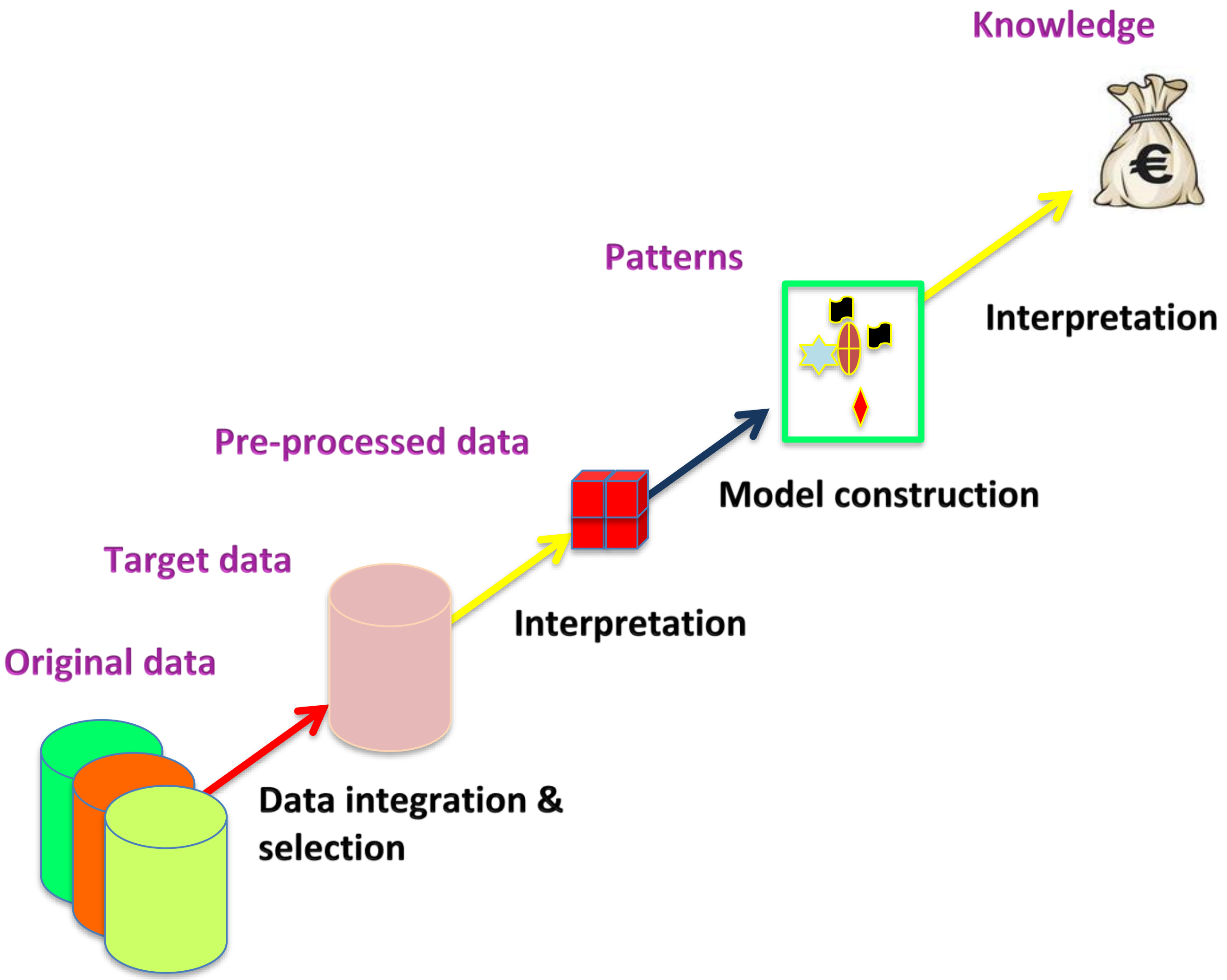
http://www.myfoodsniffer.com/

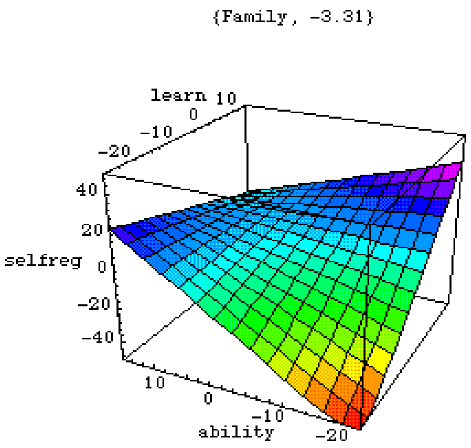
https://tellspec.com/

https://phasmafood.eu/

PAT application tools

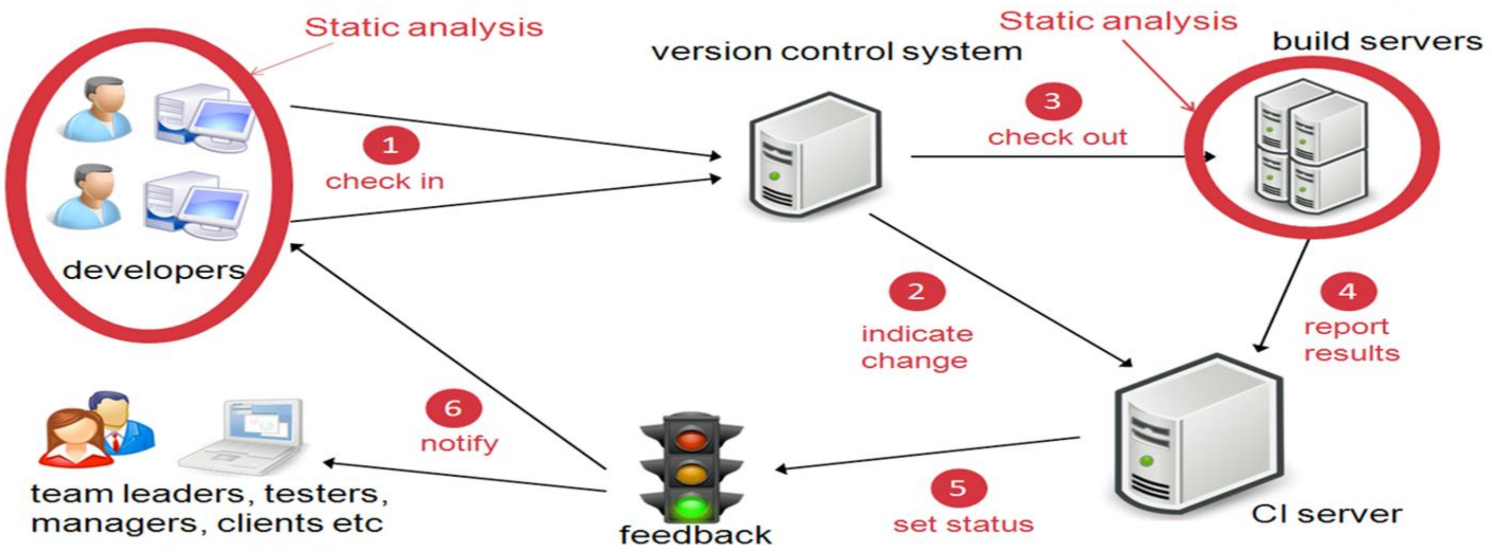
(II) Multivariate statistics and data mining





⚙ PAT application tools

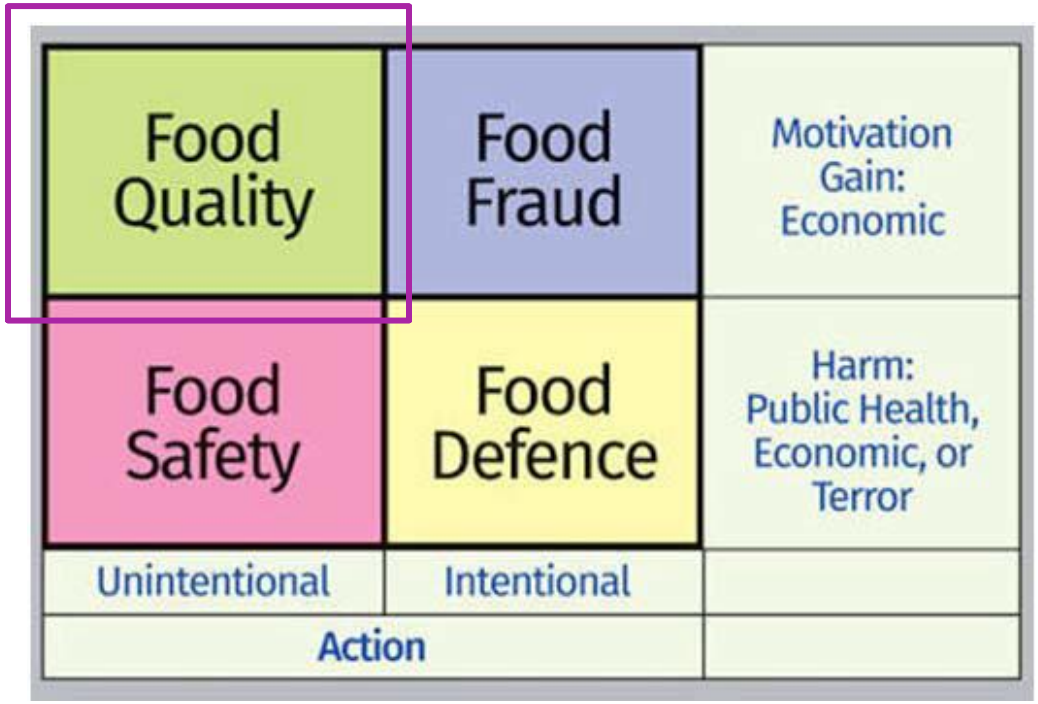
(III) Information/data management and ongoing improvement



PAT concept applications in foods

- ✓ *Microbiological quality*
- ✓ *Milk coagulation*
- ✓ *Cheese ripening*
- ✓ *Emulsification*
- ✓ *Physicochemical parameters*
- ✓ *Food additives*
- ✓ *Antioxidants*

Food risk matrix*



*Spink and Moyer, 2011; https://doi.org/10.1111/j.1750-3841.2011.02417.x

Food Research International 44 (2

Contents lists available at SciV

Food Research Int

journal homepage: www.elsevie

Contribution of Fourier transform infrared (FT
quantitative determination of minced pork me

O. Papadopoulou [a,b], E.Z. Panagou [a,*], C.C. Tassou [b], G.-J.E. N

[a] Laboratory of Microbiology and Biotechnology of Foods, Department of Food Science and Technolog
[b] Institute of Technology of Agricultural Products, National Agricultural Research Foundation, Sofokli Venizelou 1, Lycovrissi, GR-141 23, Greece

Food Control 29 (2013) 461–470

Contents lists available at SciVerse ScienceDi

Food Control

journal homepage: www.elsevier.com/locate

A comparison of Raman and FT-IR spectroscopy for tl
spoilage

Anthoula A. Argyri [a,*], Roger M. Jarvis [b], David Wedge [b], Yun Xu [b], Efs
Royston Goodacre [b], George-John E. Nychas [a]

Food Bioprocess Technol (2013) 6:2268–2279
DOI 10.1007/s11947-012-0886-6

ORIGINAL PAPER

Using Multispectral Imaging for Spoilage Detection of Pork Meat

Bjørn Skovlund Dissing · Olga S. Papadopoulou ·
Chrysoula Tassou · Bjarne Kjaer Ersbøll ·
Jens Michael Carstensen · Efstathios Z. Panagou ·
George-John Nychas

IEEE Access

Received May 19, 2020, accepted June 4, 2020, date of publication June 8, 2020, date o
Digital Object Identifier 10.1109/ACCESS.2020.3000690

Estimation of the Microbiologica
of Meat Using Rapid and Non-In
Spectroscopic Sensors

LEMONIA-CHRISTINA FENGOU[1], IOSIF MPORAS[2], EVGEN
ALEXANDRA LIANOU[1], AND GEORGE-JOHN NYCHAS[1]

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Contents

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journal homep

Evaluation of Fourier transform infra
imaging as means of estimating the m
bream

Lemonia-Christina Fengou, Alexandra Lianou*,
Efstathios Z. Panagou, George-John E. Nychas

Computers and Electronics in Agriculture 155 (2018) 212–219

Contents lists available at ScienceDirect

Computers and Electronics in Agriculture

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

Original papers

A unified spectra analysis workflow for the assessment of microbial contamination of ready-to-eat green salads: Comparative study and application of non-invasive sensors

Panagiotis Tsakanikas*, Lemonia-Christina Fengou, Evanthia Manthou, Alexandra Lianou,
Efstathi

Food Anal. Methods (2018) 11:840–847
https://doi.org/10.1007/s12161-017-1063-3

Rapid Assessment of the Microbiological Quality of Pasteurized Vanilla Cream by Means of Fourier Transform Infrared Spectroscopy in Tandem with Support Vector Machine Analysis

Alexandra Lianou[1] · Christos Malavazos[2] · Ioannis Triantafyllou[2] ·
George-John E. Nychas[1] · Efstathios Z. Panagou[1]

sensors

MDPI

Article

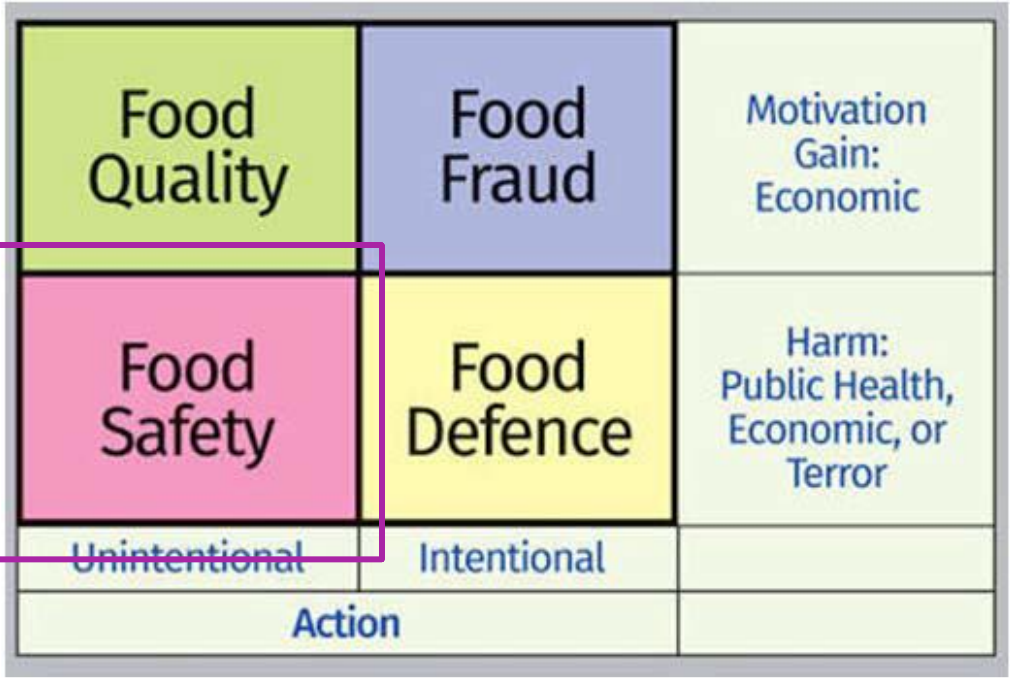
Online Feature Selection for Robust Classification of the Microbiological Quality of Traditional Vanilla Cream by Means of Multispectral Imaging

Alexandra Lianou [1], Arianna Mencattini [2], Alexandro Catini [2], Corrado Di Natale [2],
George-John E. Nychas [1], Eugenio Martinelli [2,*] and Efstathios Z. Panagou [1,*]

PAT concept applications in foods

Food risk matrix

- ✓ *Biological hazards*
- ✓ *Chemical hazards*
- ✓ *Environmental contaminants*



Spink and Moyer, 2011; https://doi.org/10.1111/j.1750-3841.2011.02417.x

sensors MDPI

Review

Recent Advances and Applications of Rapid Microbial Assessment from a Food Safety Perspective

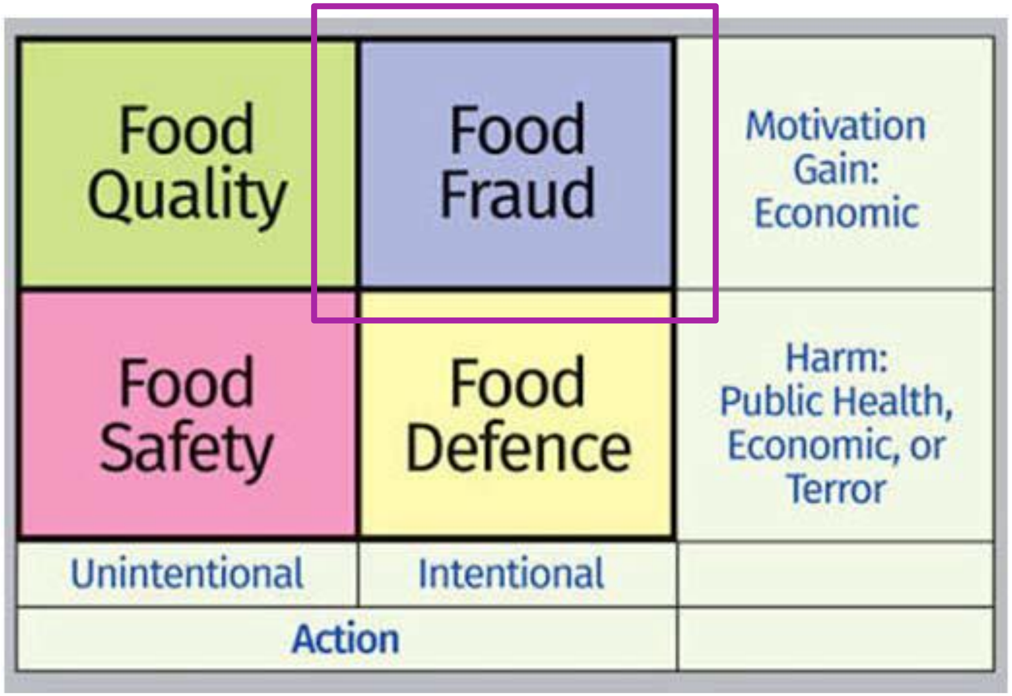
George Pampoukis [1,2], Anastasia E. Lytou [1], Anthoula A. Argyri [3], Efstathios Z. Panagou [1] and George-John E. Nychas [1,*]

Technique	Microorganisms	Purpose	Data Analysis
Fluorescence spectroscopy	*E. coli* O157:H7, *S.* Typhimurium, *L. monocytogenes*	On-site detection in lettuce samples	Savitzky–Golay filter, WA Multiscale Peak Detection, Linear regression
THz-TDS	*S. aureus*, *E. coli*, *P. aeruginosa*, *A. baumanii.*	Detection and alive/dead cells discrimination in culture media	Fourier transformation, standard algorithm
LIBS	*P. aeroginosa*, *E. coli*, *S.* Typhimurium	Detection in culture media	Neural network
3D SERS and LIBS	*S. aureus*, *S.* Typhimurium, *E. coli*	Direct quantification in water	PCA, HCA, Voigt profile fitting
ETLIBS	*S.* Typhimurium	Quantification in bacterial suspensions and detection in spiked food samples	Voigt profile fitting, Log-log linear regression
LTRS	14 microbial species	Discrimination in single cells	Convolutional neural network (ConVet), Occlusion-Based Raman Spectra Feature Extraction ORSFE) tool
SR-FTIR microspectroscopy	10 foodborne bacteria	Discrimination in bacterial suspensions	PCA
HSI	*E. coli* O157:H7 and *Staph. aureus*	Quantification in pork samples	Voigt profile fitting, 2nd derivatives, SNV VCPA, IRIV, GA

https://doi.org/10.3390/s22072800

PAT concept applications in foods

Food risk matrix



- ✓ *EMA (detection of adulterants)*
- ✓ *Misbranding (authentication)*

Spink and Moyer, 2011; https://doi.org/10.1111/j.1750-3841.2011.02417.x

foods

MDPI

Article

Detection of Meat Adulteration Using Spectroscopy-Based Sensors

Lemonia-Christina Fengou [1,*], Alexandra Lianou [2,*], Panagiotis Tsakanikas [1], Fady Mohareb [3] and George-John E. Nychas [1]

Food Control 125 (2021) 108002

Contents lists available at ScienceDirect

Food Control

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

ELSEVIER

Check for updates

Rapid detection of minced pork and chicken adulteration in fresh, stored and cooked ground meat

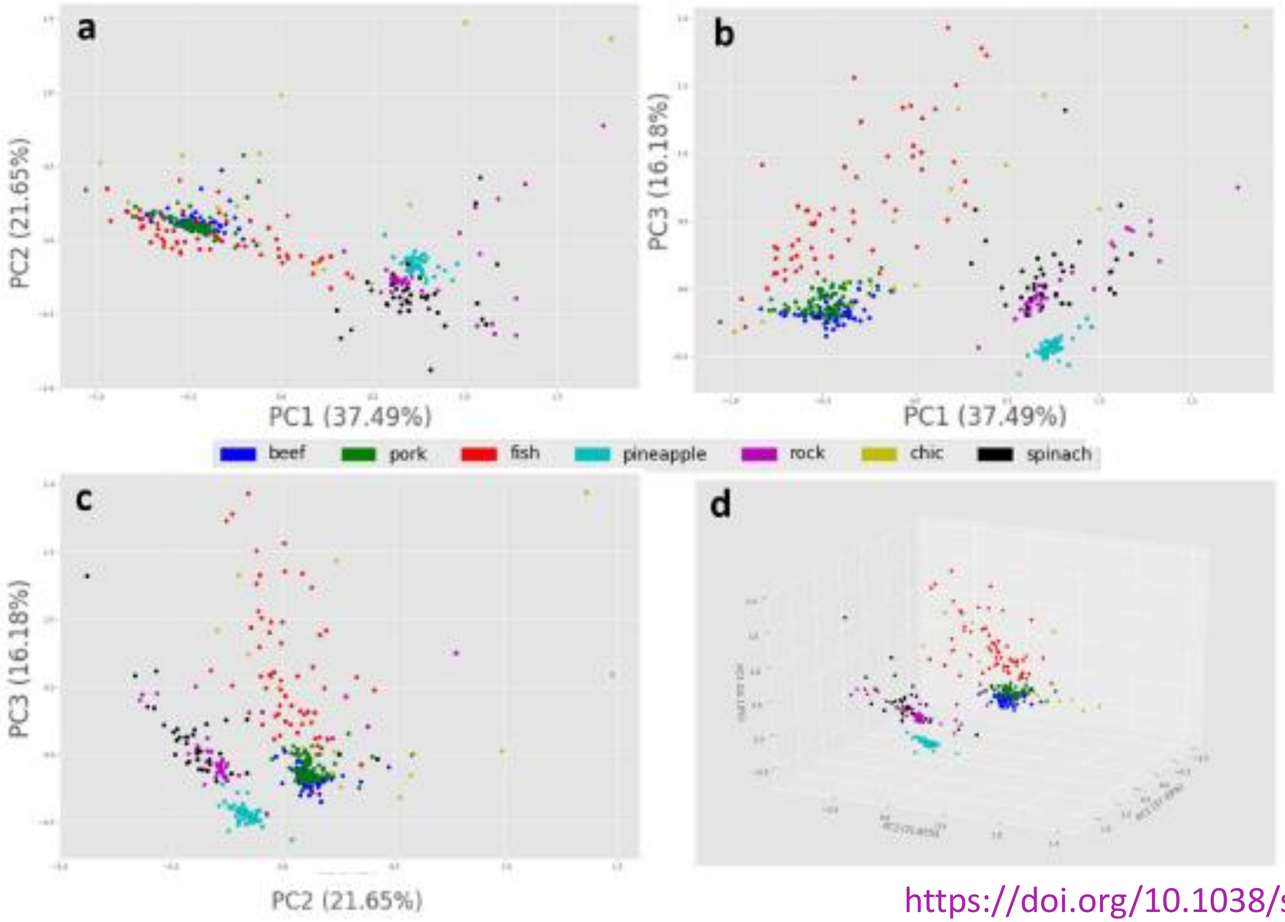
Lemonia-Christina Fengou [*], Panagiotis Tsakanikas, George-John E. Nychas [**]

SCIENTIFIC REPORTS
natureresearch

OPEN

A machine learning workflow for raw food spectroscopic classification in a future industry

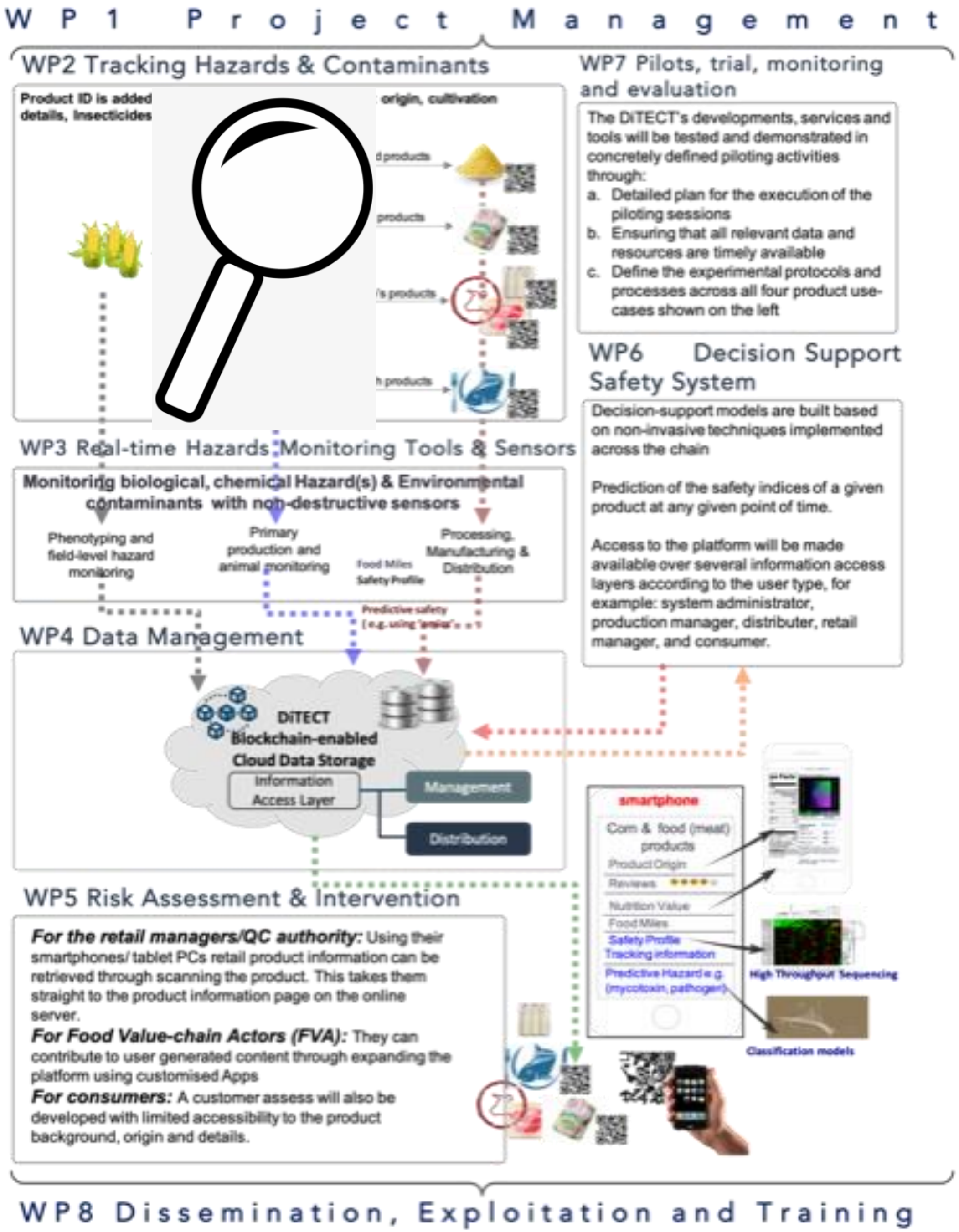
Panagiotis Tsakanikas, Apostolos Karnavas, Efstathios Z. Panagou & George-John Nychas

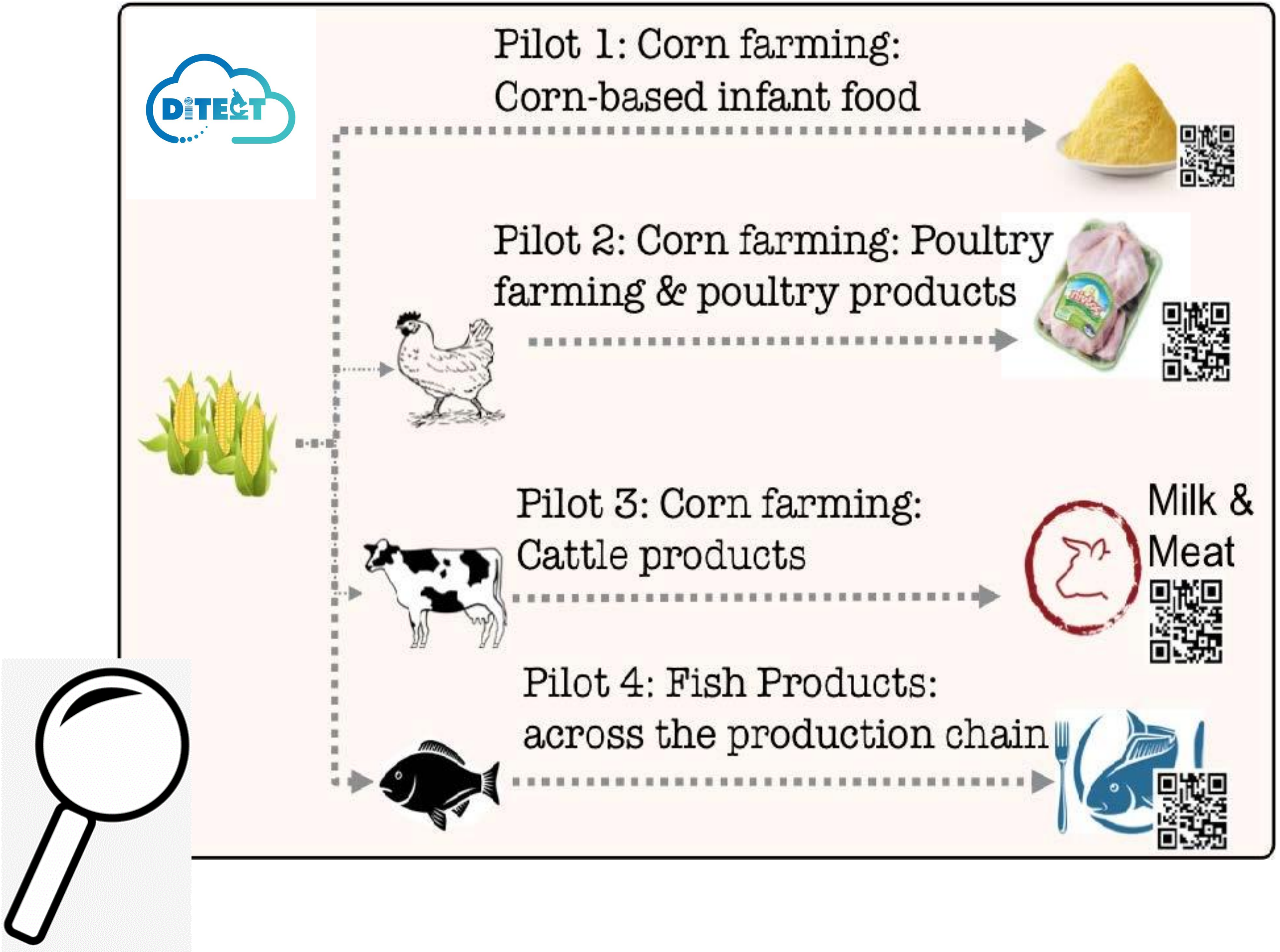


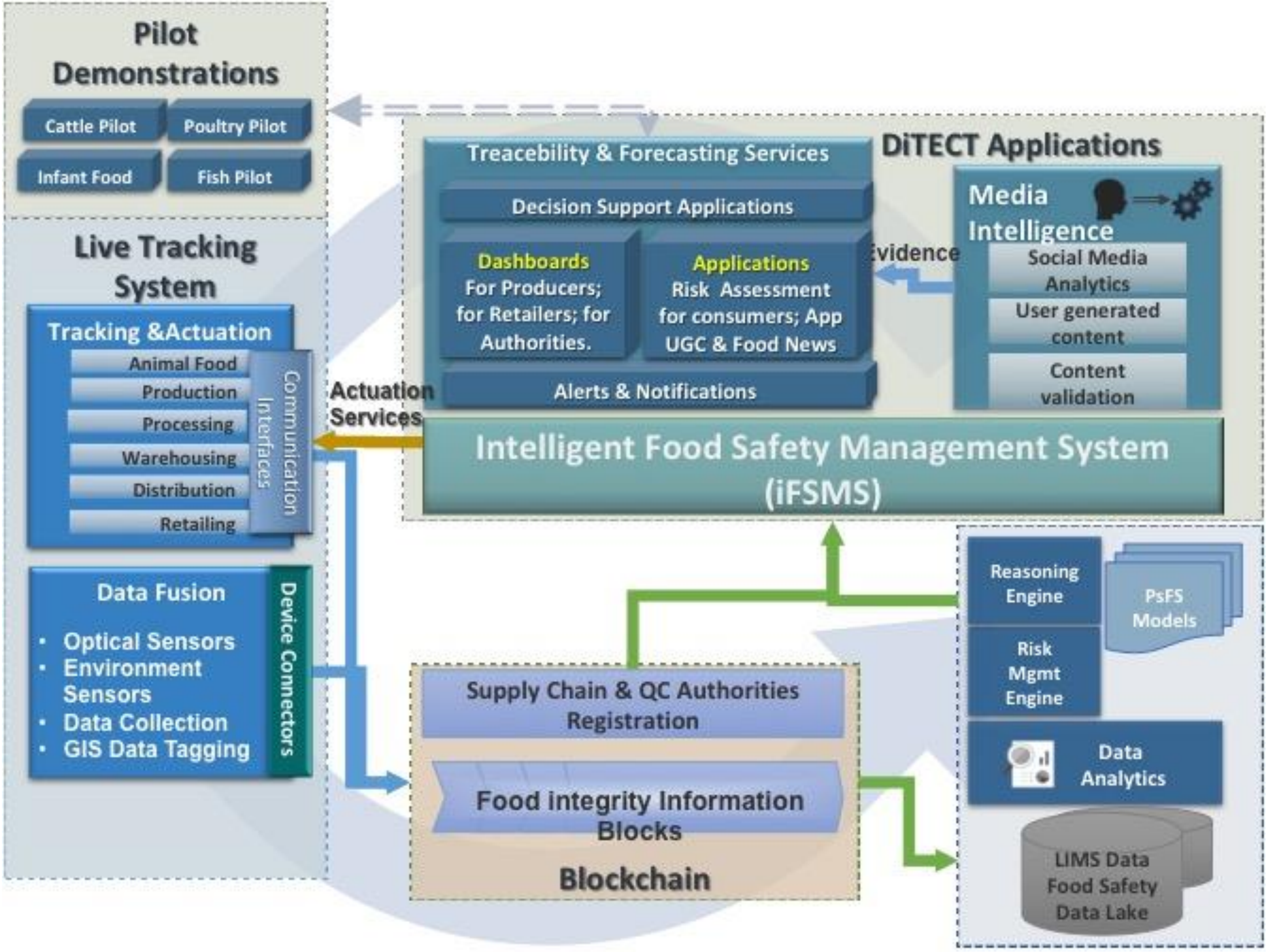
https://doi.org/10.1038/s41598-020-68156-2

Digital TEChnologies as an enabler for a conTinuous transformation of food safety management systems (EU-CHINA project 861915)

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 861915. Coordinated by George-John Nychas gjn@aua.gr


W P 1 P r o j e c t M a n a g e m e n t
WP2 Tracking Hazards & Contaminants
Product ID is added
details, Insecticides
origin, cultivation
WP7 Pilots, trial, monitoring and evaluation
The DiTECT's developments, services and tools will be tested and demonstrated in concretely defined piloting activities through:
a. Detailed plan for the execution of the piloting sessions
b. Ensuring that all relevant data and resources are timely available
c. Define the experimental protocols and processes across all four product use-cases shown on the left
WP6 Decision Support Safety System
Decision-support models are built based on non-invasive techniques implemented across the chain
Prediction of the safety indices of a given product at any given point of time.
Access to the platform will be made available over several information access layers according to the user type, for example: system administrator, production manager, distributer, retail manager, and consumer.
WP3 Real-time Hazards Monitoring Tools & Sensors
Monitoring biological, chemical Hazard(s) & Environmental contaminants with non-destructive sensors
Phenotyping and field-level hazard monitoring
Primary production and animal monitoring
Processing, Manufacturing & Distribution
Food Miles
Safety Profile
WP4 Data Management
DiTECT
Blockchain-enabled
Cloud Data Storage
Information Access Layer
Management
Distribution
smartphone
Corn & food (meat) products
Product Origin
Reviews
Nutrition Value
Food Miles
Safety Profile
Tracking information
High Throughput Sequencing
Classification models
WP5 Risk Assessment & Intervention
For the retail managers/QC authority: Using their smartphones/ tablet PCs retail product information can be retrieved through scanning the product. This takes them straight to the product information page on the online server.
For Food Value-chain Actors (FVA): They can contribute to user generated content through expanding the platform using customised Apps
For consumers: A customer assess will also be developed with limited accessibility to the product background, origin and details.
WP8 Dissemination, Exploitation and Training


DiTECT
Pilot 1: Corn farming:
Corn-based infant food
Pilot 2: Corn farming: Poultry
farming & poultry products
Pilot 3: Corn farming:
Cattle products
Milk &
Meat
Pilot 4: Fish Products:
across the production chain


Pilot Demonstrations
Cattle Pilot
Poultry Pilot
Infant Food
Fish Pilot
Live Tracking System
Tracking &Actuation
Animal Food
Production
Processing
Warehousing
Distribution
Retailing
Communication Interfaces
Data Fusion
Optical Sensors
Environment Sensors
Data Collection
GIS Data Tagging
Device Connectors
Actuation Services
DiTECT Applications
Treacebility & Forecasting Services
Decision Support Applications
Dashboards
For Producers; for Retailers; for Authorities.
Applications
Risk Assessment for consumers; App UGC & Food News
Alerts & Notifications
Media Intelligence
Evidence
Social Media Analytics
User generated content
Content validation
Intelligent Food Safety Management System (iFSMS)
Supply Chain & QC Authorities Registration
Food integrity Information Blocks
Blockchain
Reasoning Engine
PsFS Models
Risk Mgmt Engine
Data Analytics
LIMS Data Food Safety Data Lake

Assessing microbiological quality of chicken burgers using spectroscopy-based sensors

Experimental design

- Six chronically independent batches purchased from a local food industry.
- The **chicken burgers** were stored at: 0, 4, and 8°C.
- Duplicate chicken burgers analyzed at regular time intervals until spoilage was pronounced.

Chicken burgers' ingredients:

- **whole boned chicken (76%),**
- fresh onion,
- fresh pepper,
- soy vegetable protein,
- flavourings,
- dried breadcrumb,
- sunflower oil,
- salt,
- yeast extract, and
- sodium caseinate

Sensors: **Microbiological analysis:**

Partial least squares discriminant analysis (PLSDA) and Support vector machines (SVM) classification models were developed and externally validated (split: 75-25%).

Microbiological quality groups:

A (satisfactory): 4-7 logCFU/g
B (acceptable): 7-8 logCFU/g
C (unacceptable): 8-10 logCFU/g

Table 1. Confusion matrices for PLSDA and SVM model classification for the external validation (n= 68) using FTIR data.

True class	Predicted class			
SVM	**A**	**B**	**C**	**Recall %**
A	15	0	0	100.00
B	2	14	2	77.78
C	0	3	32	91.43
Precision %	88.24	82.35	94.12	89.71
PLSDA				
A	15	0	0	100.00
B	2	12	4	66.67
C	0	4	31	88.57
Precision %	88.24	75.00	88.57	76.19

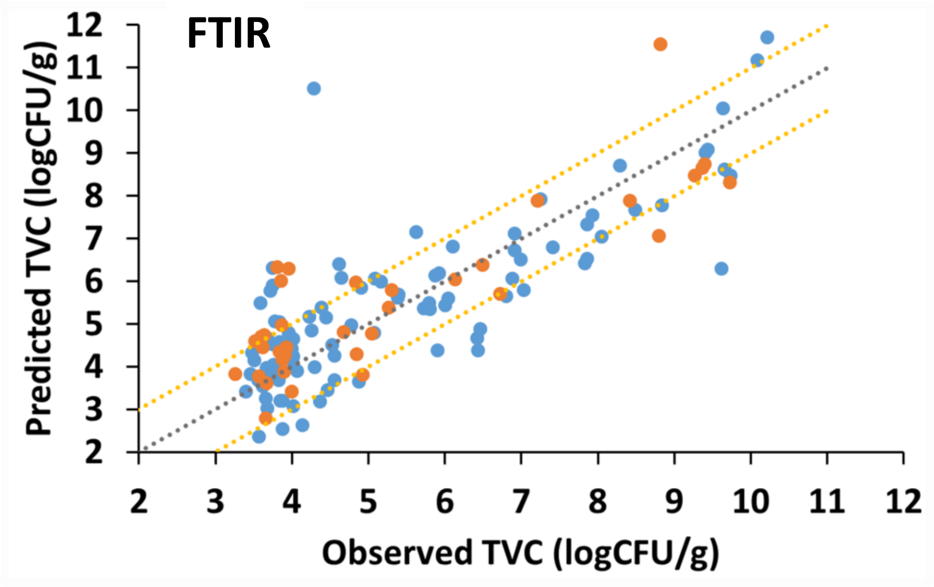
Table 2. Confusion matrices for PLSDA and SVM model classification for the external validation (n= 67) using MSI data.

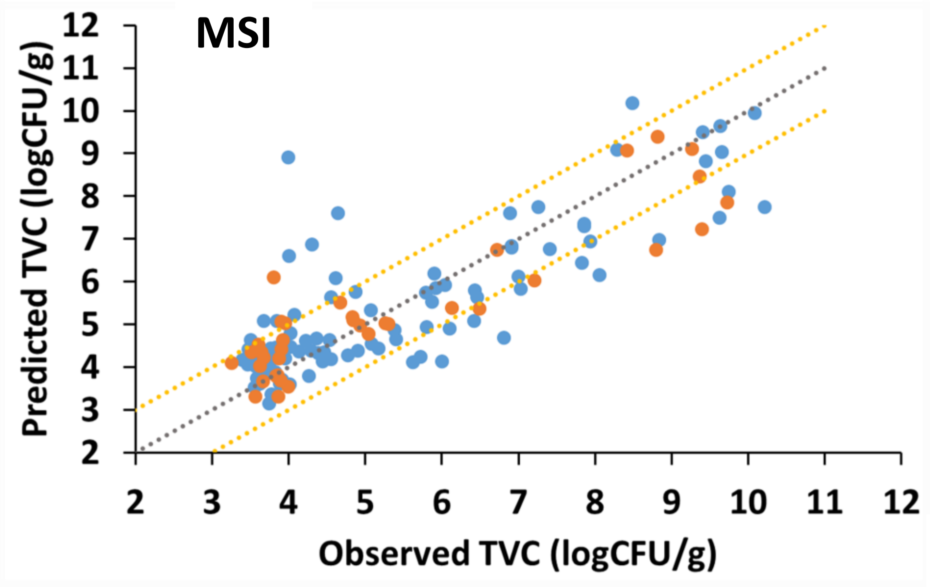
True class	Predicted class			
SVM	**A**	**B**	**C**	**Recall %**
A	13	1	0	92.86
B	1	11	6	61.11
C	0	3	32	91.43
Precision %	92.86	61.11	91.43	83.58
PLSDA				
A	14	0	0	100.00
B	3	11	4	61.11
C	0	3	32	91.43
Precision %	82.35	78.57	88.89	85.07

DiTECT

Fengou et al., 17th Annual Conference of the Metabolomics Society. Metabolomics (online), June 2021

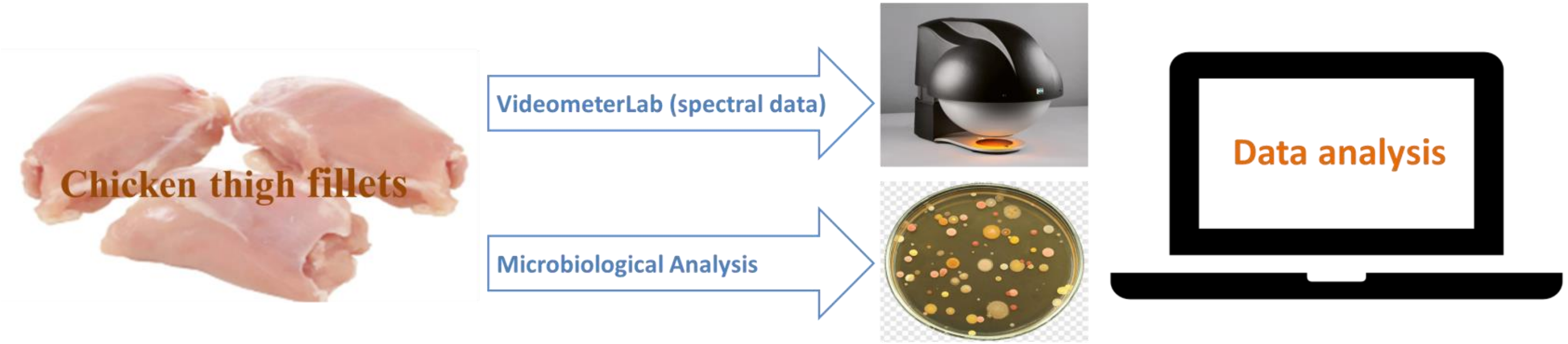
Assessment of the microbiological quality of beef using spectral data





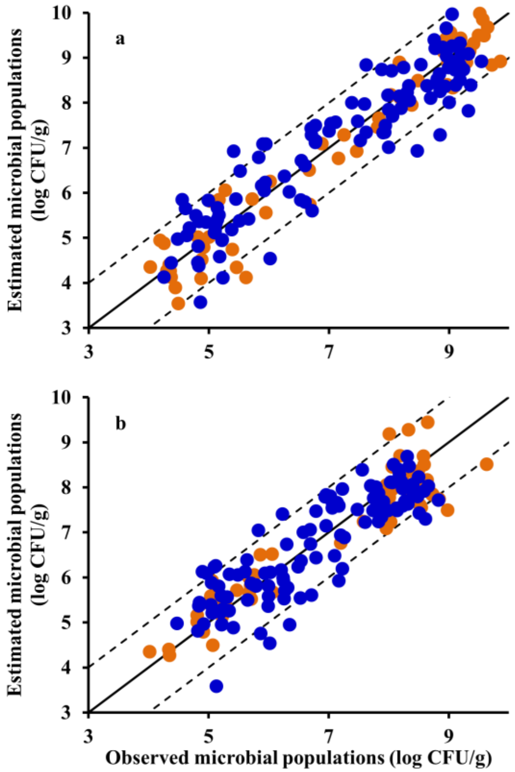
Zegna et al., 9th Conference of MIKROBIOKOSMOS, Athens, Greece, 16-18 December 2021

Multispectral imaging for estimating the microbiological quality of chicken fillets stored under different packaging conditions



Performance metrics of the PLS-R models correlating total mesophilic microbial populations based on MSI data

Packaging	Dataset	Slope	Offset	R^2	RMSE
AIR	Training	0.88	0.91	0.88	0.54
	Cross-validation	0.85	1.09	0.82	0.66
	Testing	1.04	-0.35	0.87	0.57
VACUUM	Training	0.81	1.33	0.81	0.51
	Cross-validation	0.80	1.37	0.72	0.62
	Testing	0.80	1.33	0.83	0.57



Munich GERMANY 4-6 May 2022

Tsekos et al., IAFP's European Symposium on Food Safety 2022, P2-30

PAT concept applications in food quality assessment and adulteration detection

P1-22 - Estimation of the **Microbiological Status of Chicken Burgers** through Fourier Transform Infrared Spectroscopy (FT-IR)

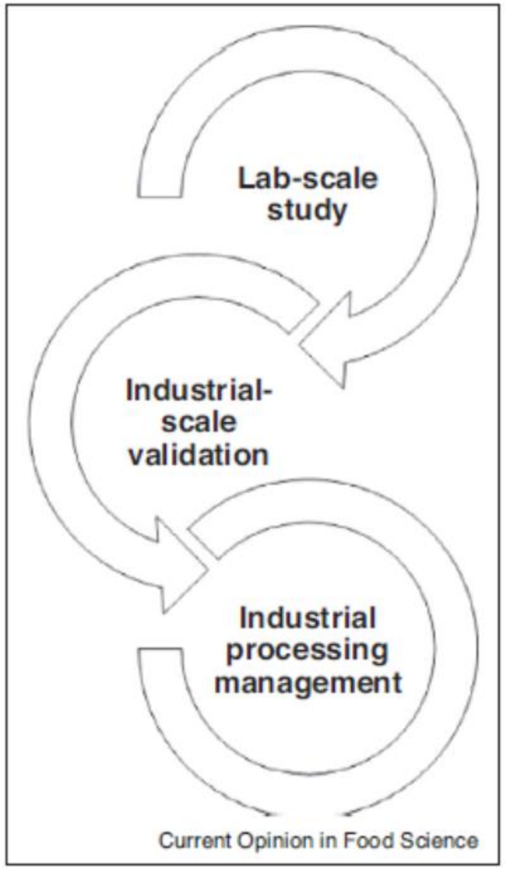
P1-23 - Fourier-Transform Infrared Spectroscopy Coupled with Support Vector Machine Analysis for **Chicken Liver Spoilage** and Safety Assessment

T4-05 - Detection of **Adulteration in Raw and Cooked Beef** Using Multispectral Imaging

T6-04 - Volatilomics in Tandem with Machine Learning for the **Quality Assessment of Chicken Meat**

Actual application of PAT in the food industry

- Scientific literature: considerable potential
- Actual industrial-scale applications: limited
- Sensors: substitutes of off-line analytical procedures
- Gap between potential and actual applications
- Knowledge dissemination and close cooperation between researchers and food industry stakeholders



Grassi and Alamprese, 2018; https://doi.org/10.1016/j.cofs.2017.12.008

Outlook…

- Enrichment of current scientific knowledge: further development and improvement of non-invasive analytical technologies and sensors
- Better understanding of the PAT concept by regulatory authorities and stakeholders
- Extension of the PAT framework to food protection issues beyond quality (safety, authenticity, adulteration)
- Significant contribution of MEMS and IT in the provision of integrated approaches

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Alexandra Lianou & George-John Nychas

thank you