Optimization of Ultrasounds Assisted Extraction of polysaccharides from cladodes of Opuntia ficus-indica (L.) Mill using Response Surface Methodology

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Food side-streams: from waste to resource

- The increase in industrial activities in the food and agricultural sectors is causing an increase in waste production
- Around **1.6 billion tons/year** of food side-streams involving by-products need to be managed
- This data is expected to **increase by 33 %** within the next decade

Most of these residues demonstrated to still contain valuable compounds with a variety of different properties

Food waste can be an alternative source of income high-value compounds

Residues from agricultural production

Residues from food processing

Storage and classification

Drying

and/or

Pretreatments

Extraction of molecules

Post-treatment / formulation

NEW INGREDIENTS

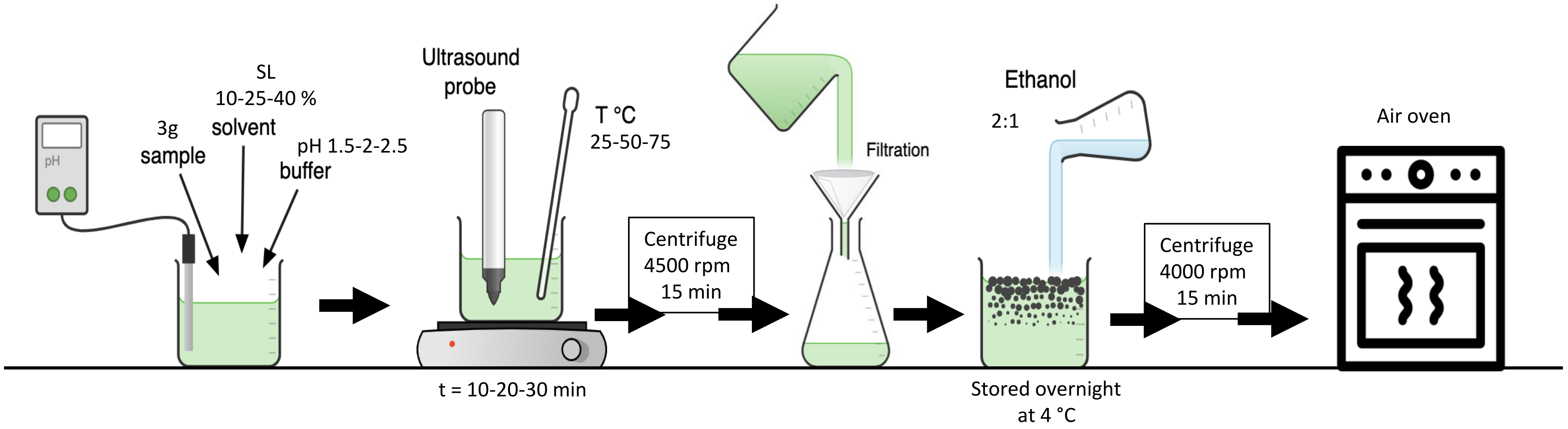
Opuntia ficus-indica (OFI)

- OFI is important in agricultural economies throughout arid and semiarid parts of the world.
- In Italy there are **7000–8300 ha** of intensive plantations.
- Specialized agro-industries produce **6–8 tons/ha** pruning wastes consisting mostly of cladodes and immature fruits.
- Recent studies have demonstrated the **nutraceutical, cosmetic and pharmaceutical potential** of cladodes.

	% dw	Method
moisture	5.88 ± 0.56	AOAC Official Method 925.09
ash	21.99 ± 0.06	Gravimetric method
lipid	1.68 ± 0.34	AOAC Official Method 920.39
protein	7.06 ± 0.29	Dumas method
TDF	40.96 ± 0.04	Enzymatic + Gravimetric method
Carbohydrates	22.43 ± 0.86	Difference

Cladodes valorisation: polysaccharides extraction

ULTRASOUND ASSISTED EXTRACTION



Cladodes valorisation: polysaccharides extraction

DESIGN OF EXPERIMENTS (DOE)

Face centered central composite response surface design (FCCRD)

Tested parameters:

SL X1	pH X2	t X3	T X4	Coded
10	1.5	10	25	-1
25	2	20	50	0
40	2.5	30	75	1

4 variables in 3 levels:
24 configurations + 3 repetitions

27 EXPERIMENTS

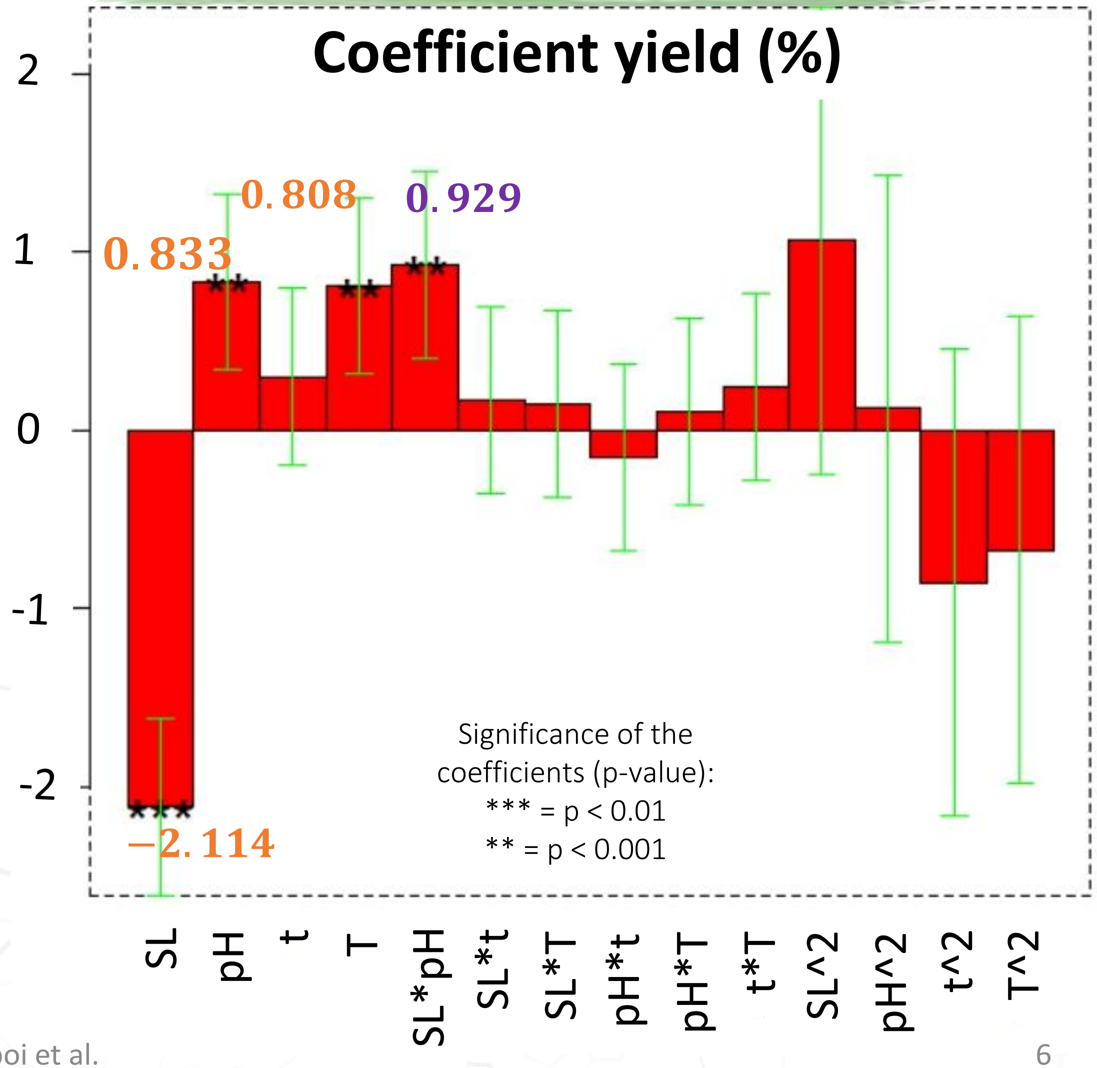
$$Y = \underbrace{\beta_0}_{\text{constant}} + \underbrace{\sum_{i=1}^{k} \beta_i X_i}_{\text{main effects}} + \underbrace{\sum_{i=1}^{k} \beta_{ii} X_i^2}_{\text{curvature}} + \underbrace{\sum_{i=1}^{k-1} \sum_{j=2(i\neq j)}^{k} \beta_{ij} X_i X_j}_{\text{interactions}}$$

Cladodes valorisation: model

$$\boldsymbol{Y}(\%) = 8.658 +$$
$$+ (-2.114 * X1) + (0.833 * X2)$$
$$+ (0.300 * X3) + (0.808 * X4) +$$
$$+(1.060 * X1^2) + (0.120 * X2^2)$$
$$+ (-0.854 * X3^2) + (-0.672 * X4^2) +$$
$$+(0.929 * X1X2) + (0.167 * X1X3)$$
$$+ (0.142 * X1X4) + (-0.149 * X2X3)$$
$$+ (0.104 X2X4) + (0.239 * X3X4)$$

(X1 = SL   X2 = pH   X3 = t   X4 = T)

EXPLAINED VARIANCE = 82.63 %



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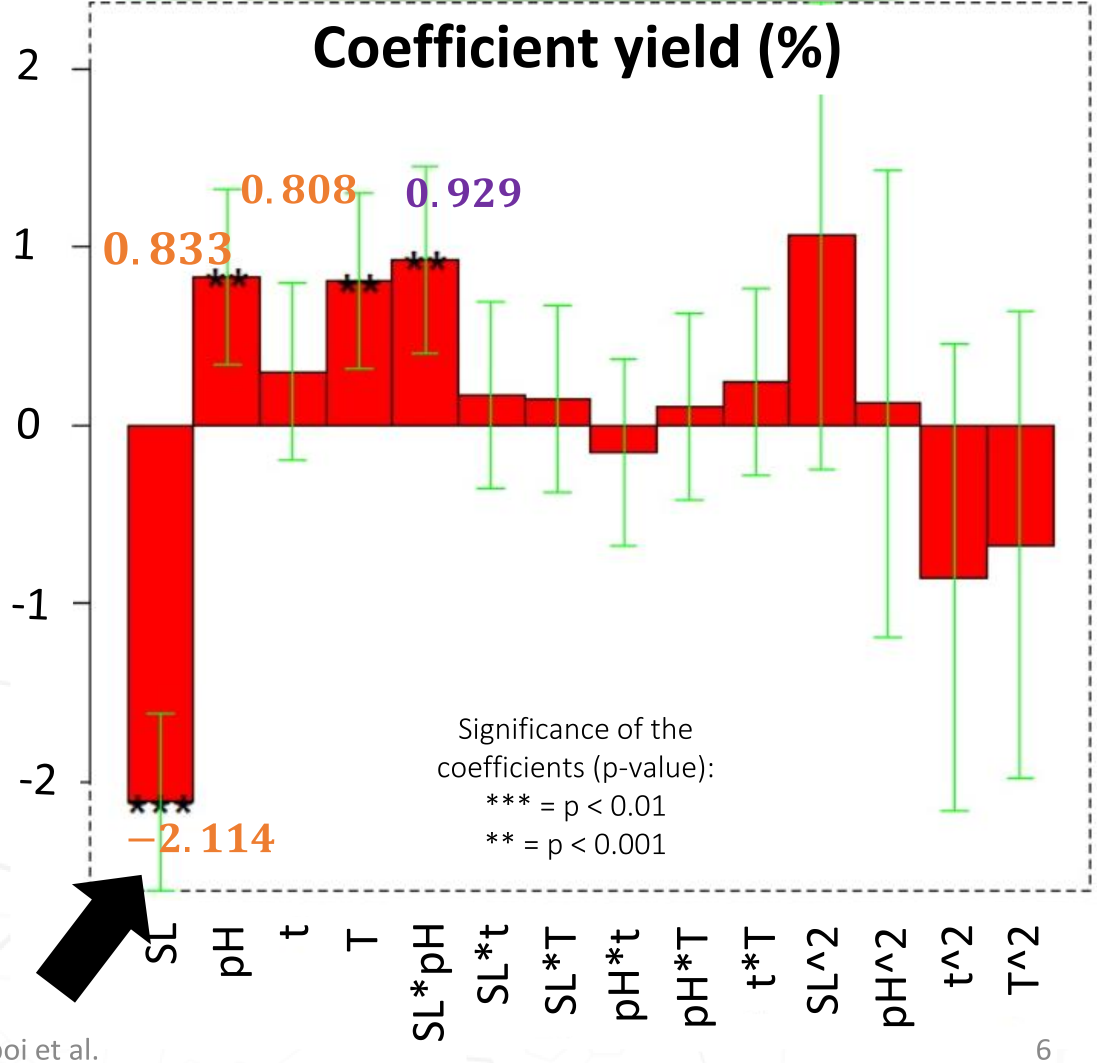

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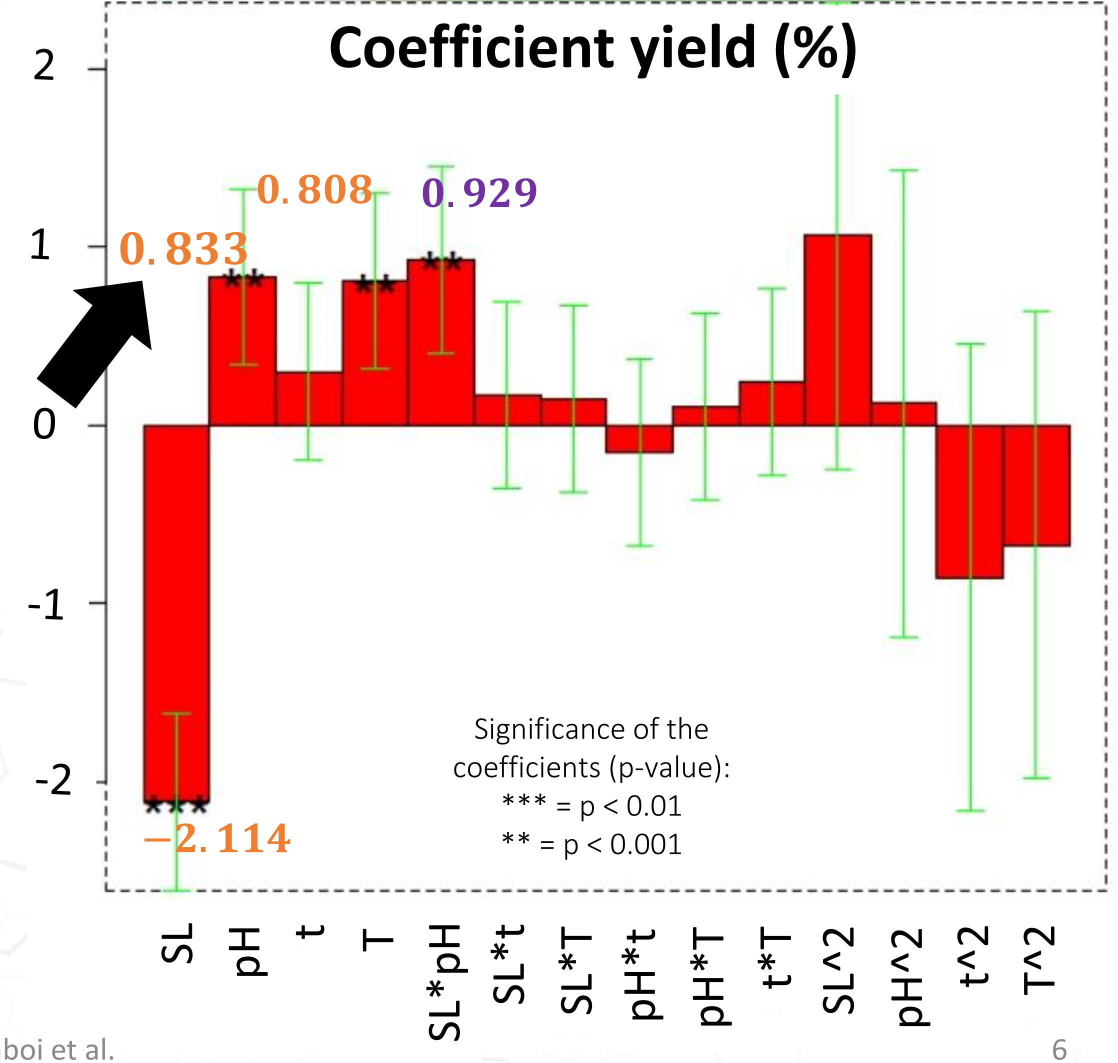

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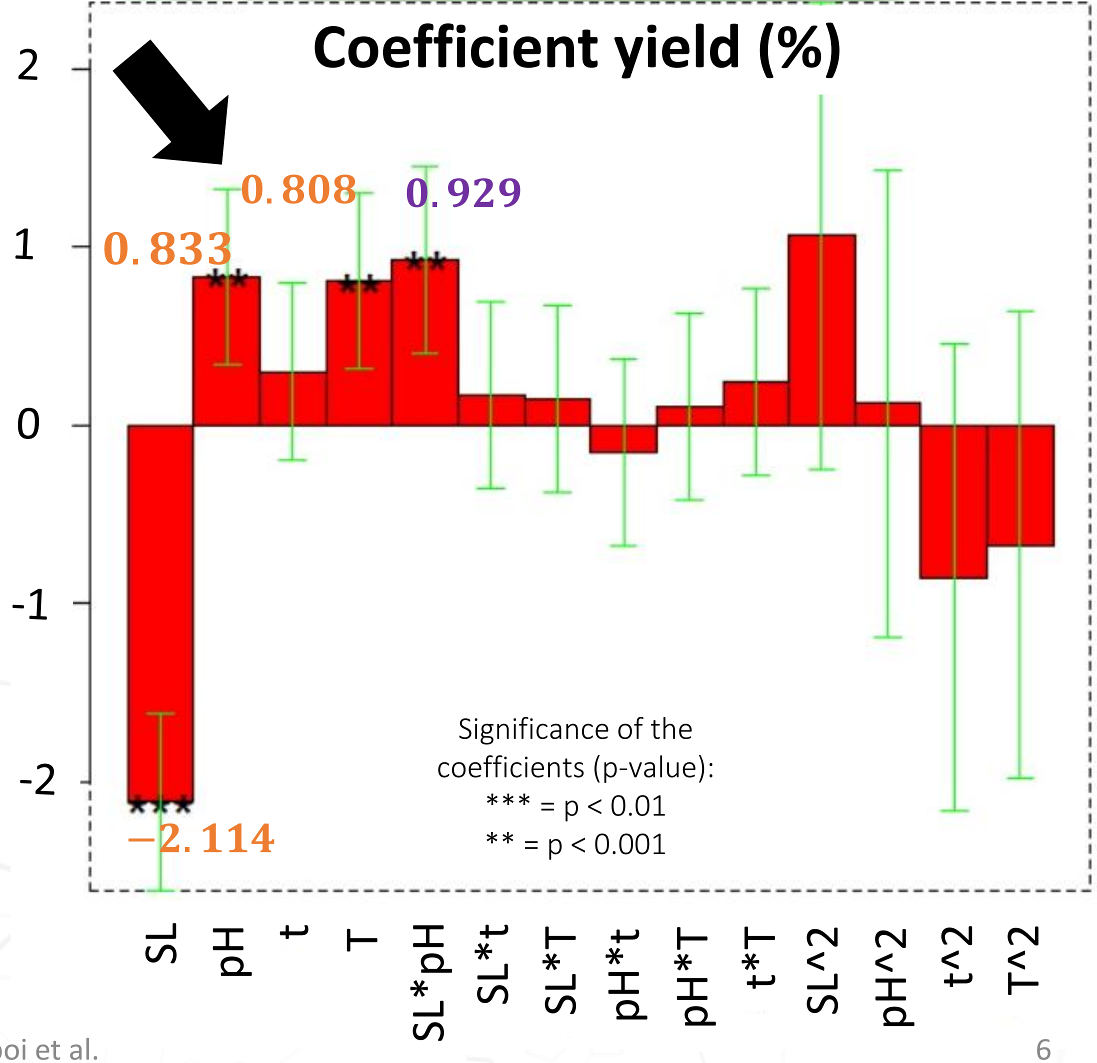

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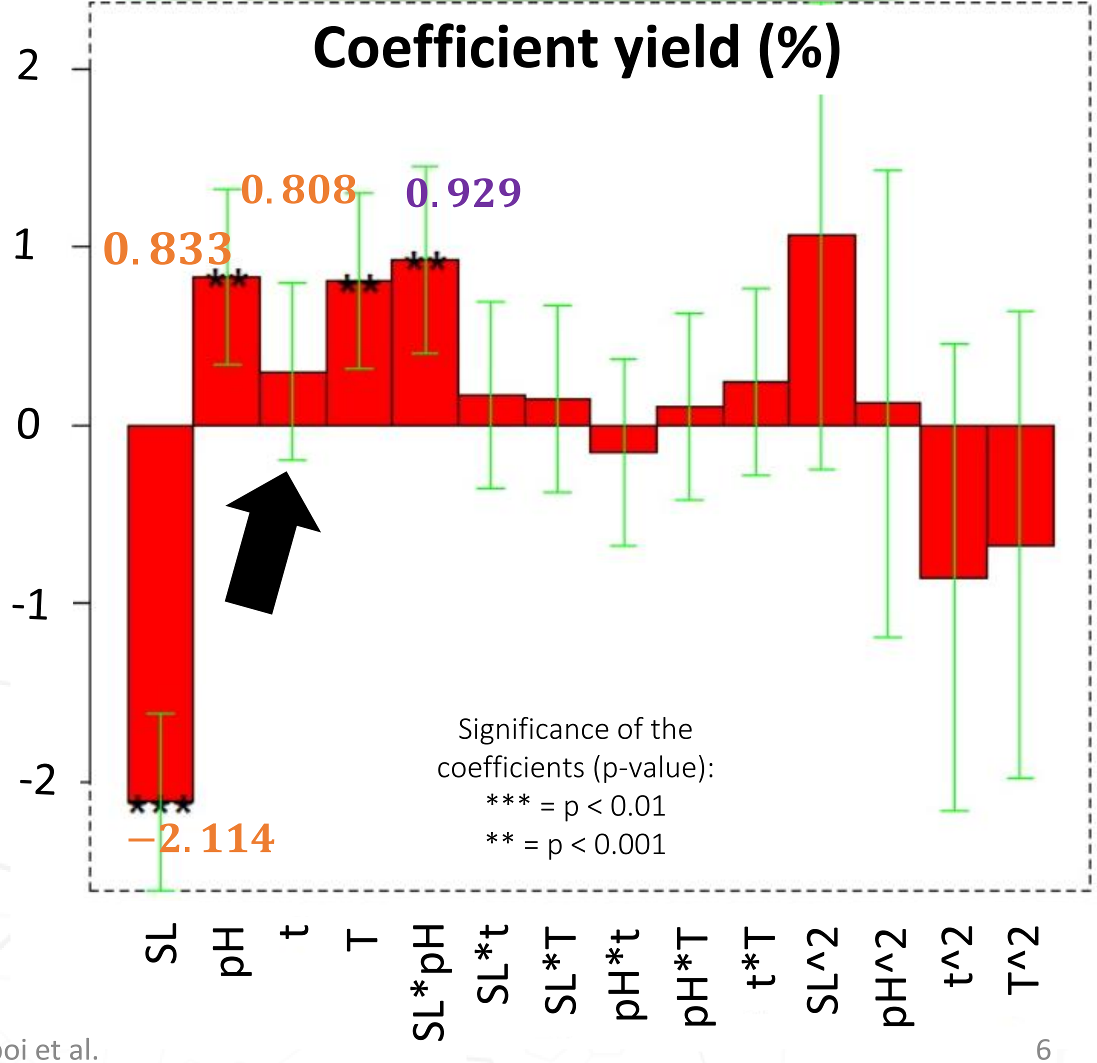

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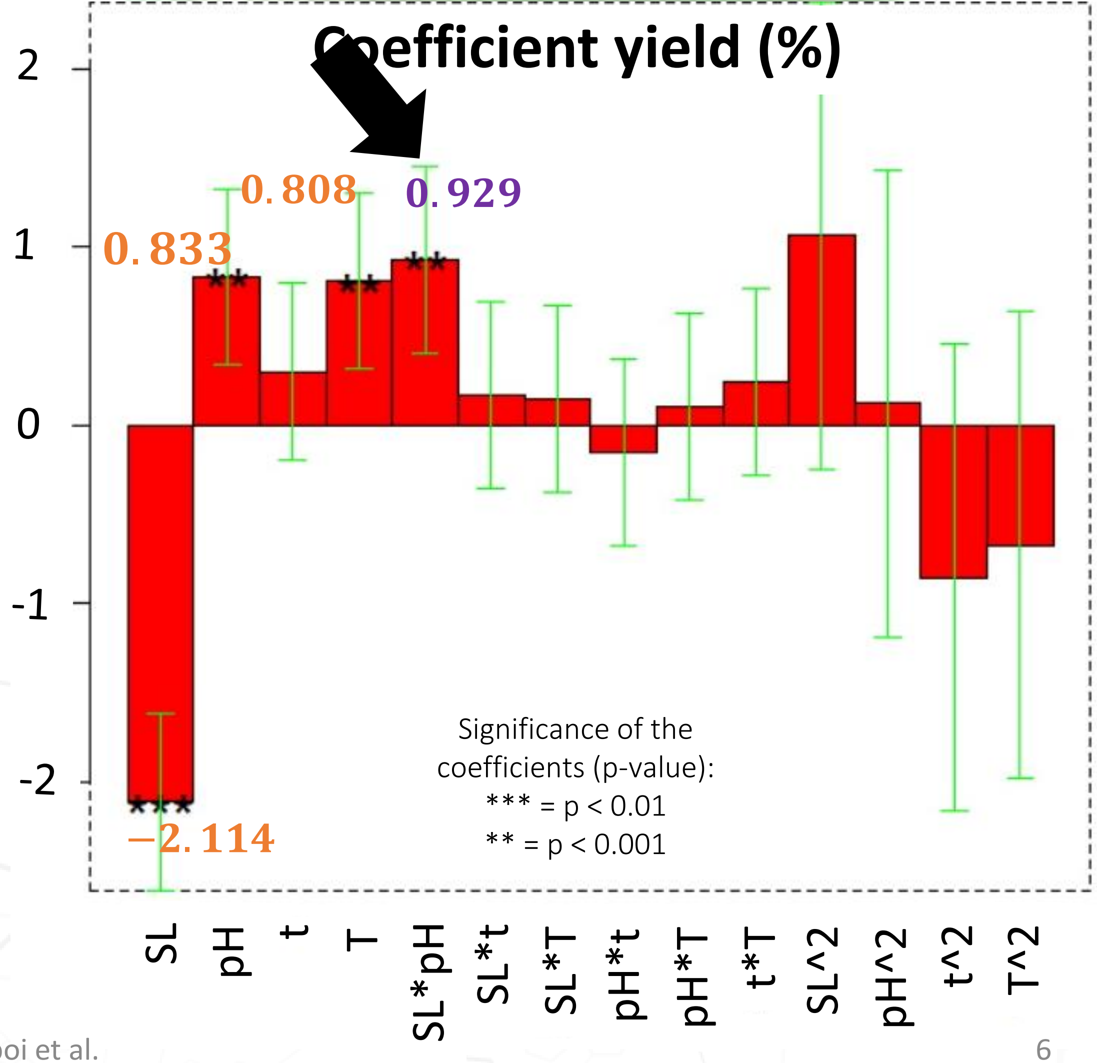
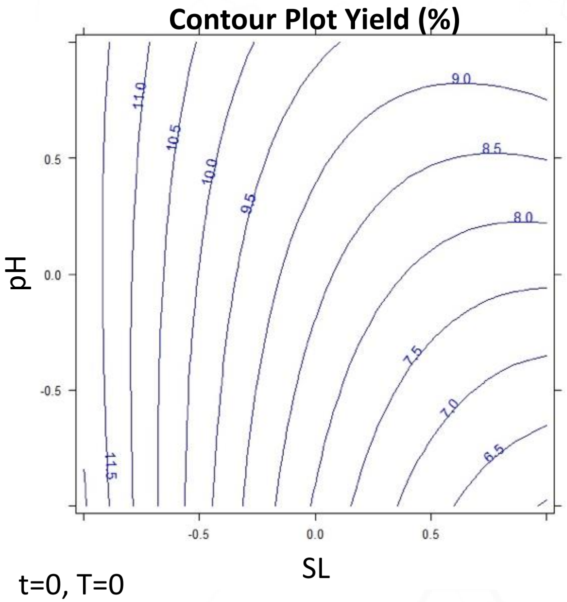
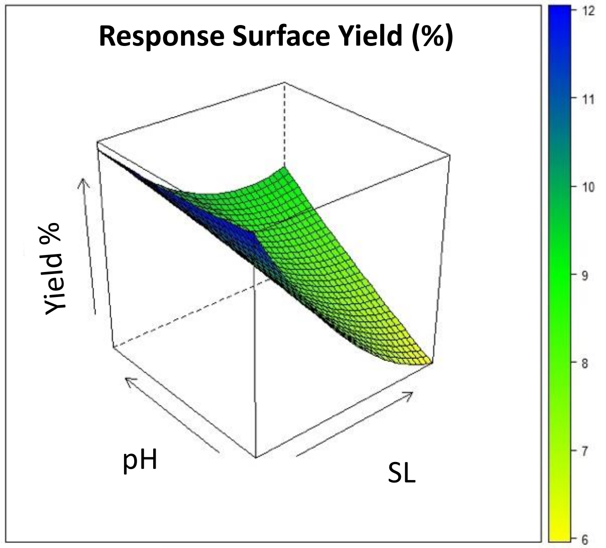


Cladodes valorisation: Response Surfaces



t=0, T=0



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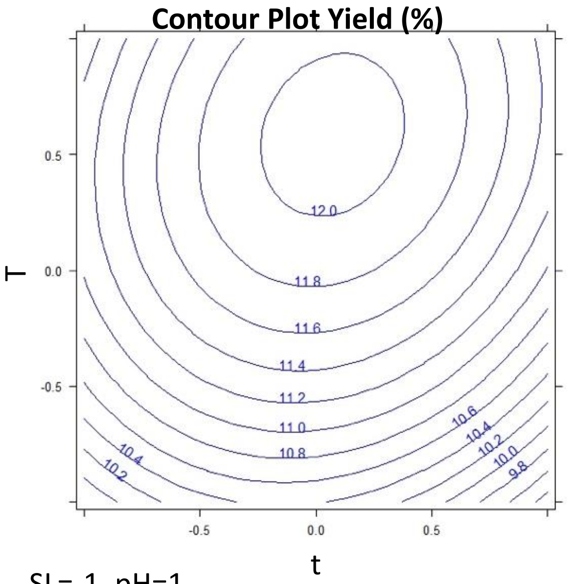
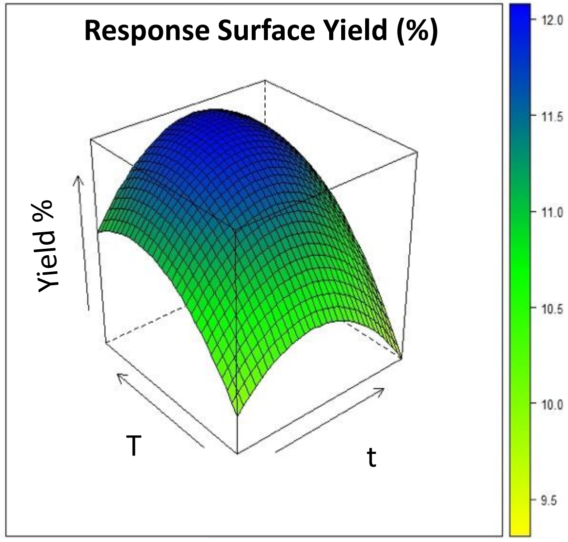


SL=-1, pH=1



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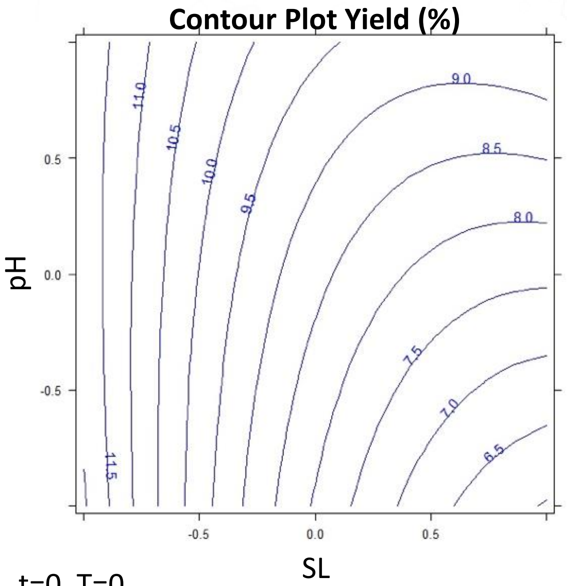

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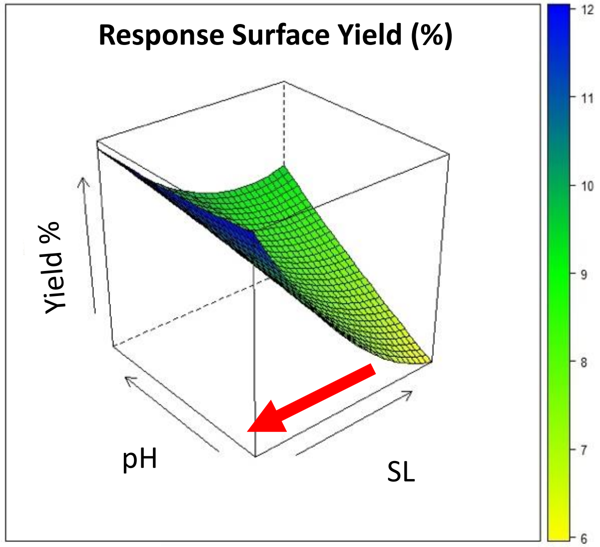

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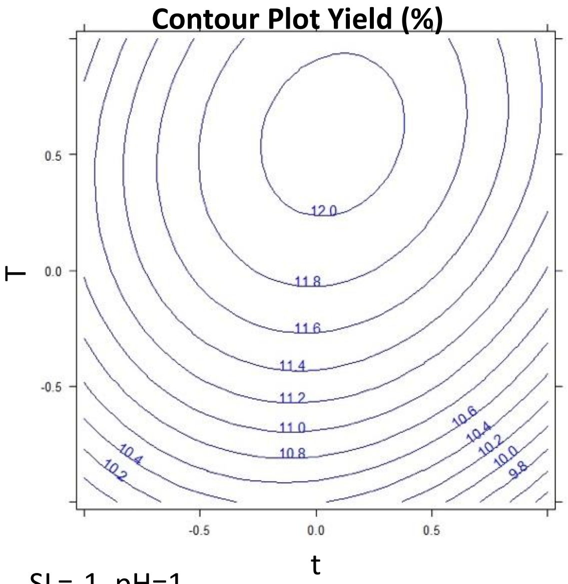

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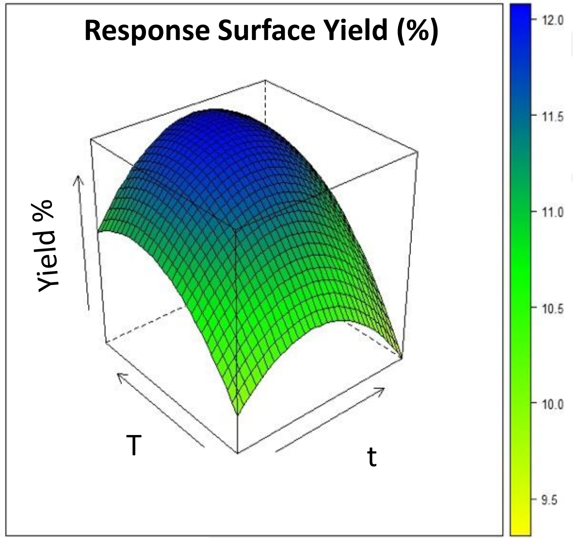

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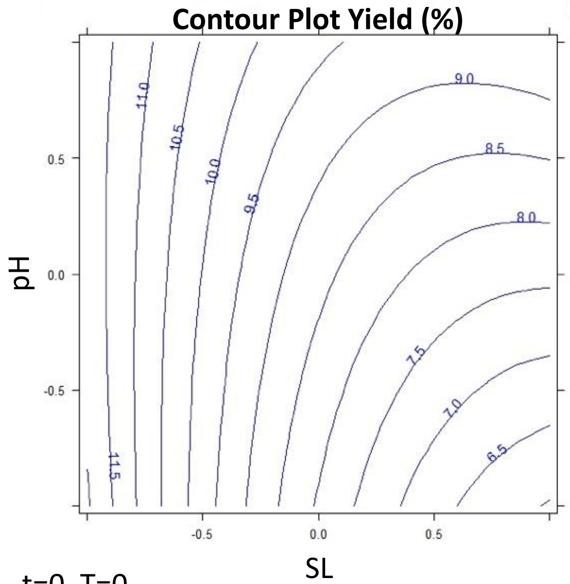

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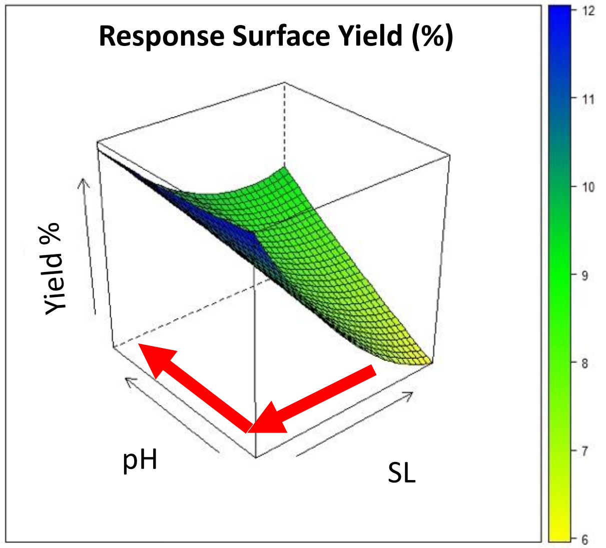

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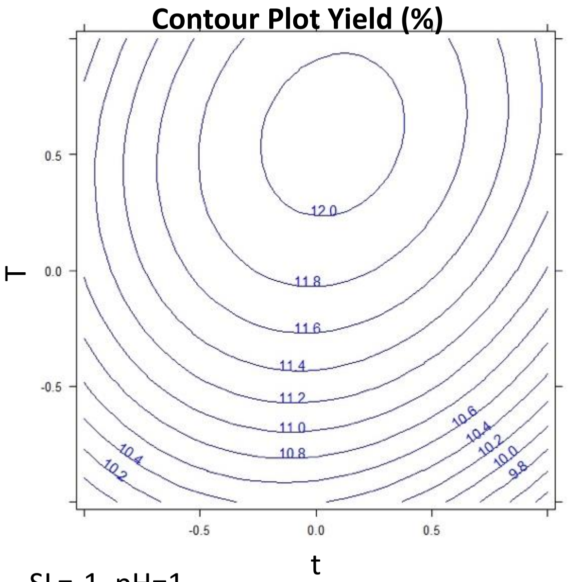

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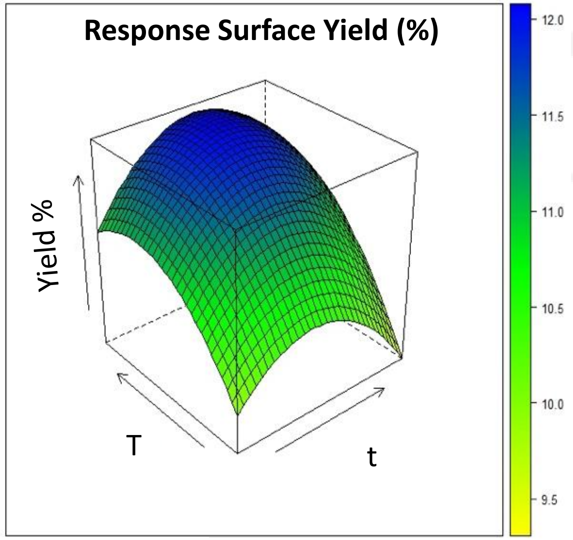

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Better operational conditions:

- SL = **10 %** (-1)
- pH = **2.5** (1)

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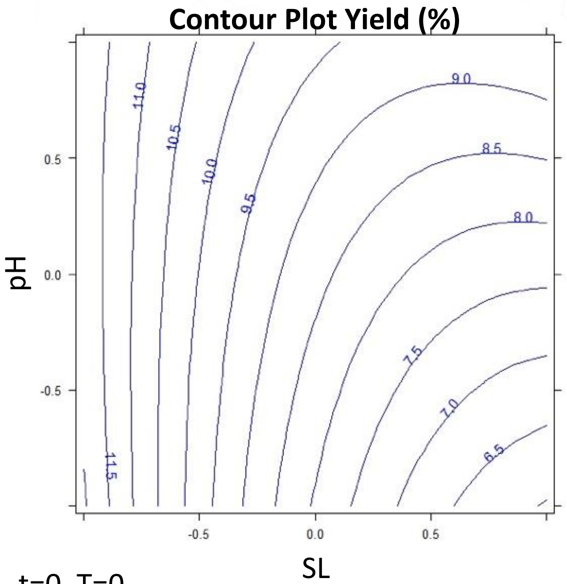

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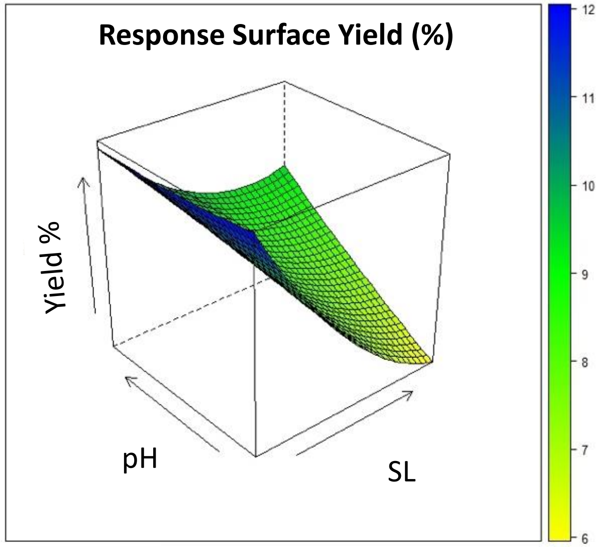

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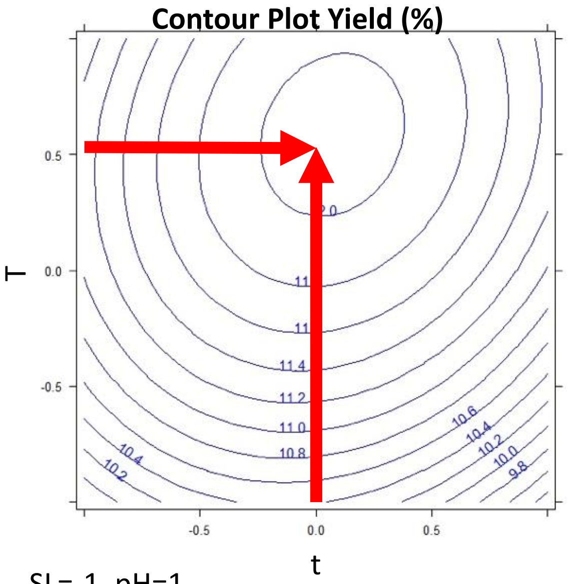

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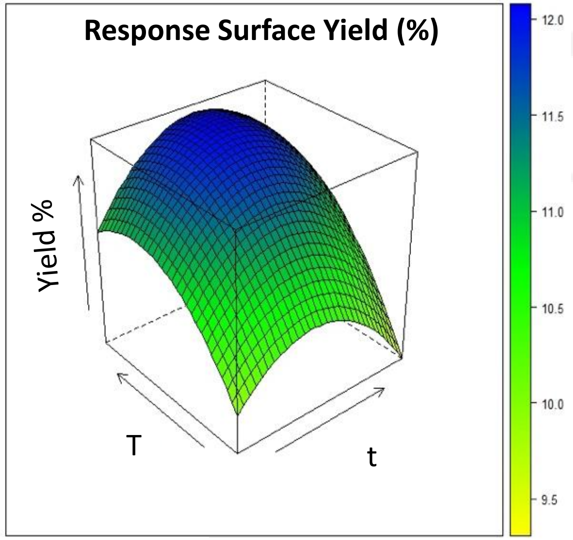

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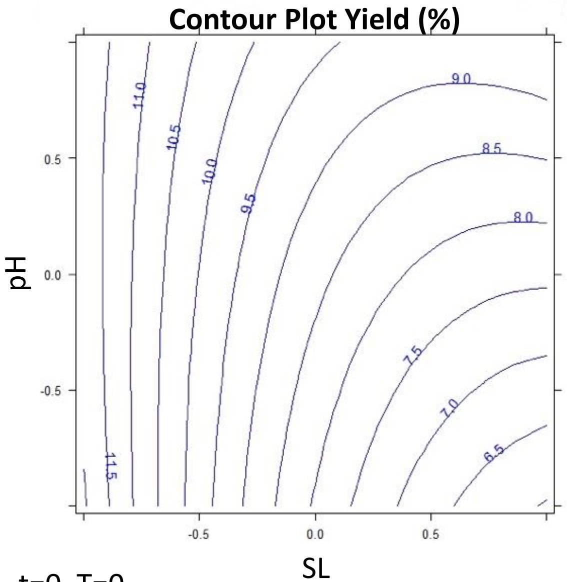
Better operational conditions:
- SL = **10 %** (-1)
- pH = **2.5** (1)
- T = **65°C** (0.5)
- t = **20 min** (0)

Cladodes valorisation: Response Surfaces



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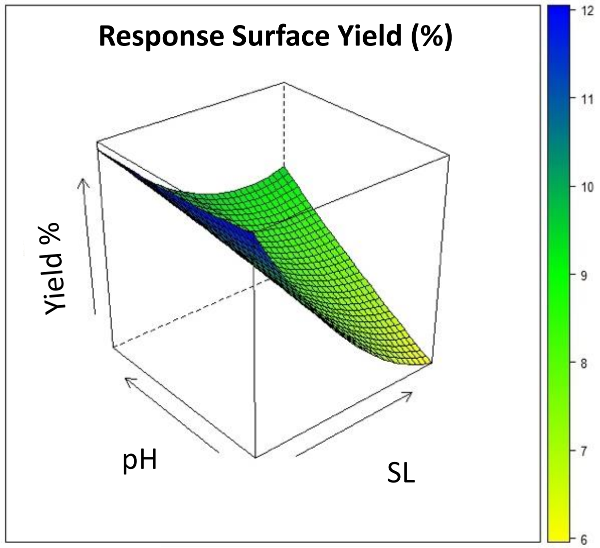

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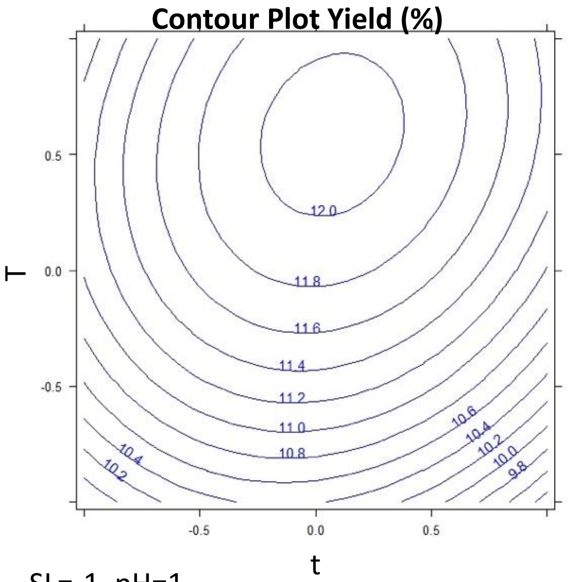

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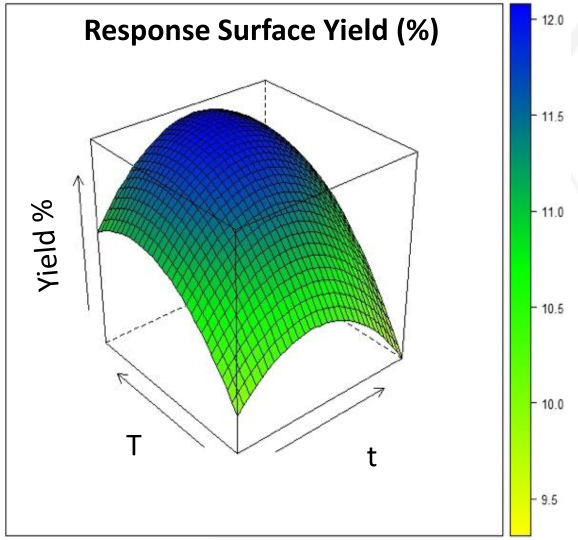

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EXTRACTION YIELD
Predicted = 12 %
Measured = 11.32 ± 0.25 %

Extract characterization: Monosaccharide composition

Method: HPLC-RID analyses.

Tested monosaccharides:

- Galacturonic acid – GalA (25.55 ± 0.30 %)
- Galactose – Gal (13.5 ± 0.22 %)
- Glucose – Glu (1.12 ± 0.03 %)
- Arabinose – Ara (14.34 ± 0.01 %)

Extract characterization: Esterification Degree (ED)

Method: acid-base titration.

- ED is a parameter used to determine the degree of methylation of the extract
  - → Low Methylated (LM) has ED ≤ 50 %
  - → High Methylated (HM) has ED > 50 %

ED = 42.84 ± 0.48 %

- ED is a fundamental parameter for having information on the ability of the extract to form gels.
- The gelling mechanism of LM extract is based on the alignment of sequences of GalA monomer, linked through electrostatic and ionic bond of carboxyl groups.

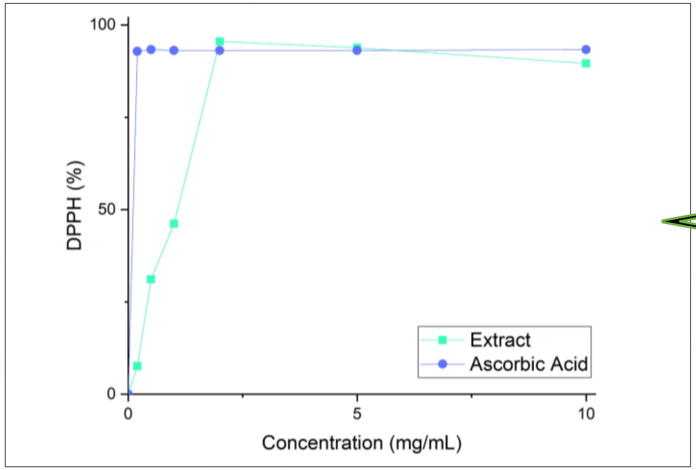
Extract characterization: phenolics

Total Phenolic Content (TPC)

Method: Folin-Ciocalteu reagent.

TPC = 41.33 ± 3.53 mgGAE/g

DPPH radical scavenging activity



the anti-radical ability increases with the concentration up to 95.56 % at a concentration of 2 mg/mL

- The use of ultrasound, as well as the extraction temperature are responsible for the release of polyphenols.
- Polyphenols are scavengers of free radicals, responsible for oxidation phenomena, and could prevent many diseases like heart diseases, cholesterol, arteriosclerosis and cancer.

Conclusions

- Cladodes of OFI are generally undervalued pruning wastes
- UAE optimized parameters (SL 10 %, pH 2.5, t 20 min, T 65 °C) allows to reduce water and chemicals consumption
- UAE allows to recover an extract that is basically a heteropolysaccharide rich in polyphenols
- The extract could be of nutraceutical and cosmetic interest

Next steps

- Further analyses are needed to identify the polyphenols present in the extract
- Further tests are needed to investigate the applicability of the extract in nutraceutical and cosmetic applications

Thanks for your attention

Aurora Zamboi

1859

chimica fisica materiali DiSAT scienza e ingegneria

CREST Catalytic Reaction Engineering for Sustainable Technologies