# New Developments for the Optimal Operation of Spirits Distillation

José Ricardo Pérez Correa

Chemical and Bioprocess Engineering Department

Pontificia Universidad Católica de Chile

# Outline

- Our University
- Our School
- Our Department
- Ongoing research
  - Problem description
  - Methodology
  - Preliminary results
  - Next steps
- Proposed research at UFSC

# Pontificia Universidad Católica de Chile

- The University was founded in 1888, with two faculties:
  - Law
  - Physical Sciences and Mathematics

- 1.800 full time academics

- 25.000 undergraduate students in 103 programs (10 with international accreditation)

- 3.400 master students enrolled in 92 programs

- 1.100 PhD students enrolled in 34 programs

- Agreements with 585 universities from 55 countries

- 1.400 foreign students from 42 countries

# School of Engineering at PUC

- In 1896 a Civil Engineering program opened with 30 students
- 10 Departments
- 168 full time academics (44% young Assistant Professors)
- 4.166 undergraduate students in 14 Disciplinary Majors & 7 Interdisciplinary Majors
- 318 master students
- 226 PhD students

# Chemical and Bioprocess Engineering Dept.

- 1960, hire of the first full time PhD, Prof. Jaime Wisniak

- 18 full time academics (include 4 share positions and 1 emeritus professor: José Miguel Aguilera, recipient of the Chilean National Prize in Applied Sciences & Technology)

- 146 undergraduate students in 1 Disciplinary Major and 1 Interdisciplinary Major

- 17 master students

- 37 PhD students

# Ongoing research: problem description

## Elaboration of fruit distillates

Harvest

Milling

Fermentation

Distillation

Aging

Bottling

Production of
Ethanol + aromas

Recovery of
Ethanol + aromas

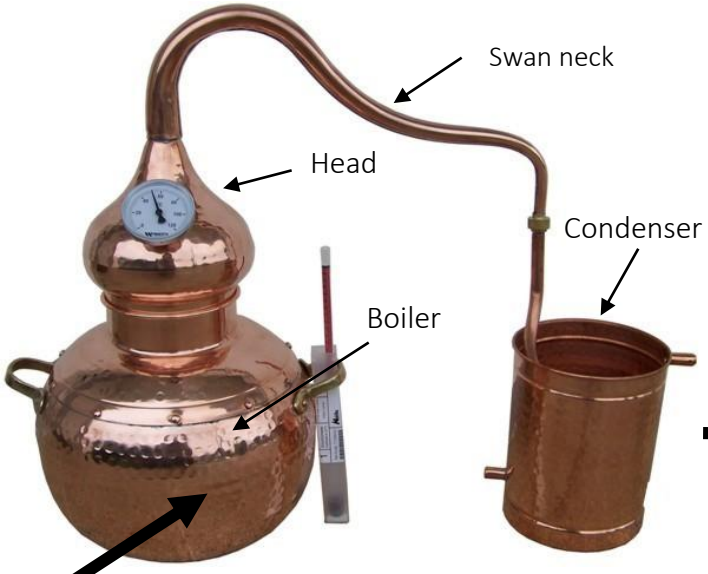
Aromas

Quality

Final product

# Ongoing research: problem description

## Charentais alembics are mostly used at small scale




Wine

**Batch & Nonlinear process**

The distillate is separated in fractions (cuts)

Head Heart Tail

water-ethanol -> 97%

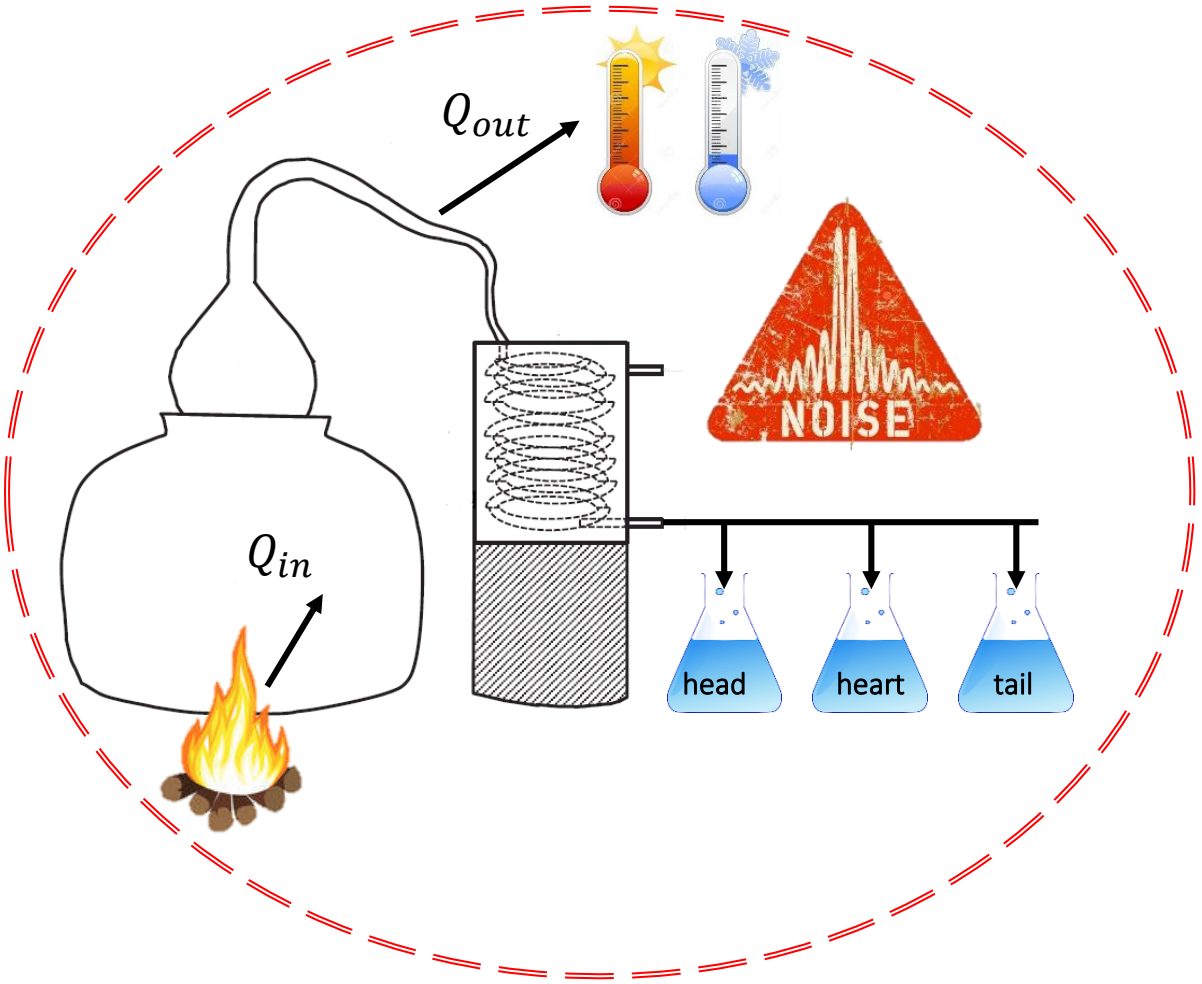
minority compounds -> 3%

Positive: terpenes, esters, higher alcohols

Negative: fatty acids, methanol, acetaldehide, furfural

# Ongoing research: problem description

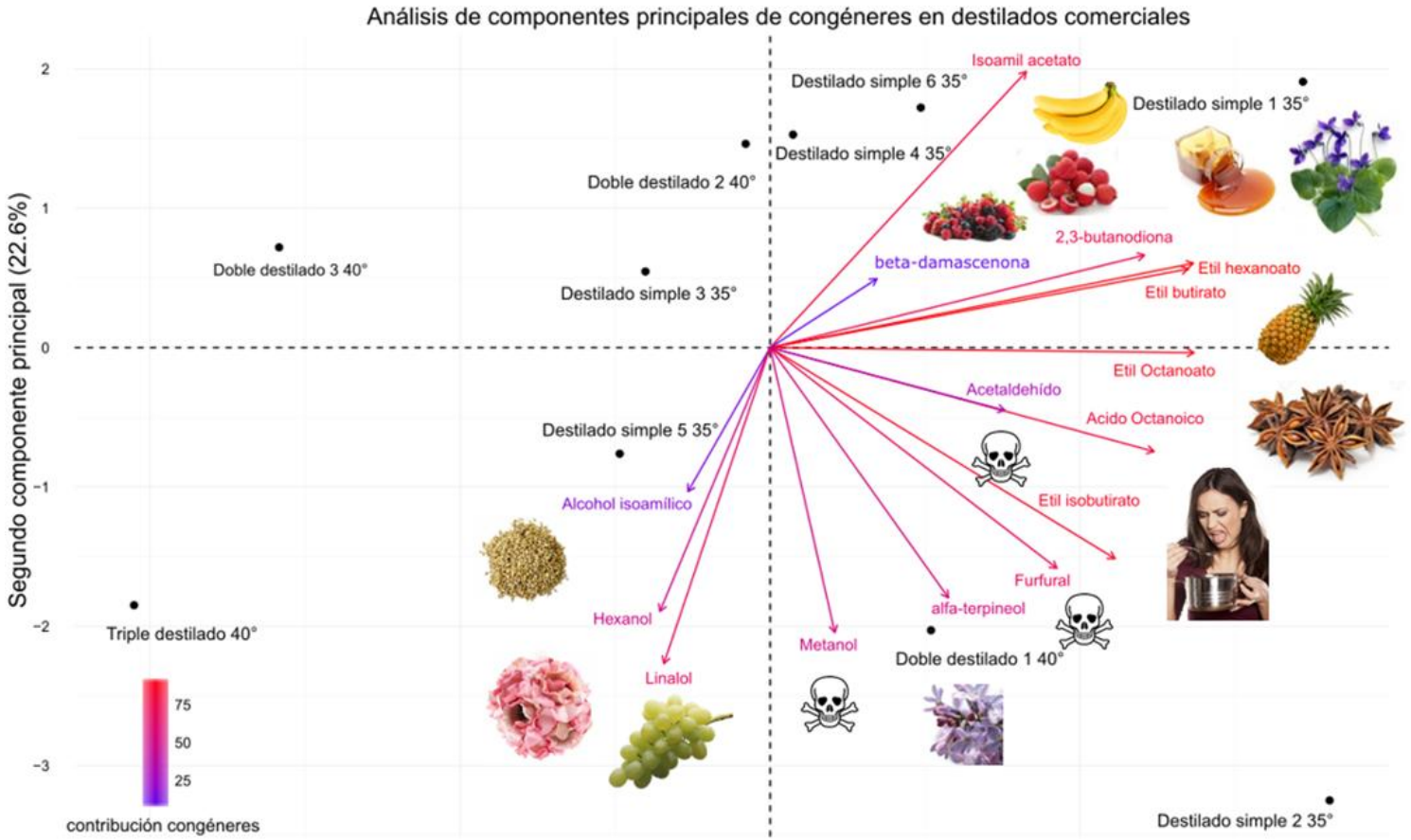
Normal operation is carried out manually and heuristically




1.0 g Acetaldehyde/L ethanol
0.08 g Furfural/L ethanol
1.5 g Methanol/L ethanol

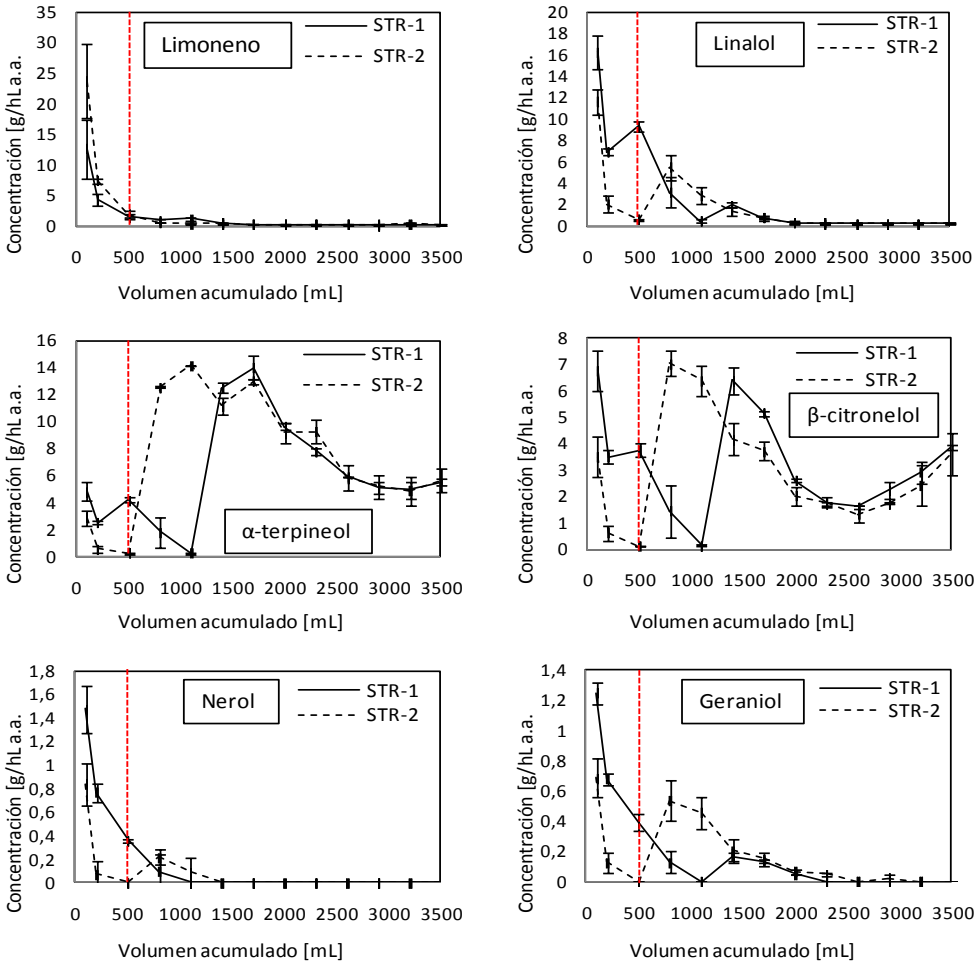
# Ongoing research: problem description

## Minority compounds and distillate quality



# Ongoing research: problem description

## Operating policies define the aromatic composition of the distillates

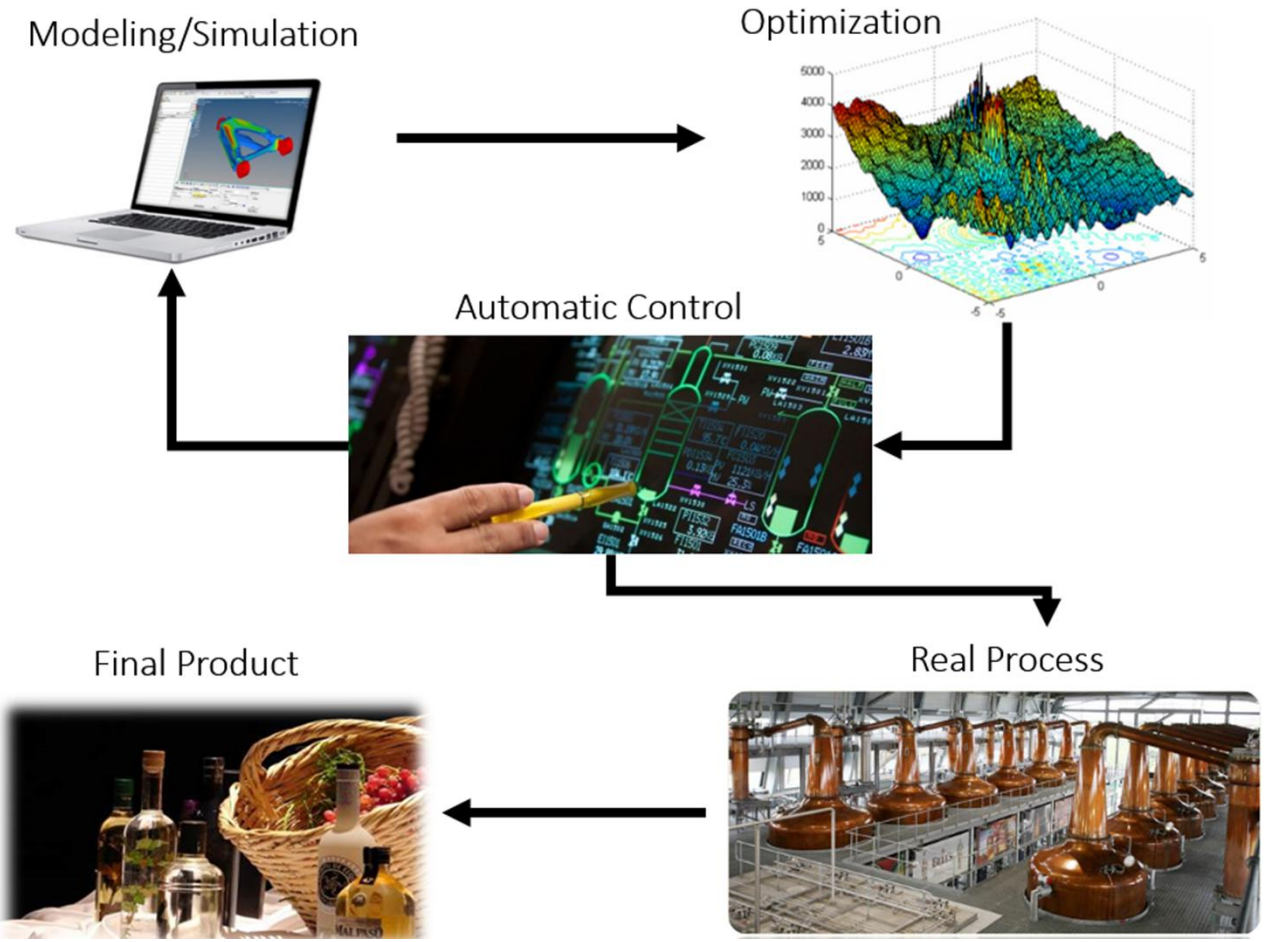





Matías-Guiu P, Rodríguez-Bencomo JJ, Orriols I, Pérez-Correa JR, López F., Food Chem ; 2016;213:40–8.

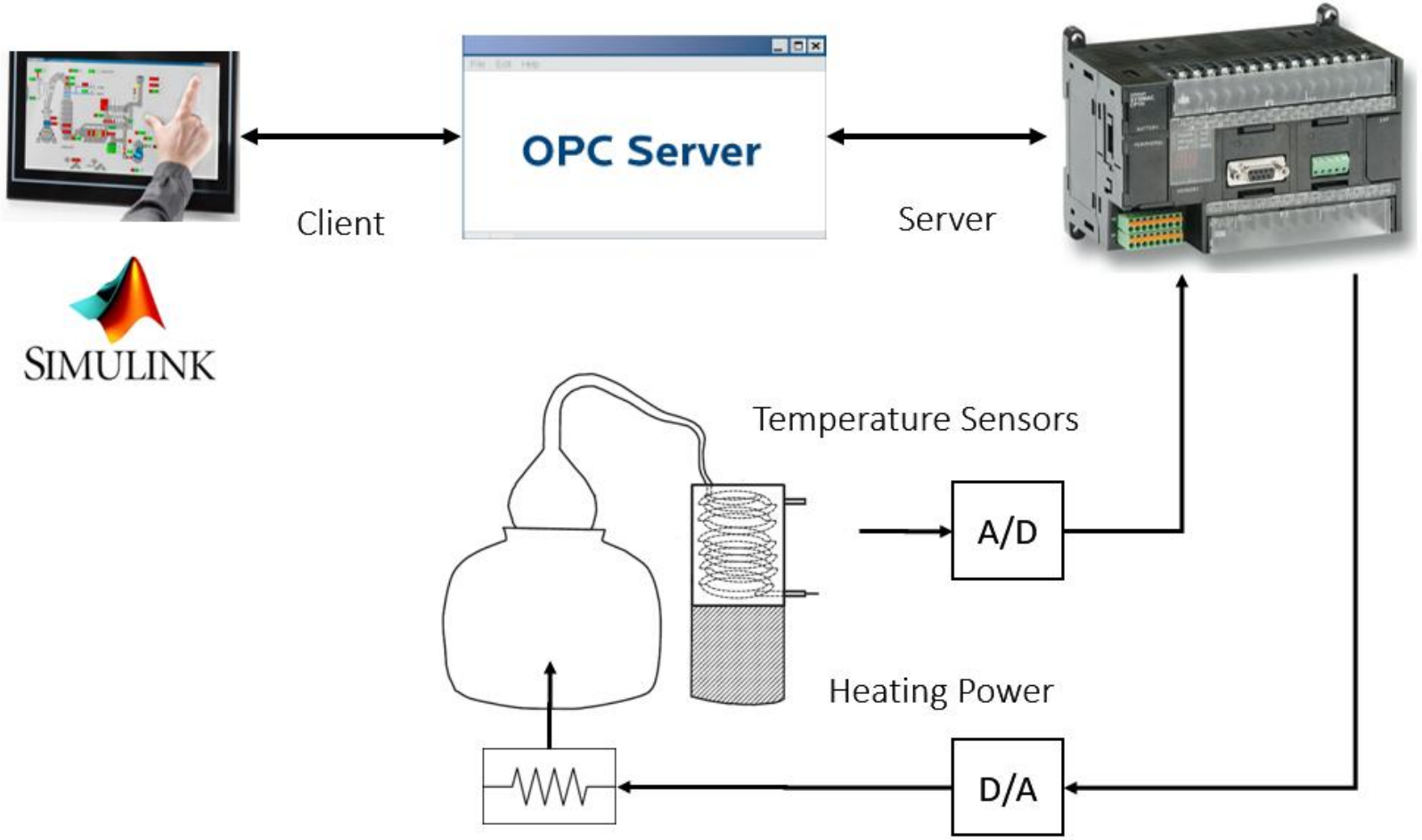
# Ongoing research: methodology

We aim to develop model based operating policies to achieve aromatically enhanced and safer distillates



# Ongoing research: methodology

## Experimental System



# Ongoing research: methodology

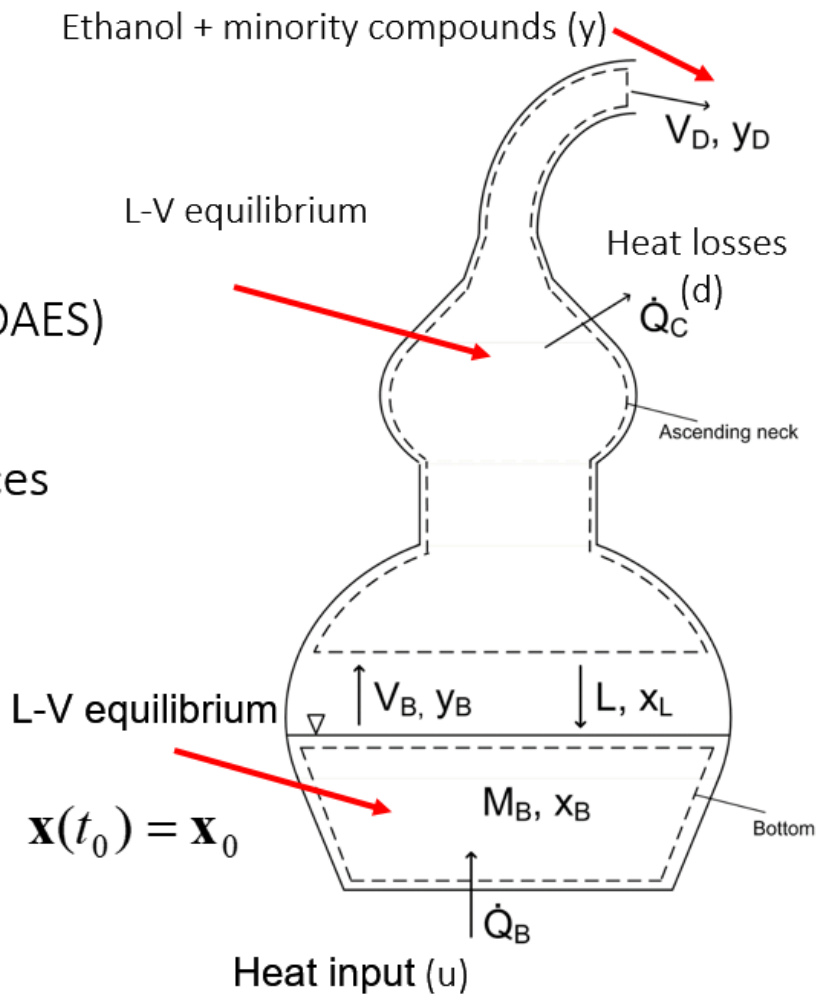
## Modeling

First principles model (DAES)

- Mass & energy balances
- Two stages

$$\frac{d\mathbf{x}}{dt} = \mathbf{f}(\mathbf{x}(t), \mathbf{y}(t), \mathbf{u}(t)), \quad \mathbf{x}(t_0) = \mathbf{x}_0$$

$$\mathbf{g}(\mathbf{x}(t), \mathbf{y}(t), \mathbf{u}(t)) = 0$$






Sacher J, García-Llobodanin L, López F, Segura H, Pérez-Correa JR., Food Bioprod. Process; 2013;91(4):447–56.

# Ongoing research: methodology

## Model Calibration

$$J = \left(\frac{V(t) - V_{exp}(t)}{V_{exp}(t)}\right)^2 + \left(\frac{T_c(t) - T_{c,exp}(t)}{T_{c,exp}(t)}\right)^2 + \left(\frac{GA(t) - GA_{exp}(t)}{GA_{exp}(t)}\right)^2 + \left(\frac{Met(t) - Met_{exp}(t)}{Met_{exp}(t)}\right)^2$$

s.a:

$$\frac{dx_i}{dt} = f(x_i(t), y_i(t), u(t)), \quad x(t_0) = x_0$$

$$g(x_i(t), y_i(t), u(t)) = 0$$

| Potencia | $UA_b$ | $UA_c$ | $M_0$ | $x_0^e$ | $x_0^m$ |
|---|---|---|---|---|---|
| 400 | 0.71±0.11 | 1.13±0.17 | 90.3±0.1 | 38.60e-3±0.99e-3 | 1.030e-4±0.012e-4 |
| 230 | 0.95±0.09 | 0.65±0.10 | 89.3±0.5 | 38.70e-3±0.44e-3 | 1.010e-4±0.001e-4 |



Ricardo Luna, Francisco López, José R. Pérez-Correa, Journal of Industrial and Engineering Chemistry, in press, 2017.

# Ongoing research: methodology

## Multiobjective Dynamic Optimization

Sequential method → 

→ *Scatter Search*
(Egea et al. (2007))

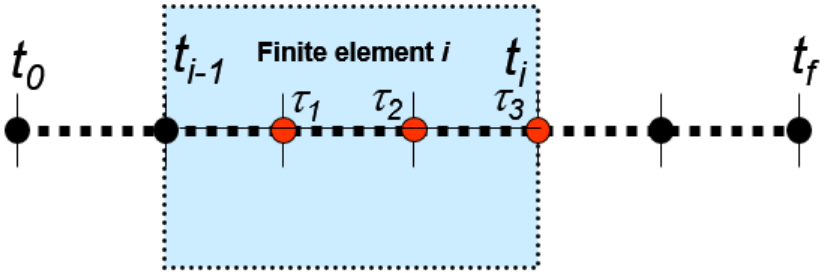
$u(\mathrm{t})$ Discretization → $u(\Delta t_i) = a_i$

Simultaneous method → 

(Fourer et al. (2003)) → *IPOPT*
(Waechter & Biegler (2003))

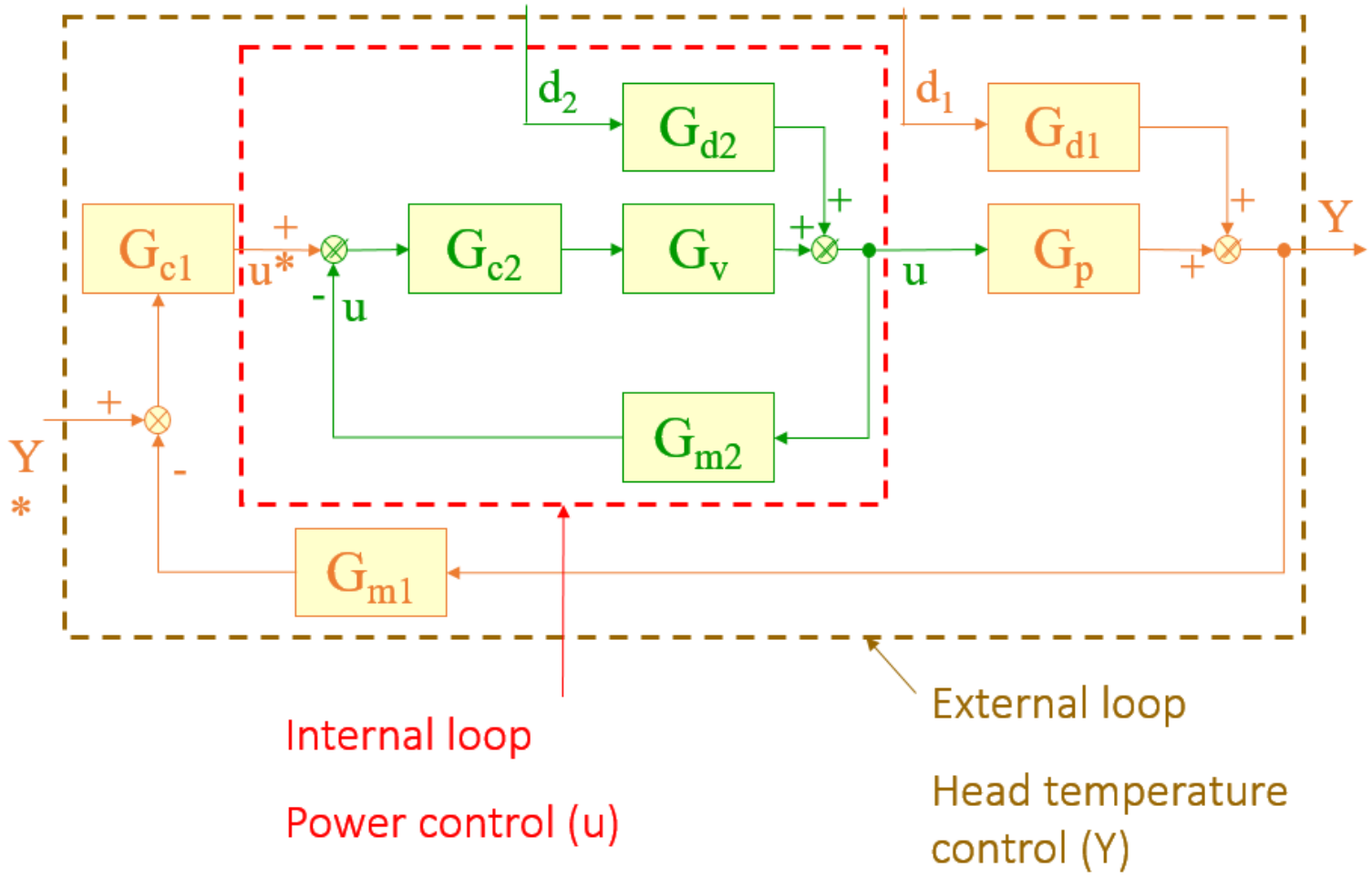
Orthogonal colocations

$$\frac{dx}{dt_{i,j}} = f(x_{i,j}, y_{i,j}, u_{i,j}, \theta) \longrightarrow x_{i,j} = x_{i-1} + h_i \sum_{j=1}^{ncp} \Omega_j(\tau_j) \cdot \frac{dx}{dt_{i,j}}$$



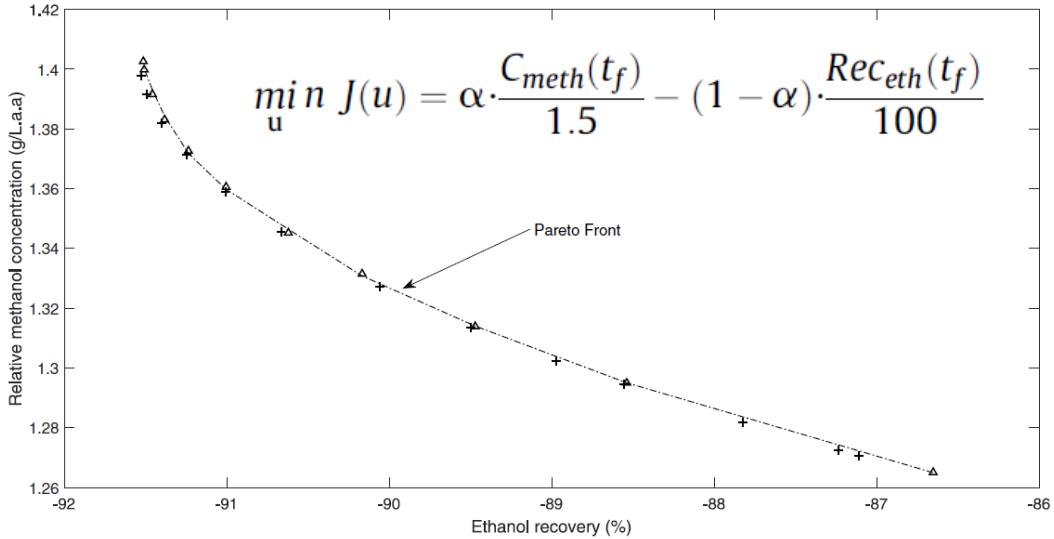
# Ongoing research: methodology

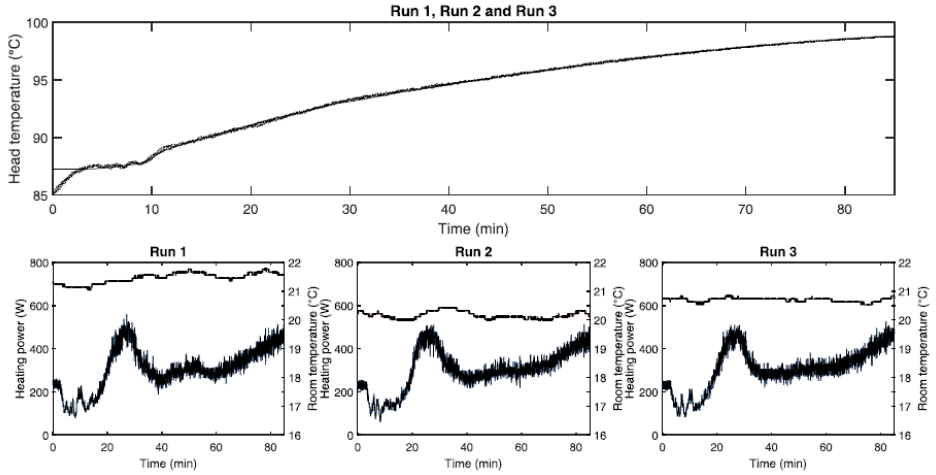
## Cascade control (Internal Model Control in both loops)



# Ongoing research: results

## Multiobjective optimization

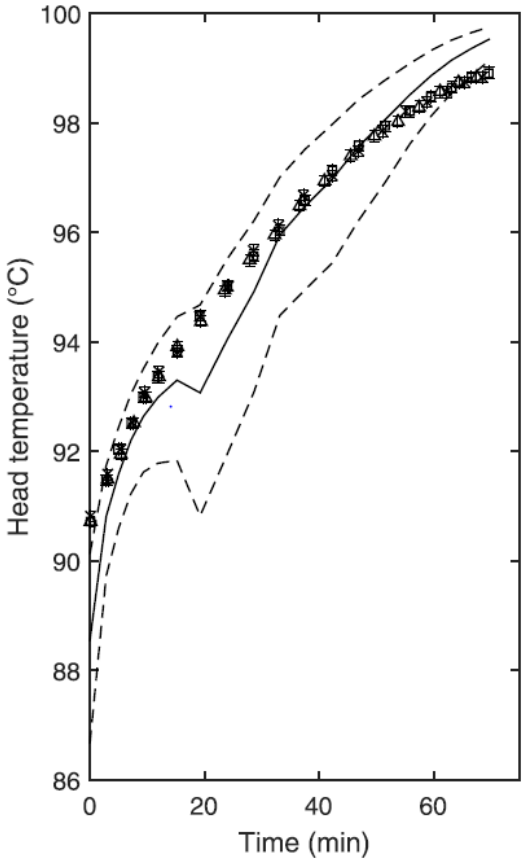


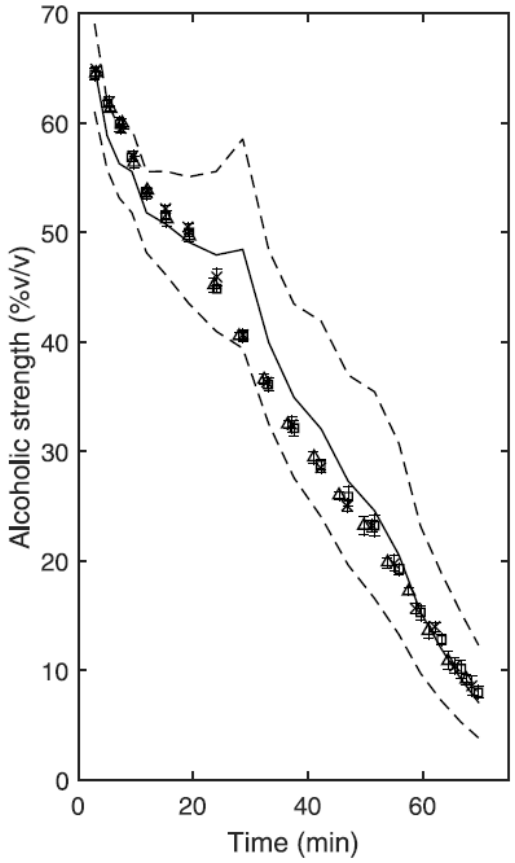



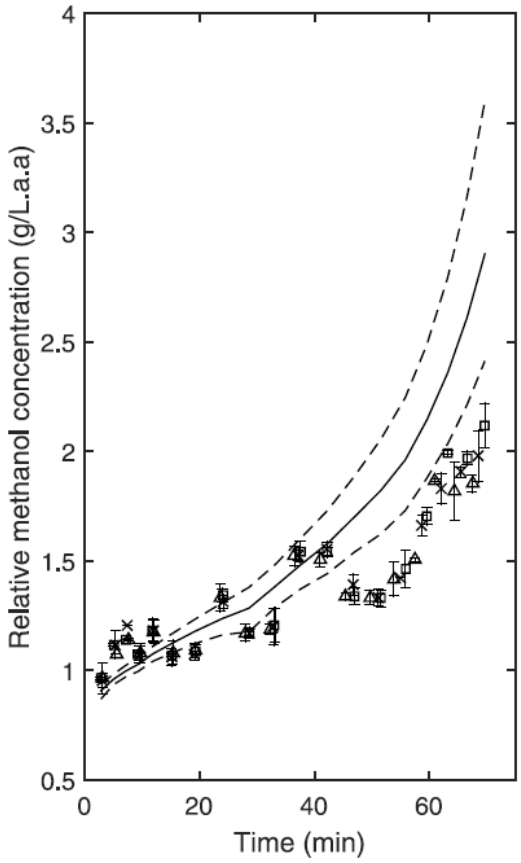

Ricardo Luna, Francisco López, José R. Pérez-Correa, Journal of Industrial and Engineering Chemistry, in press, 2017.

# Ongoing research: results

## head temperature & alcohols concentrations



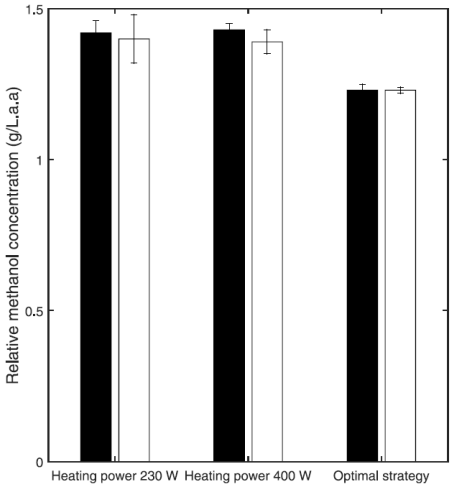


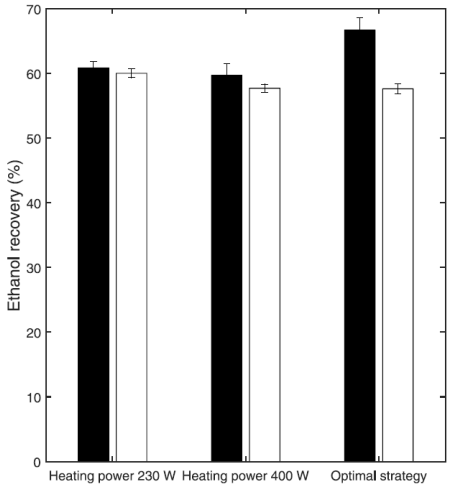

Ricardo Luna, Francisco López, José R. Pérez-Correa, Journal of Industrial and Engineering Chemistry, in press, 2017.

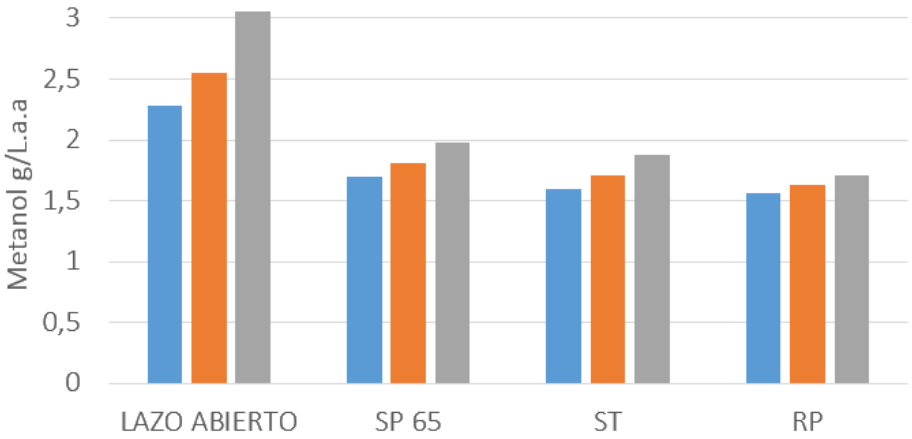
# Ongoing research: results

## methanol content vs ethanol recovery

predicted values (■) and experimental data (□).



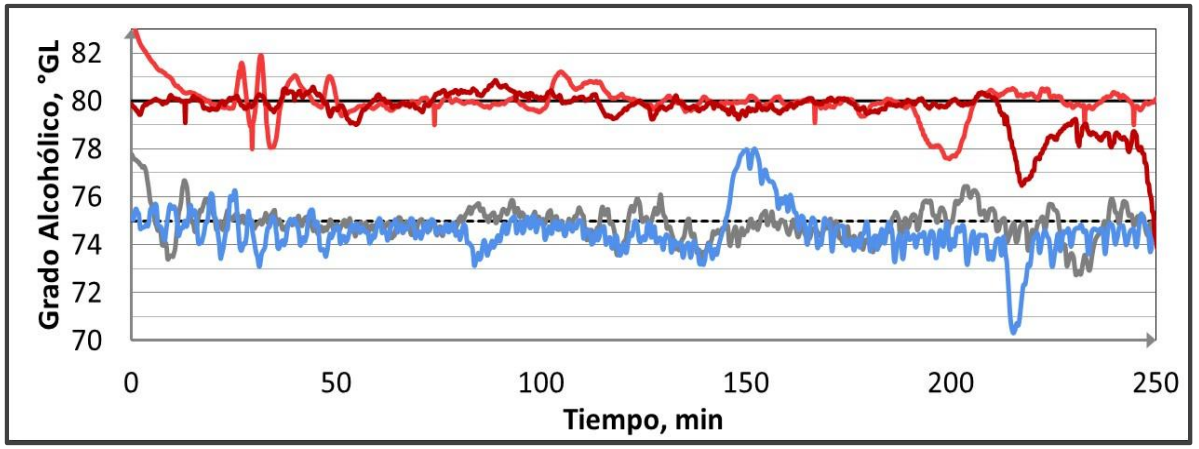

Ricardo Luna, Francisco López, José R. Pérez-Correa, Journal of Industrial and Engineering Chemistry, in press, 2017.

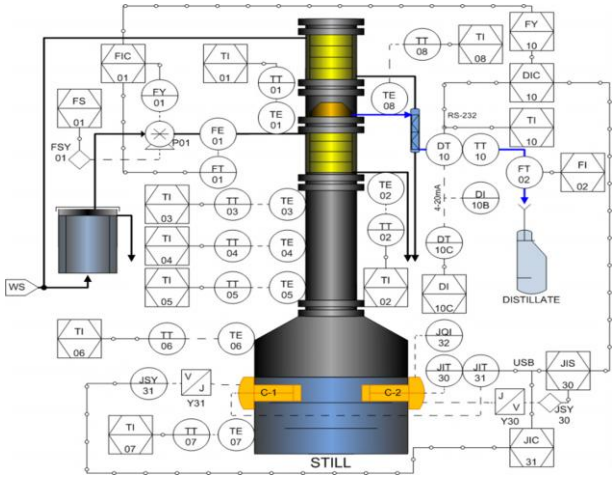
# Ongoing research: next steps

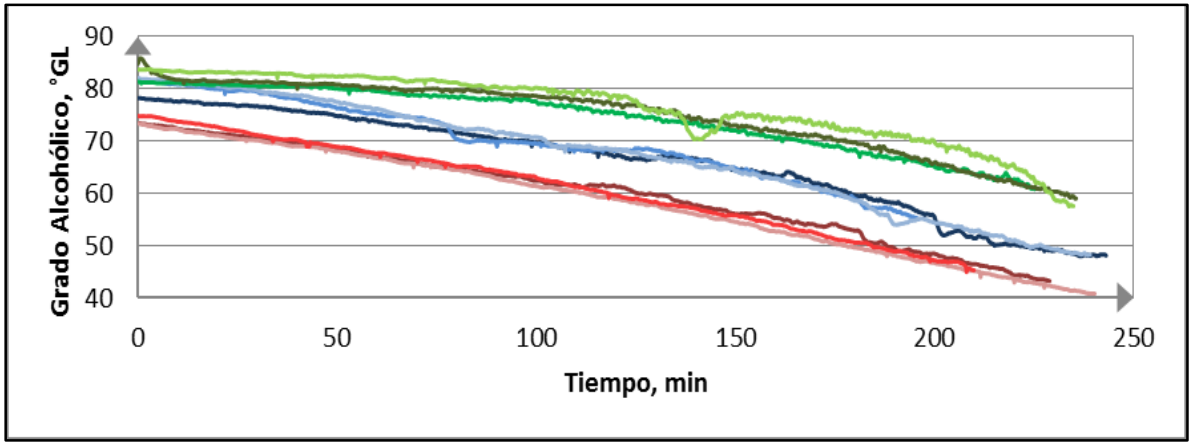
- Application of multiobjective optimization with more objectives (distillation time, productivity, energy consumption, safety, aromatic quality)
- Define operating strategies to enhance the safety and aromatic quality of the spirits
  - experimental tests with synthetic wines
  - experimental tests and sensory assessments with real wines
- Apply this technology to batch stills with rectification columns (production of Pisco in Chile, Armagnac in France, Grappa in Italy)
- Apply this technology to the wine industry
- Develop a low cost tailor made technology package for the spirits industry

# Proposed research at UFSC: distillation

## Advanced process Control of a Batch Still with Packed Rectification Column

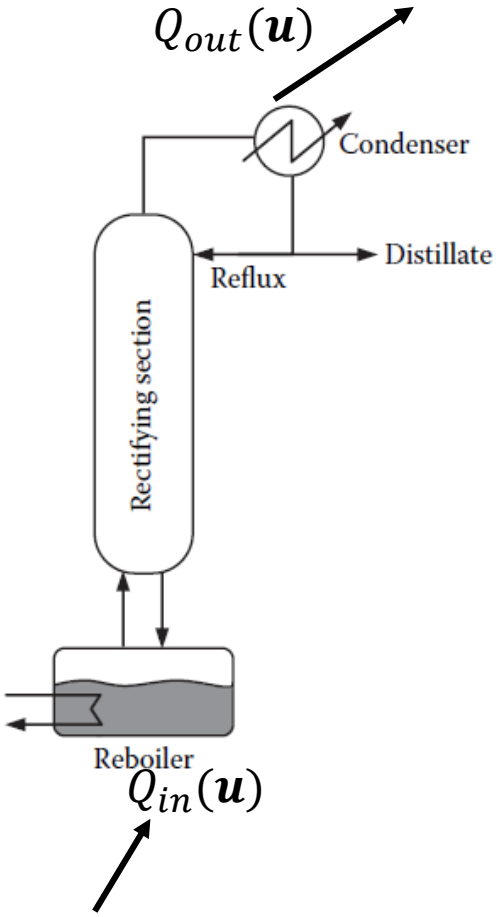


# Proposed research at UFSC: distillation

## Dynamic Model



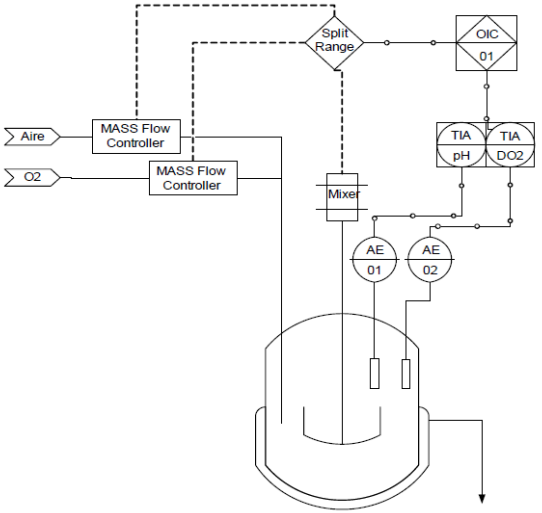

- Mass & energy balances
- Vapor/Liquid equilibrium
- Transport phenomena

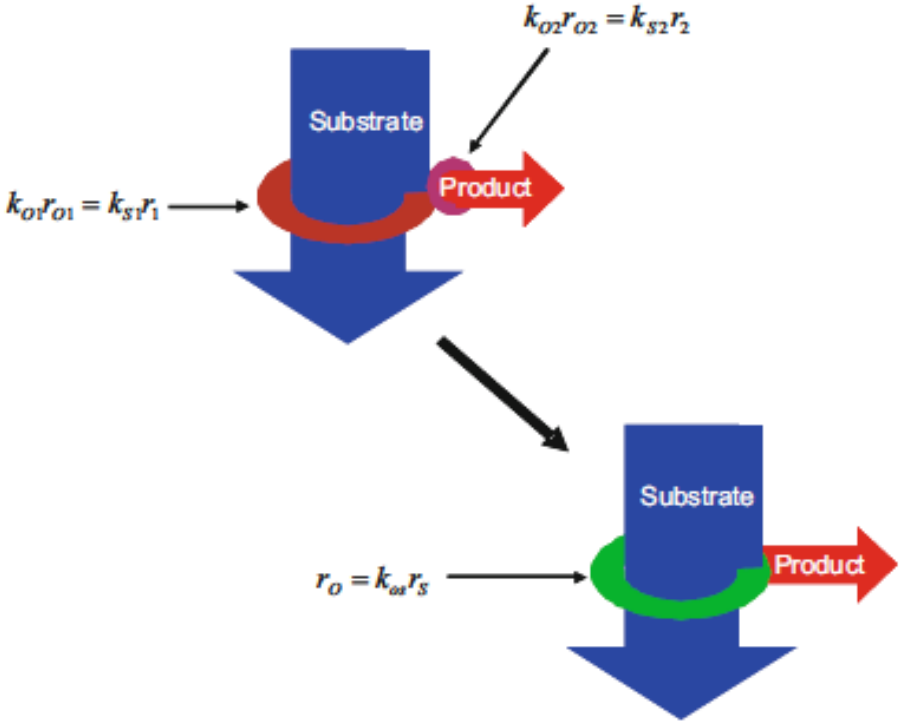
$$\frac{d\mathbf{x}}{dt} = \mathbf{f}(\mathbf{x}(t), \mathbf{y}(t), \mathbf{u}(t)), \quad \mathbf{x}(t_0) = \mathbf{x}_0$$

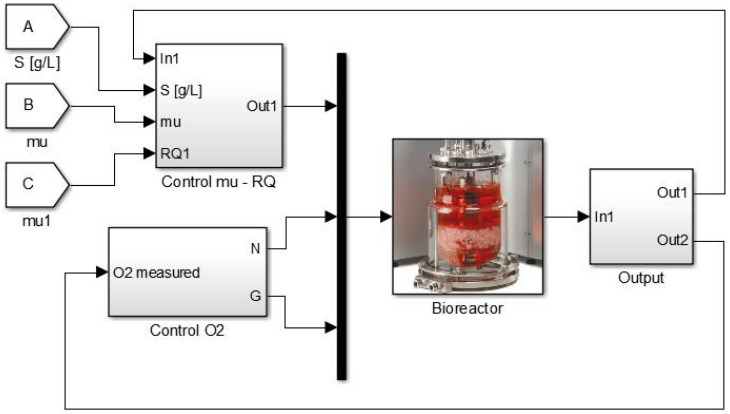
$$\mathbf{g}(\mathbf{x}(t), \mathbf{y}(t), \mathbf{u}(t)) = 0$$

# Proposed research at UFSC: yeasts fed-batch cultivations

## Advanced Process Control of high density cultures of yeasts (presenting overflow metabolism and oxygen limitations)

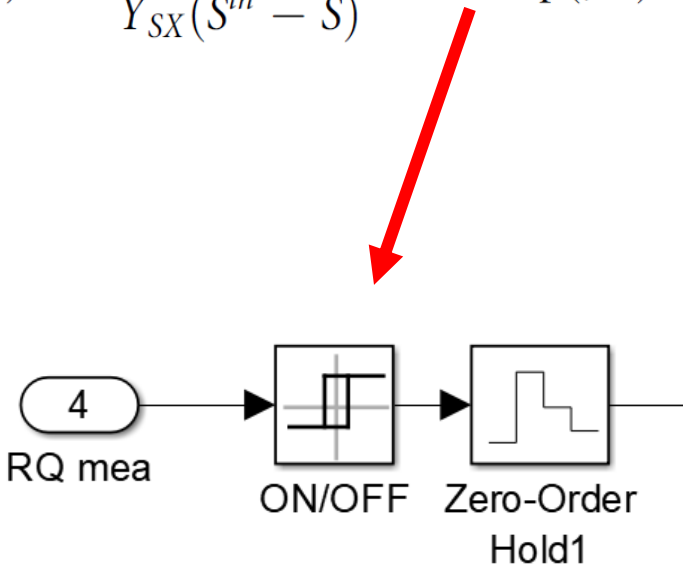


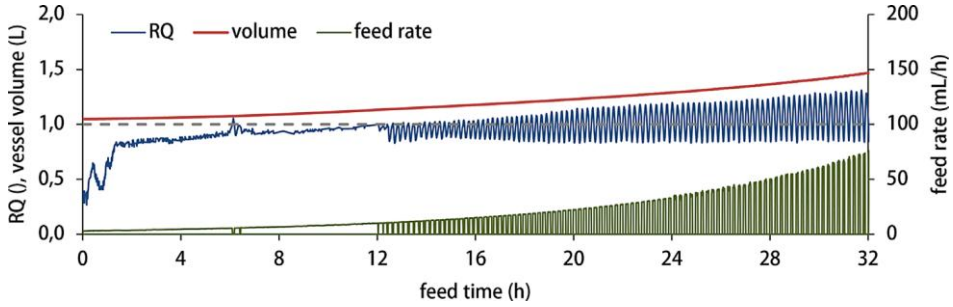



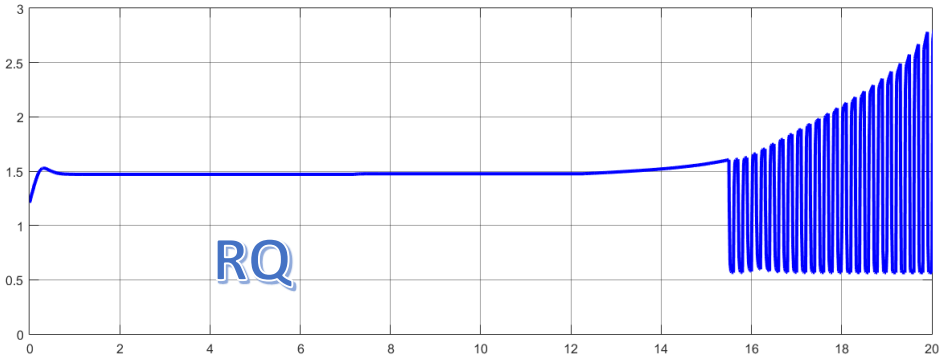



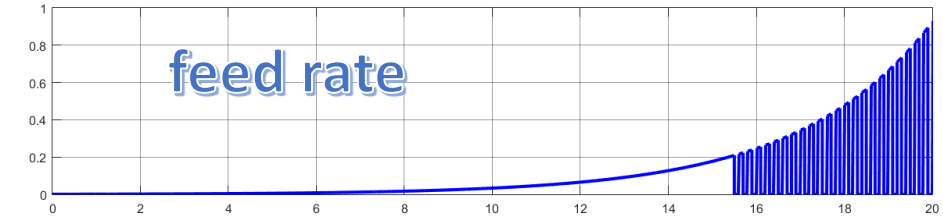
# Proposed research at UFSC: yeasts fed-batch cultivations

$$v(t) = \frac{\mu X_0 V_0 \exp(\mu t)}{Y_{SX}(S^{in} - S)} = v_0 \exp(\mu t)$$



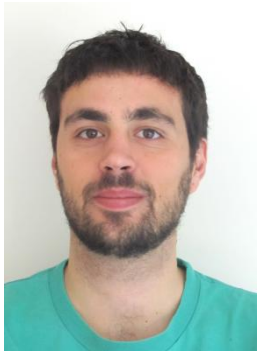



# Collaborators

Francisco López

Juan José Rodriguez-Bencomo



Pau Matías-Guiu

Ignacio Orriols

Mario Fernández

Ricardo Luna

Simon Díaz