

# An integrated process for utilization of unused chokeberries



**P. Tzatsi, D. Fotiou, D. Karipoglou, E.G. Stampinas,  
A.M. Goula**

*Department of Food Science and Technology, School of Agriculture, Forestry  
and Natural Environment, Aristotle University, Thessaloniki, Greece*

# Chokeberry

- *Aronia* is a member of the *Rosaceae* family
- Two species can be distinguished:
  - ✓ *Aronia melanocarpa* (black chokeberry)
  - ✓ *Aronia arbutifolia* (red chokeberry)



The most important growing regions are:

- North America
- East Canada
- Germany
- Russia



# Composition-Polyphenol Content of Chokeberry

Component	Content (%)
Total solids	25.60
Moisture	74.40
Total sugars	10.00
Proteins	0.70
Crude Fiber	5.60
Fat	0.15
Ash	1.30
Total phenolics	7.85



Phenolic compound	Content (mg/100g dry matter)
Procyanidins	5,182
Anthocyanins	1,959
Quercetin	101
Catechin	15.4
Chlorogenic acid	302
Neochlorogenic acid	291

- ❖ Antioxidant activity
- ❖ Anti-mutagenic activity
- ❖ Anti-hypertension activity
- ❖ Anti-inflammatory activity
- ❖ Anti-atherosclerotic activity

Kulling & Rawel, 2008

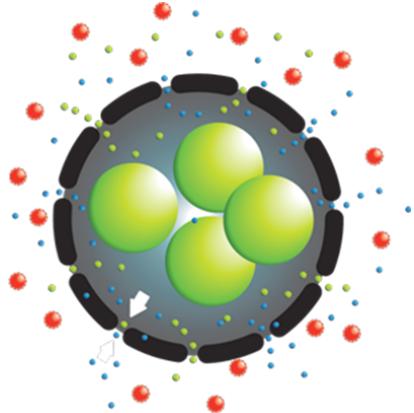
# Applications of chokeberry



# Encapsulation of phenolic compounds

1

Masking of astringency



2

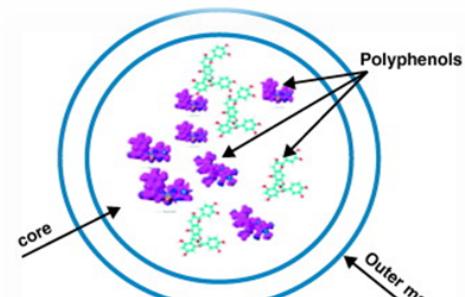
Improvement of color

3

Suitability for use as an additive in functional foods

4

Increase of their stability during storage and passage through the gastrointestinal tract



*Shahidi & Han, 1993*

# Encapsulation methods

Encapsulation method	Encapsulation efficiency (%)	Reference
Spray drying	99.8	Kaderides et al., 2015
Freeze drying	97.22	Saikia et al., 2015
Spray chilling	91.3	Sukransik et al., 2018
Rotating disk	80.0	Akhtar et al., 2014
Yeast encapsulation	70.0	Gonzalez et al., 2019
Emulsions	86.6	Guldiken et al., 2019



# Wall material characteristics

1

Good rheological properties at high concentration

2

Ability to disperse or emulsify the active material  
and stabilize the emulsion produced

3

Non reactivity with the material to be encapsulated

4

Ability to provide maximum protection to the  
active material against environmental conditions  
(e.g., heat, light, humidity)

5

Chemical non reactivity with the active material

6

Ability to seal and hold the active material within  
its structure during processing or in storage



# Wall materials used for encapsulation of phenolic compounds

<b>Phenolic extract</b>	<b>Encapsulation method</b>	<b>Wall material</b>	<b>Reference</b>
Pomegranate peel extract	Spray drying	Maltodextrin; Whey protein; Skim milk powder	Kaderides et al., 2015
Blueberry juice	Spray drying & Freeze drying	Cyclodextrins	Wilkowska et al., 2016
<i>Hibiscus sabdariffa</i> L. extract	Spray drying	Fruit fibers	Chiou & Langrish, 2007
Olive leaf extract	Spray drying	Sodium caseinate; Lecithin	Kosaraju et al., 2008
Yerba mate extract	Co-crystallization	Sucrose	Deladino et al., 2007
Red wine	Freeze drying	Maltodextrin DE10	Sanchez et al., 2011
<i>Rubus chamaemorus</i> extract	Freeze drying	Maltodextrin DE 5-8 & DE18.5	Laine et al., 2008

# Objective



**The exploitation of chokeberry wastes** based on:

- Ultrasound & microwave-assisted extraction of phenolic compounds from chokeberries
- Encapsulation of extract by spray drying using maltodextrin; skim milk powder and whey protein concentrate as wall material

❖ **Optimization:**

1. *Ultrasound & microwave-assisted extraction of phenolic compounds*
2. *Encapsulation by spray drying of phenolic compounds*

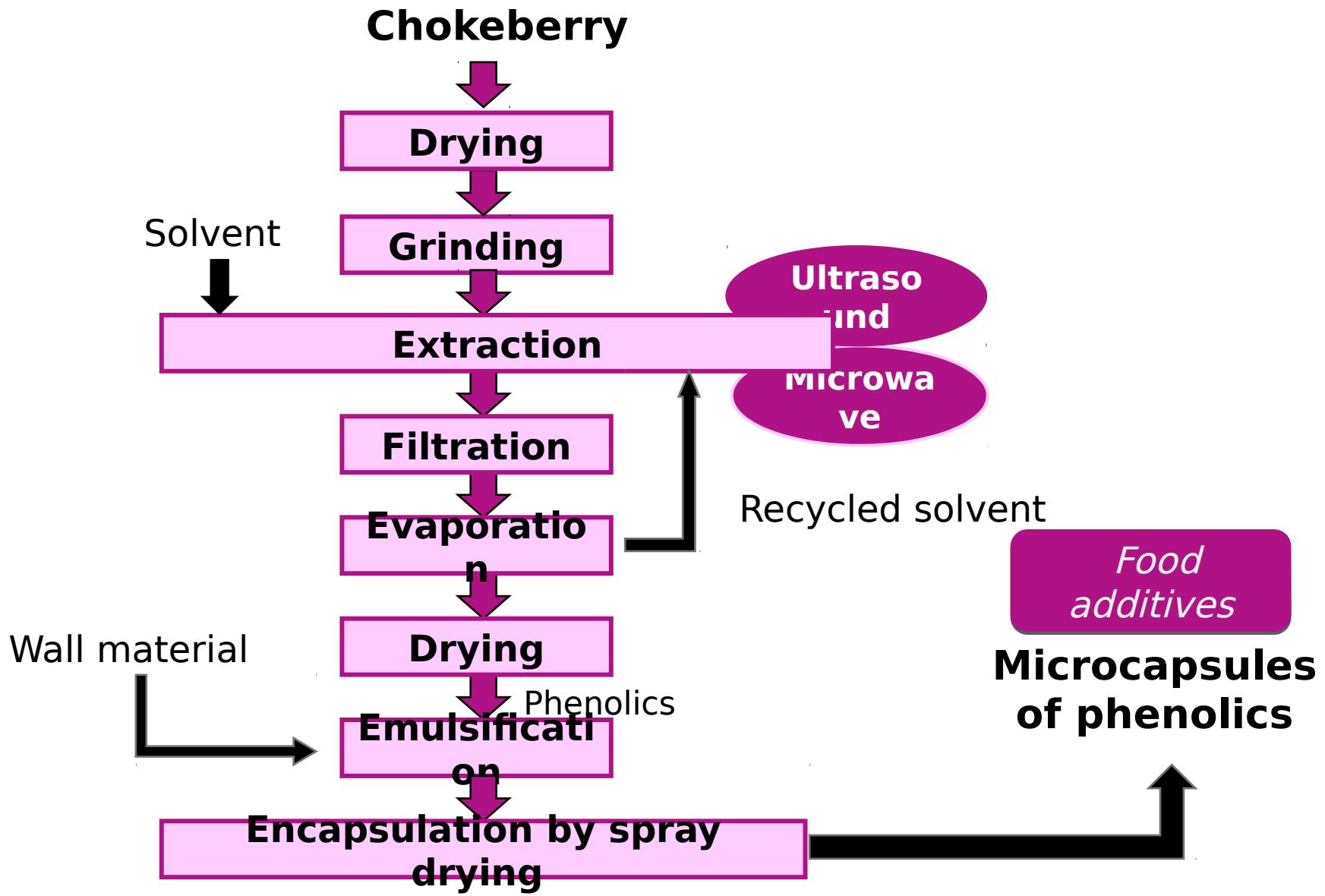
❖ **Study of:**

1. *Encapsulation efficiency*
2. *Physical properties of microcapsules (moisture content, bulk density, rehydration ability and solubility)*



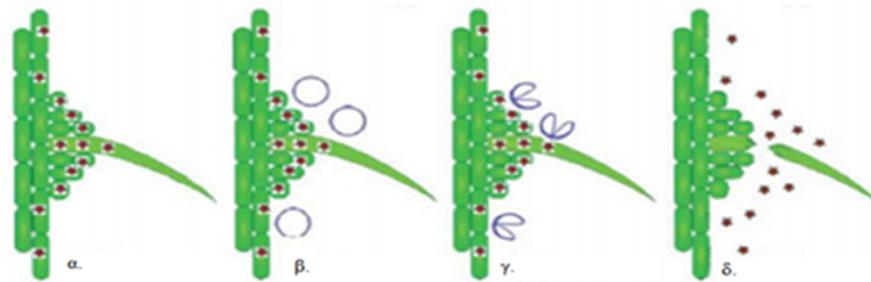
# Materials & Methods

# Proposed process for chokeberry Application in food industry



# Factors affecting the Ultrasound-assisted extraction

1. Extraction temperature
2. Solvent type
3. Liquid/Solid ratio
4. Amplitude level
5. Pulse duration/Pulse interval ratio
6. Extraction time



130 W, 20 kHz VCX-130 Sonics and Materials (Danbury, CT, USA)  $\mu\text{e}$  Ti-Al-V probe (13 mm)

# Experimental design for optimization of Ultrasound-assisted extraction of phenolic compounds from chokeberry

- ❖ Response Surface Methodology: **31 experiments**

Parameters	Levels				
<b>Solvent type (% ethanol)</b>	0	25	50	75	100
<b>Extraction temperature (T, °C)</b>	20	30	40	50	60
<b>Amplitude level (A, %)</b>	20	30	40	50	60
<b>Liquid/solid (mL/g)</b>	8	12	16	20	24

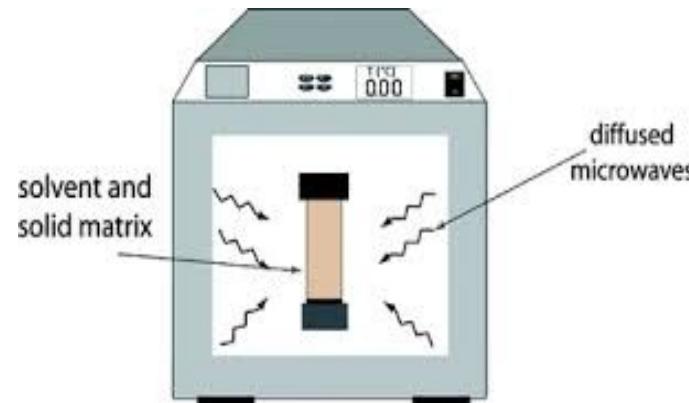
- ❖ Each experiment in  
**2,5,10,20,30 min**

# Factors affecting the Microwave-assisted extraction

1. Power
2. Solvent type
3. Liquid/Solid ratio
4. Extraction time



Microwave system (MultiwaveB30MC030A) (Anton Paar, Austria)



# Experimental design for optimization of Microwave-assisted extraction of phenolic compounds from chokeberry

- ❖ Response Surface Methodology: **20 experiments**

Parameters	Levels				
<b>Solvent type (% ethanol)</b>	0	25	50	75	100
<b>Power (W)</b>	100	200	350	500	600
<b>Liquid/solid (mL/g)</b>	8	12	16	20	24

- ❖ Each experiment in **1,2,3,4,5,6 min**

# Factors affecting the spray drying encapsulation process

1. Inlet air temperature
2. Feed solids concentration
3. Ratio of core to wall material
4. Drying air flow rate
5. Drying air humidity



Buchi, B-191,  
Buchi Laboratoriums-  
Technik,  
Flawil, Switzerland

# Experimental design for optimization of spray drying encapsulation of phenolic compounds from chokeberry

❖ Response Surface Methodology: **20 experiments x 2 wall materials**

Parameters	Levels				
<b>Ratio of wall to core material (w/c)</b>	2.3	3.7	5.6	7.3	1/9
<b>Inlet air temperature (<math>T_i</math>, °C)</b>	150	158	170	182	190
<b>Drying air flow rate (<math>Q_a</math> %)</b>	50	53	57.5	62	65

## Wall material:

- ❖ Maltodextrin/SMP: 50/50
- ❖ Maltodextrin/WPC: 50/50

- *SMP: Skimm milk powder*
- *WPC: Whey protein concentrate*

# Yield and Efficiency of microencapsulation

## ❖ Microencapsulation efficiency (E)

$$E_{ef} = \left( 1 - \frac{\text{Phenolics on microcapsule surface}}{\text{Total phenolics of microcapsule}} \right) * 100$$

## ❖ Microencapsulation yield (Y)

$$Y = \frac{\text{Mass of microcapsules (g)}}{\text{Total mass of initial substances (g)}} * 100$$

Solids feed  
collected in product  
container

# Physical properties of microcapsules



1

Moisture content

2

Bulk density

3

Rehydration ability

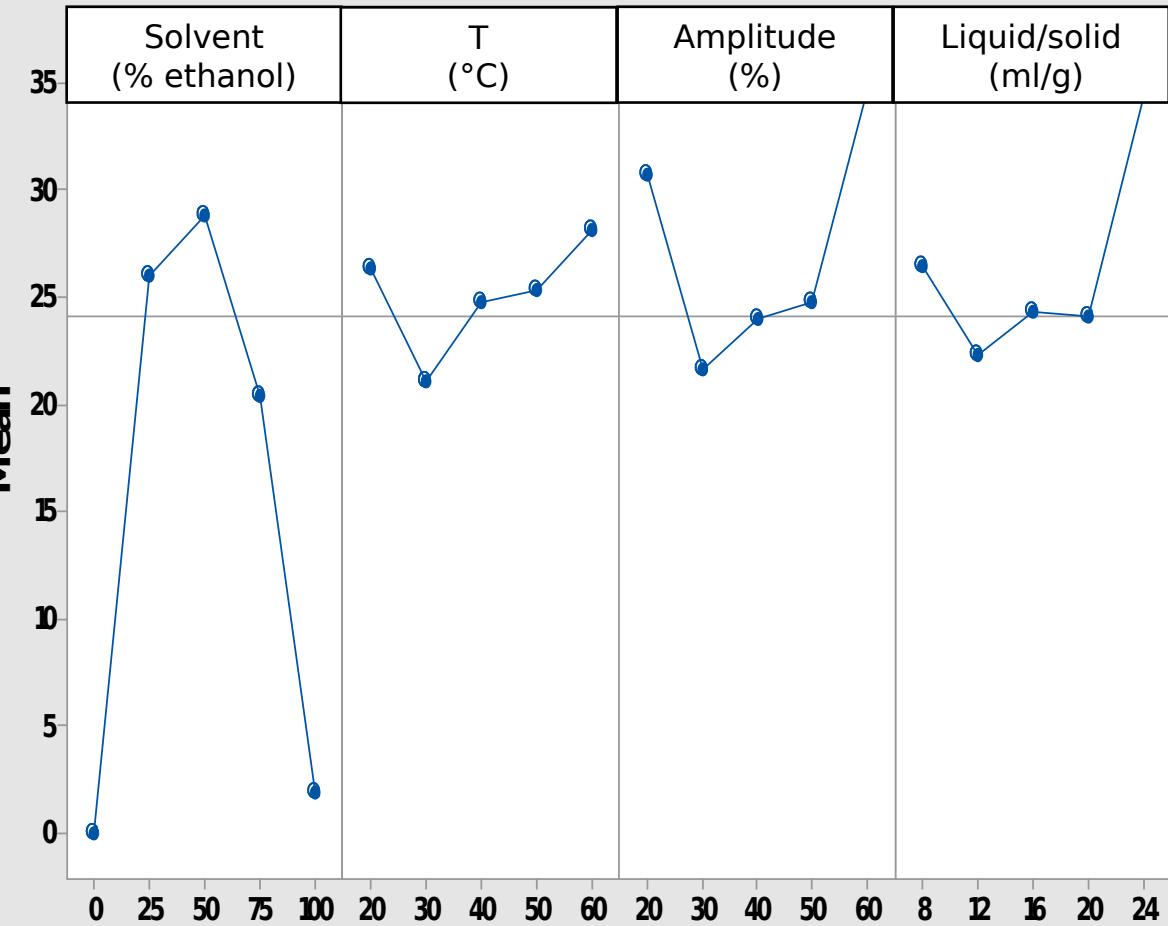
4

Solubility

# Results

# Ultrasound-assisted Extraction Yield-Effects of various parameters

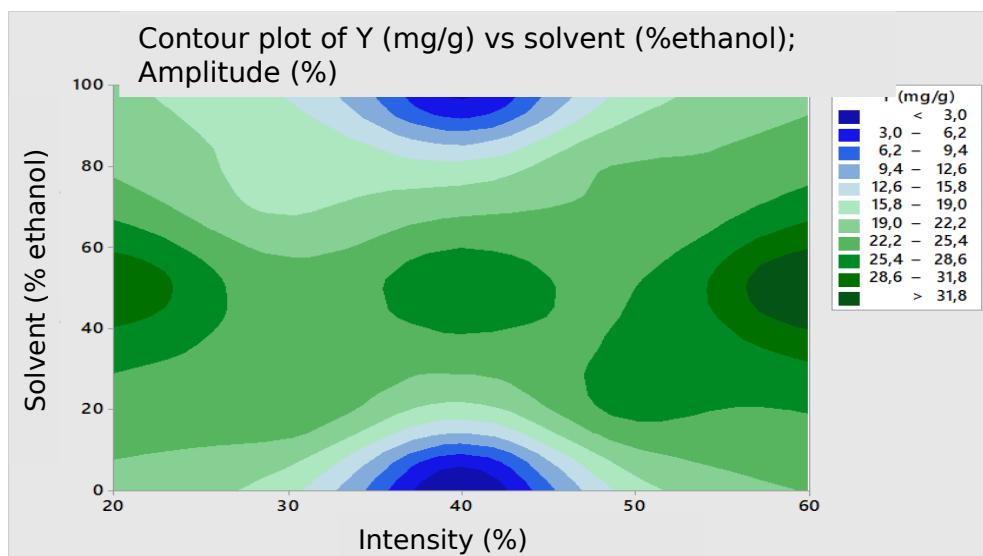
**Main Effects Plot for Y (mg/g)**  
Data Means



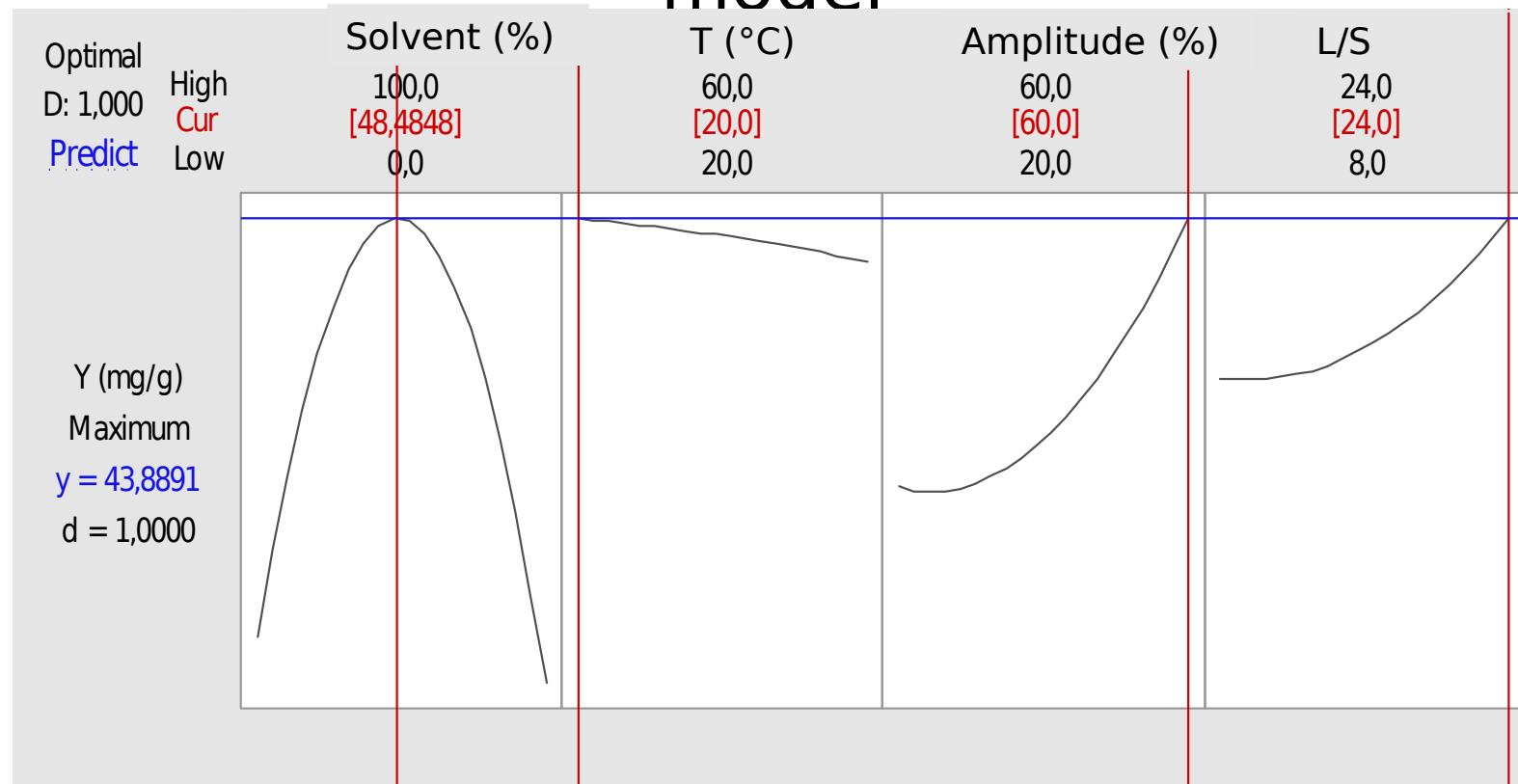
Contour plot of Y (mg/g) vs T (°C); Intensity (%); Liquid/solid (ml/g)



Contour plot of Y (mg/g) vs solvent (%ethanol); Amplitude (%)



# Extraction Yield-Optimization-Empirical model

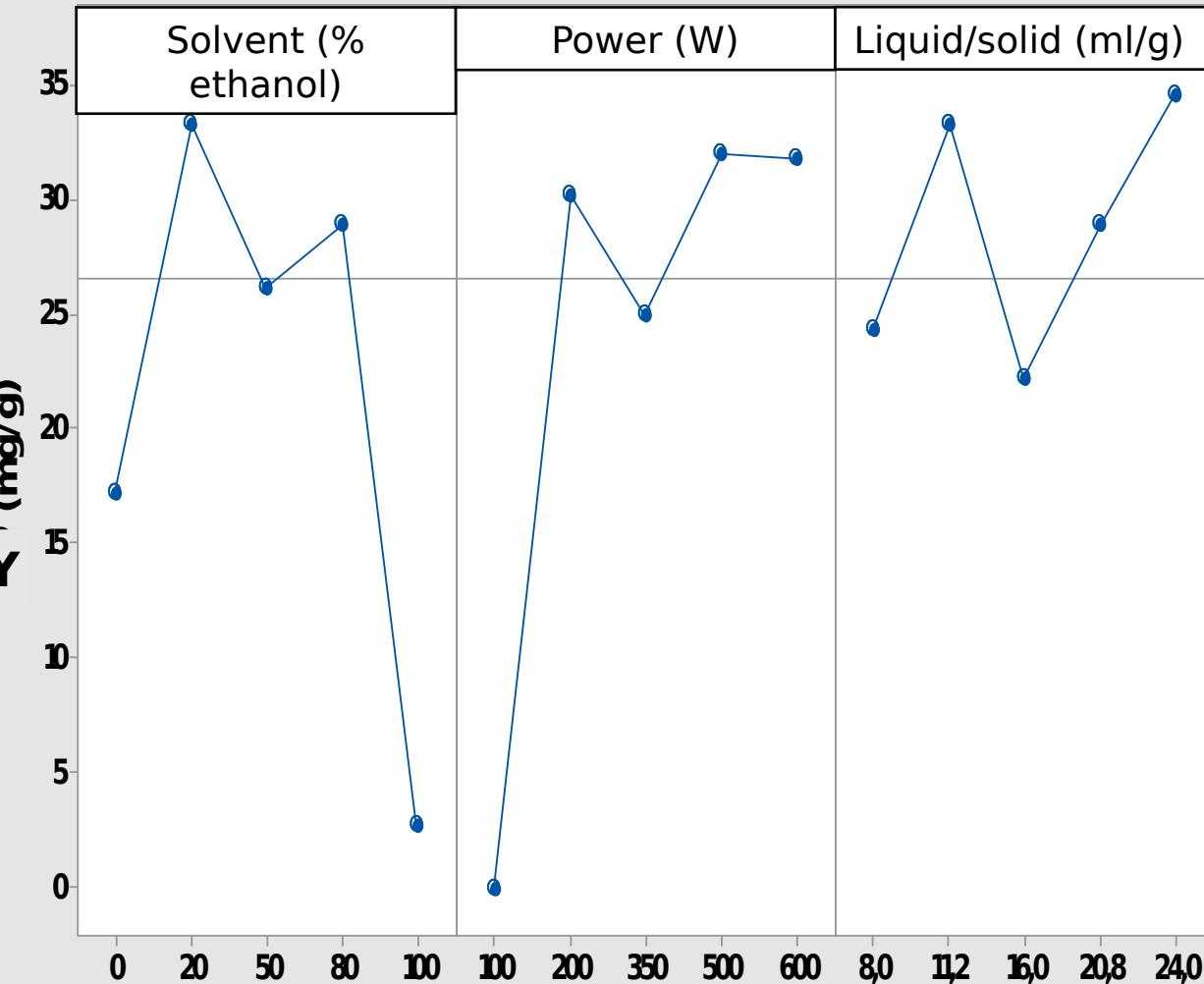


## *Empirical model of extraction yield:*

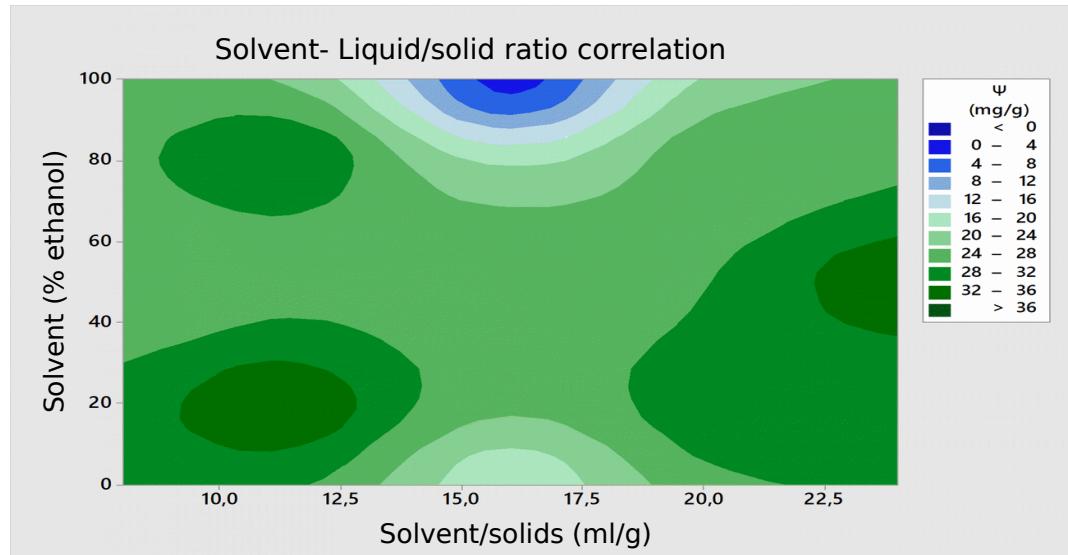
$$Y (\%) = 20.9 + 0.748 * S + 0.543 * T - 0.841 * A - 1.58 * s/s - 0.0106 * L^2 - 0.00061 * T^2 + 0.0304 * A^2 + 0.0469 * (L/S)^2 + 0.00204 * L * T + 0.00294 * S * A + 0.00282 * L * L/S - 0.0946 * T * A - 0.038 * T * L/S + 0.0104 * A * L/S$$

# Microwave-assisted Extraction Yield-Effects of various parameters

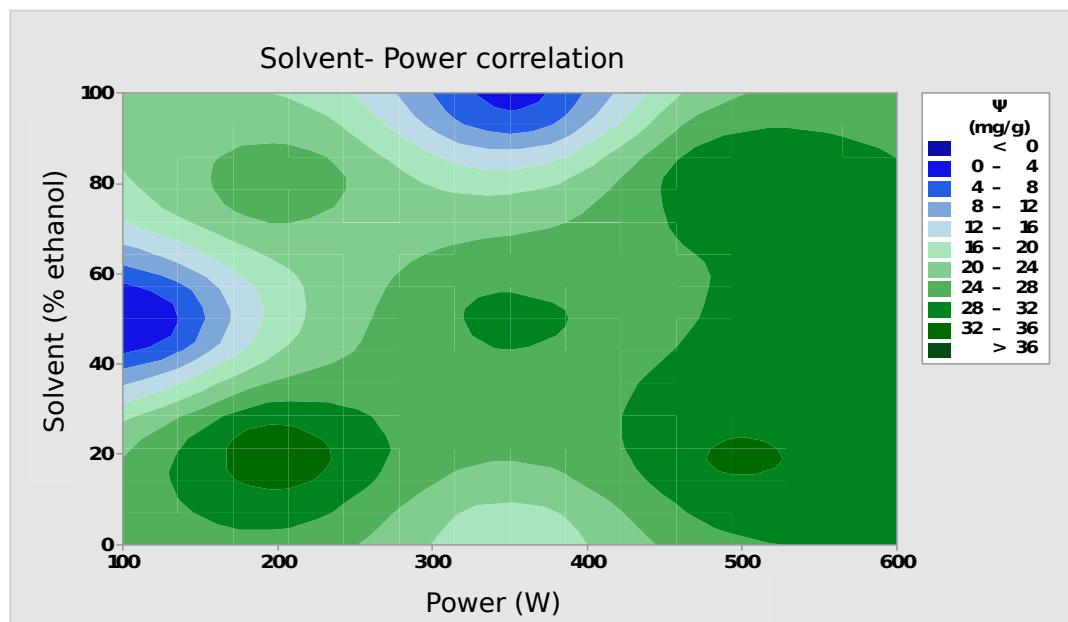
Main Effects Plot for  $\Psi$ (mg/g)



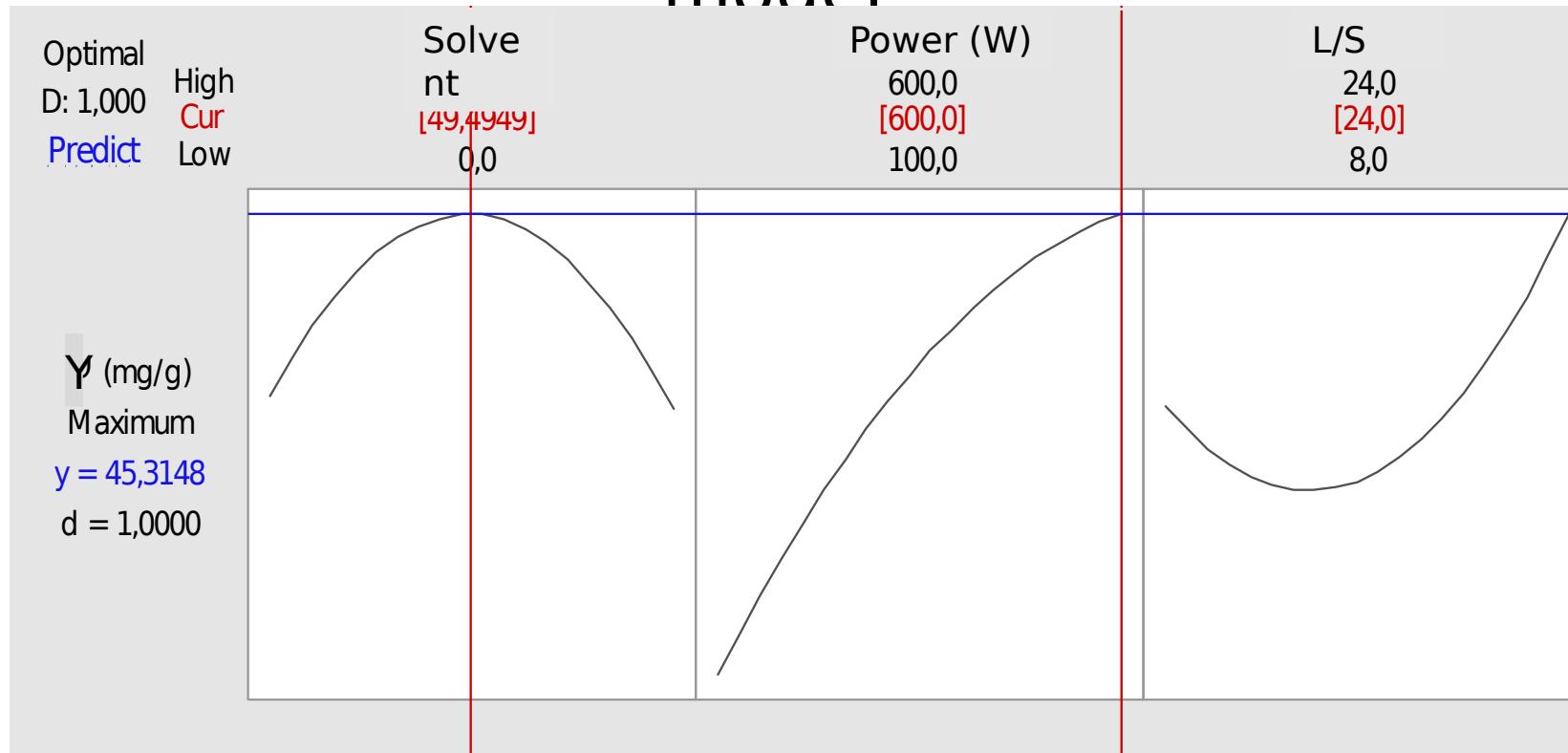
Solvent- Liquid/solid ratio correlation



Solvent- Power correlation



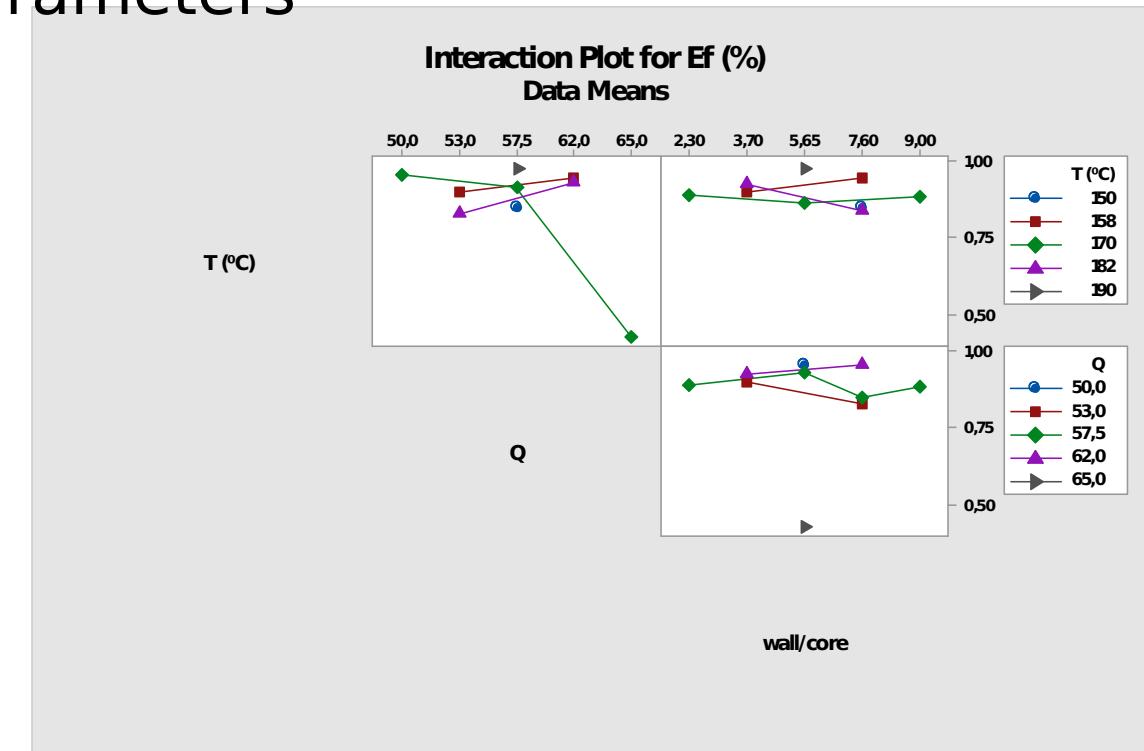
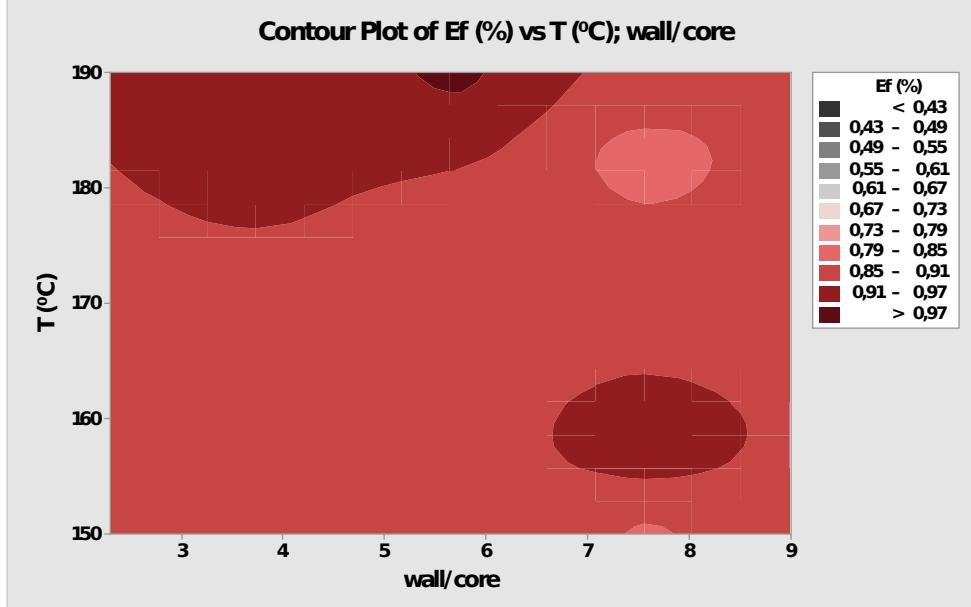
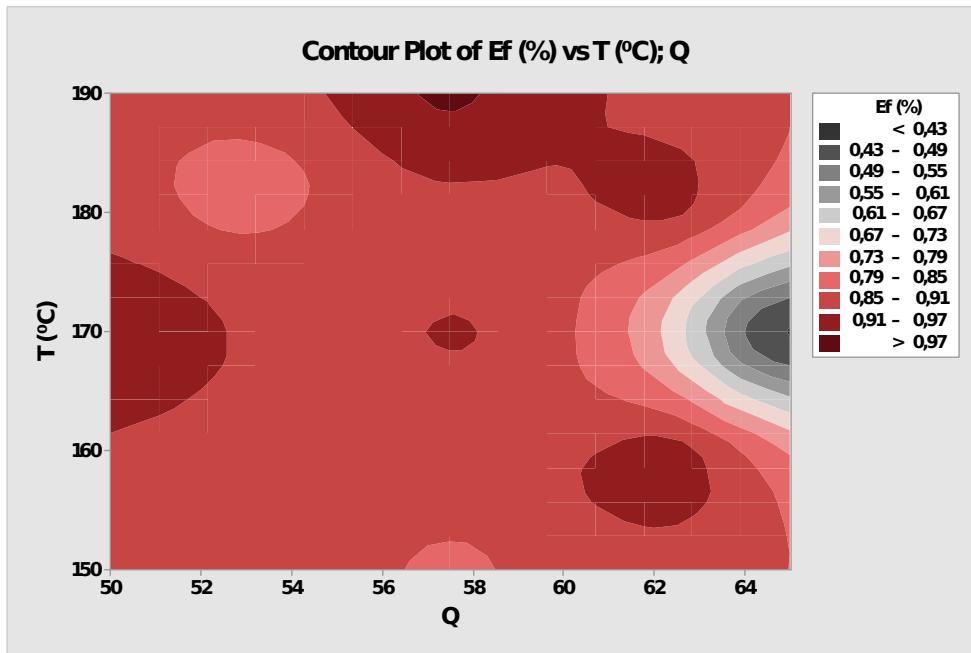
# Extraction Yield-Optimization-Empirical model



## *Empirical model of extraction yield:*

$$\begin{aligned} Y (\%) = & 28.03 - 3.09 * S + 4.46 * P + 0.01 * L/S - 3.73 * S^2 - 1.64 * P^2 + 3.17 * (L/S)^2 \\ & + 1.83 * S * P - 0.11 * S * (L/S) + 1.90 * P * (L/S) \end{aligned}$$

# Encapsulation efficiency-Effects of various parameters

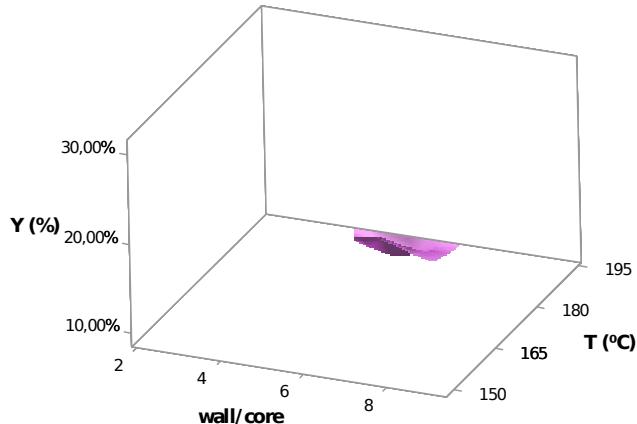


❖ **Microencapsulation efficiency (E)**

$$E_f = \frac{(1 - \frac{\text{Phenolics on microcapsule surface}}{\text{Total phenolics of microcapsule}})} * 100$$

# Encapsulation Yield-Effects of various parameters

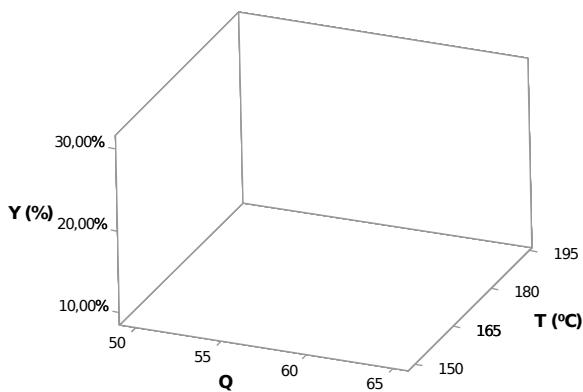
Surface Plot of Y (%) vs T (°C); wall/core



Coded Coefficients

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		0,2174	0,0138	15,71	0,000	
T (°C)		0,0134	0,0067	0,42	0,686	1,09
Q		0,0179	0,0089	0,58	0,574	1,00
wall/core		0,0213	0,0107	0,155	0,69	0,506
T (°C) *T (°C)		0,1774	0,0887	0,264	3,36	0,007
Q*Q		-0,0420	-0,0210	0,0254	-0,83	0,428
wall/core*wall/core		-0,1041	-0,0520	0,0261	-1,99	0,074
T (°C) *Q		-0,0389	-0,0195	0,0334	-0,58	0,573
T (°C) *wall/core		0,1079	0,0540	0,0322	1,68	0,125
Q*wall/core		0,0487	0,0243	0,0344	0,71	0,495

Surface Plot of Y (%) vs T (°C); Q



## ❖ Microencapsulation yield (Y)

$$Y = \frac{\text{Mass of micocapsules (g)}}{\text{Total mass of initial substances (g)}} * 100$$

# Conclusions

- Microwave assisted extraction is more effective than ultra sound-assisted extraction and in shorter time
- Interactions between factors are not statistically significant
- In microwave-assisted extraction no factor effects statistically significant
- In ultrasound-assisted extraction solvent and amplitude effect statistically significant
- Solvent ratio has a very significant effect on extraction

Thank you for your  
attention!