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Optimization of a green method for the recovery of polyphenols from onion solid wastes

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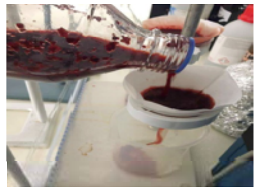


Exploitation of plant by-products by the food industry

Food production

(raw materials, final products)

Food waste or by-products

(leaves, roots, seeds)

Phytochemicals

(antioxidants, antimicrobials)

Extraction of phytochemicals with conventional and non-conventional techniques

Production of functional - novel food products

onion solid wastes

water :

89 – 93 %

Proteins :
0,9 – 1,6 %

Lipids : 0,1
– 0,4 %

Carbohydrates :

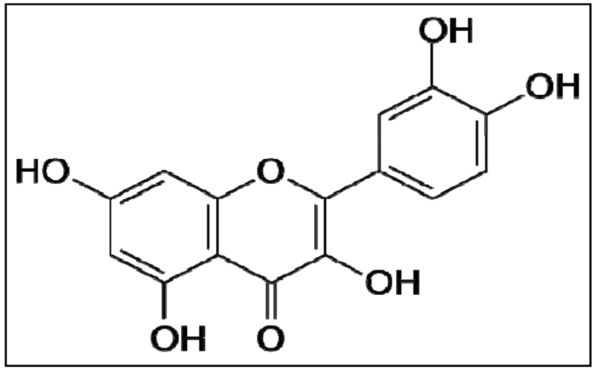
5,2 – 9,0 %

Dietay
fibers: 0,5 %

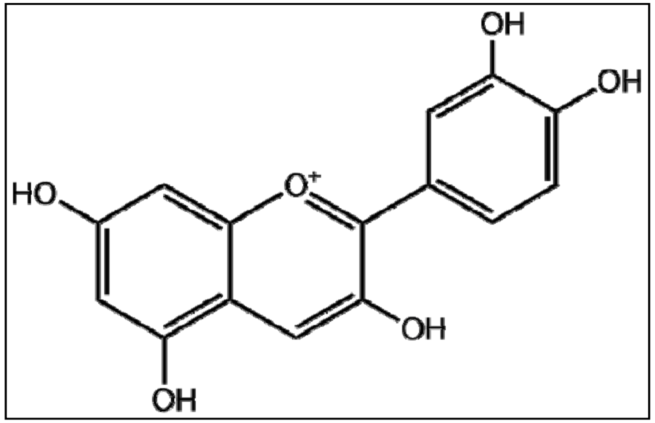
Flavonoids

450,000 tonnes/year waste in
Europe

Onion polyphenols



Quercetin

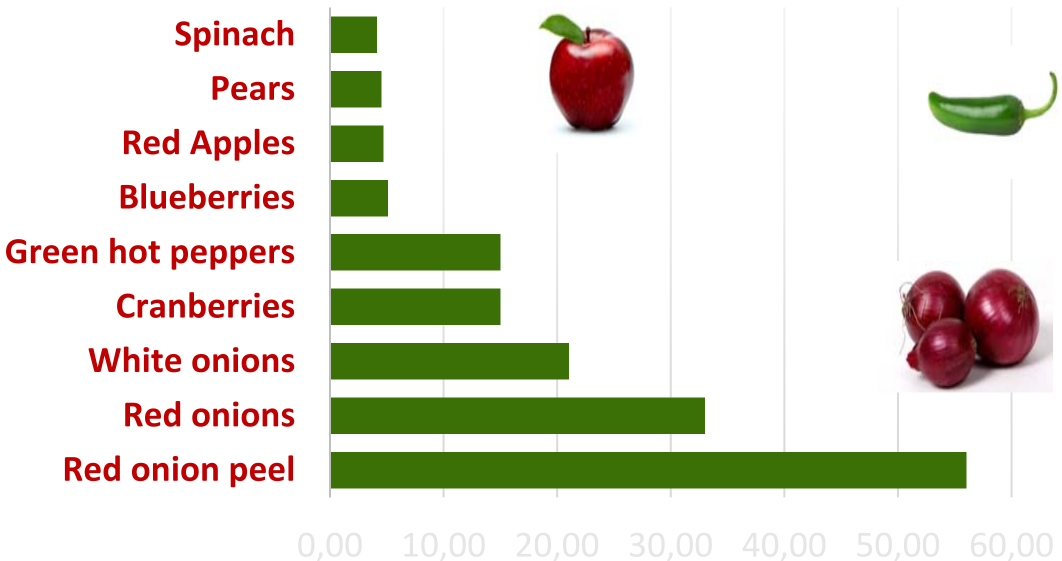


Anthocyanin

Onion leaves

- Contain polyphenols, inulin

Quercetin content (mg/100g)

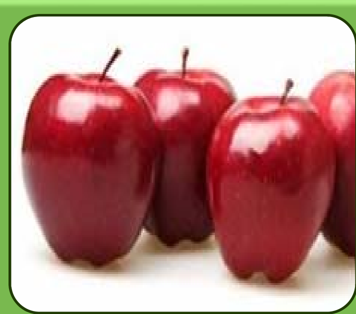


Glycerol as co-solvent

Low cost, by-product of bio-diesel industry

Low dielectric constant

Ideal solvent for polyphenol extraction



3.6% glycerol (w/v)→ more efficient solvent than water

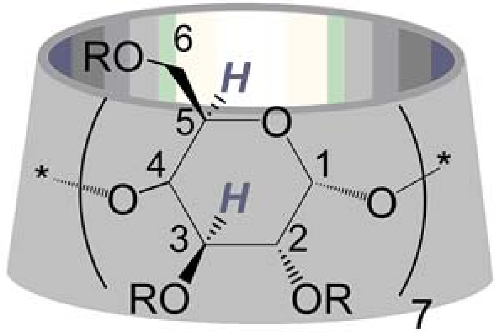
20% glycerol (w/v) → maximum efficiency of flavonoids extraction

Similar efficiency with water/ethanol mixtures

50% (v/v) ethanol, 50% (v/v) butanodiol και 70% (w/v) glycerol for the extraction of polyphenols

Cyclodextrins

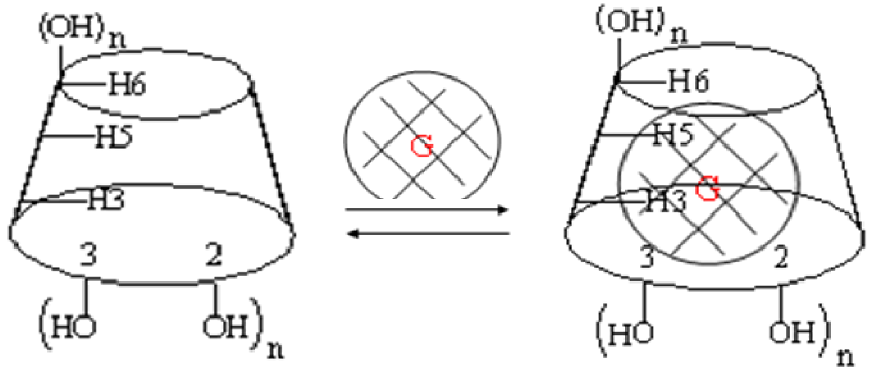
Formation of inclusion complexes with polyphenols



Aqueous solutions of cyclodextrins can be used as extraction co-solvents

R=H: β-CD
R=CH_3-CH(OH)-CH_2: HP-β-CD,
DS=0.5-1.3

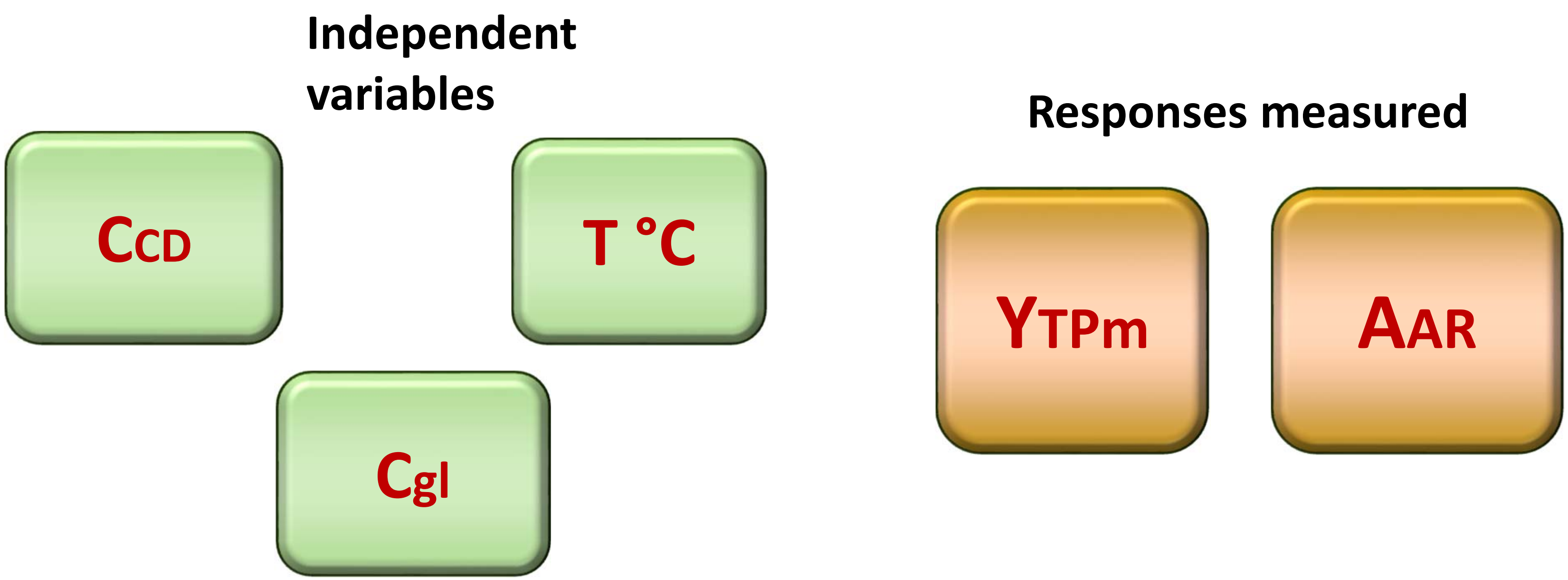
Protection against oxidation and increased stability of the ligand



Aim of the study

Optimization of an extraction process for efficient recovery of polyphenols from onion solid waste, using 'green'' **water/glycerol/2-hydroxypropyl-β-cyclodextrin** ternary mixtures

It was based on a Box-Behnken experimental design



Experimental values and coded levels of the independent variables used

Independent variables	Code units	Coded variable level		
		-1	**0**	**1**
C_{CD} (%,w/v)	X_1	1	7	13
C_{gl} (%, w/v)	X_2	0	30	60
T (°C)	X_3	40	60	80

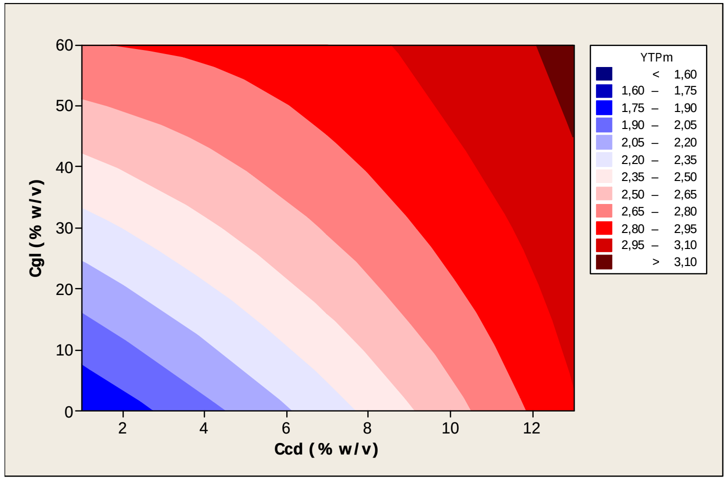
Measured and predicted values of **YTP** and **AAR**, determined for individual design points, for the extractions performed with water/glycerol mixtures

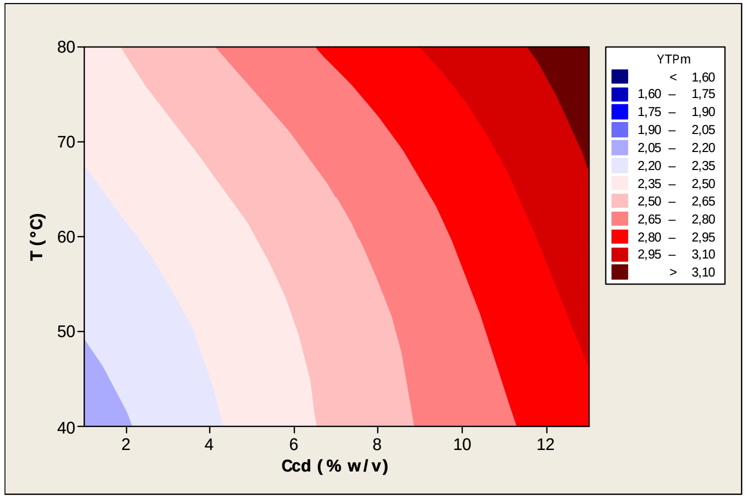
Design point	Independent variables			Response (Y_{TP}, mg GAE g^{-1} dw)		Response (A_{AR}, µmolTR dw)	
	X_1	X_2	X_3	Measured	Predicted	Measured	Predict
1	-1	-1	-1	9.69	7.61	222.55	216.75
2	-1	-1	1	18.96	18.37	202.14	201.98
3	-1	1	-1	14.42	17.52	251.23	238.82
4	-1	1	1	56.78	55.10	311.49	316.14
5	1	-1	-1	19.51	20.74	235.67	231.96
6	1	-1	1	20.6	17.05	207	220.35
7	1	1	-1	22.05	22.19	276.5	277.60
8	1	1	1	43.69	45.317	351.35	358.09
9	-1	0	0	22.24	23.49	183.18	196.90
10	1	0	0	24.6	25.16	242.96	225.47
11	0	-1	0	30.24	35.23	276.5	272.82
12	0	1	0	57.51	54.32	352.81	352.72
13	0	0	-1	23.15	20.76	249.28	270.09
14	0	0	1	33.51	37.70	327.53	302.95
15	0	0	0	40.42	36.42	269.7	276.38
16	0	0	0	36.05	36.42	275.53	276.38

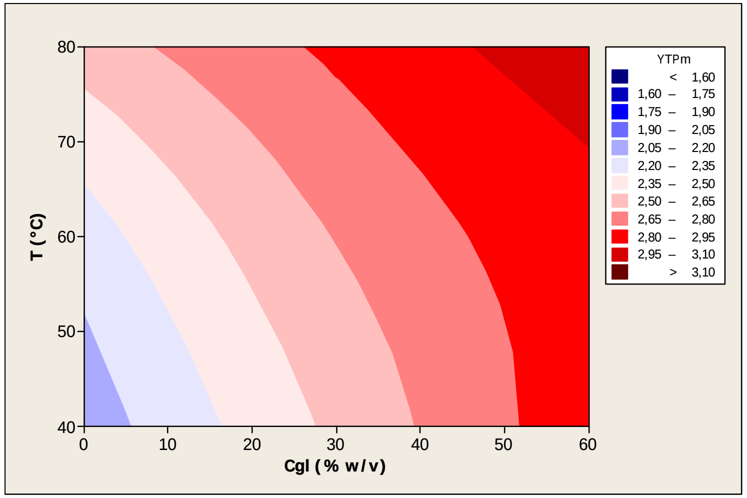
Polynomial equations and statistical parameters describing the effect of the independent variables considered on the responses (Y_{TPm}) and (A_{AR})

Response	**Polynomial equation**	**R^2**	***p***
Y_{TPm}	2.65 + 0.38X1 + 0.31X2 + 0.15X3 – 0.21X1X2	0.94	0.0056
A_{AR}	245.87 + 13.17X1 + 36.49X2 + 14.80X3 + 21.22X2X3 - 46.46X1^2 + 32.75X2^2	0.95	0.0037

Contour plots illustrating the effect of the independent variables examined on the YTPm

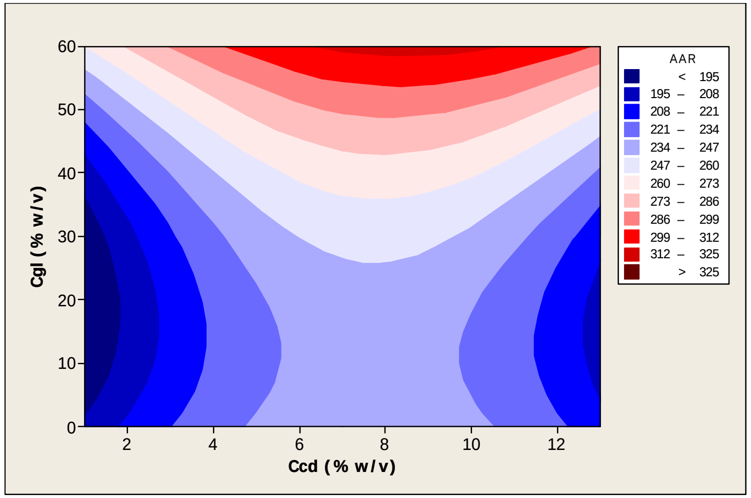


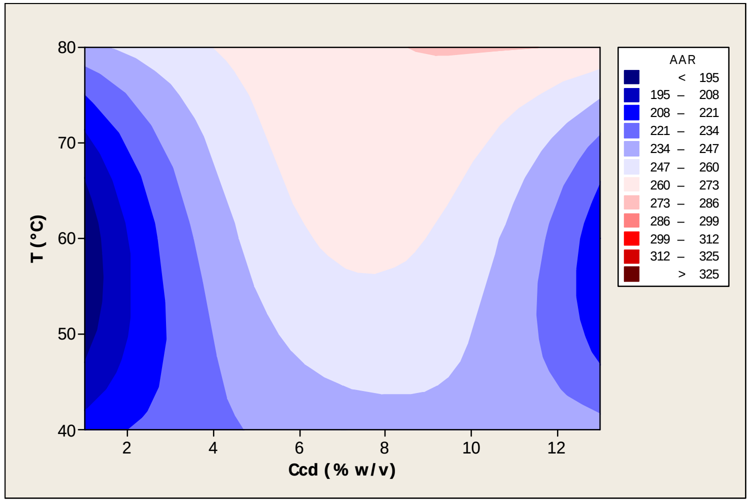


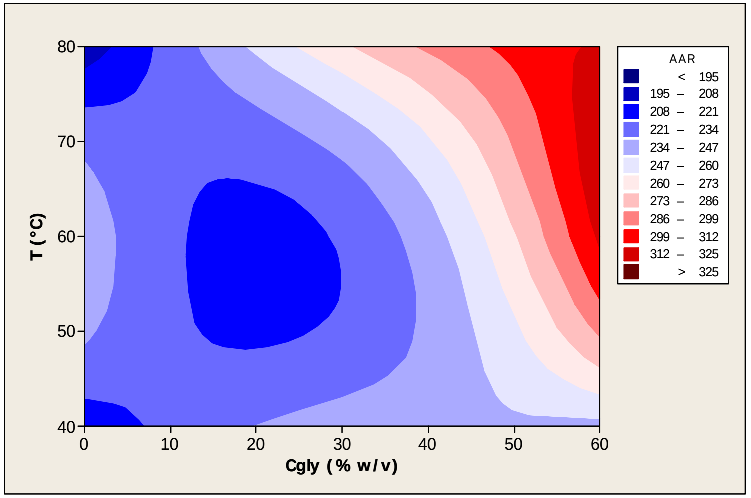


C_{CD} = 13% w/v, C_{gl} = 60% w/v, T = 80°C

Contour plots illustrating the effect of the independent variables examined on the AAR

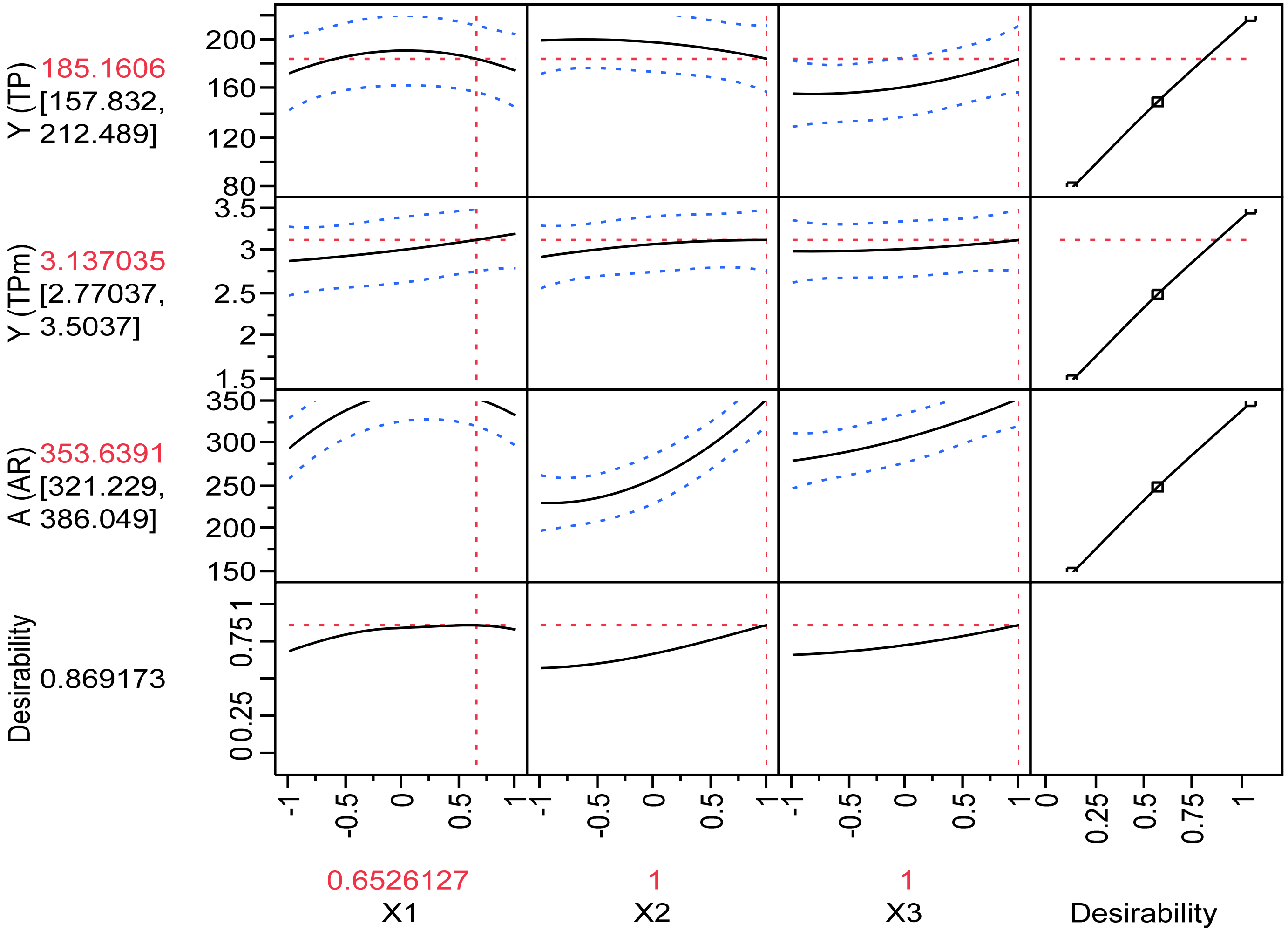






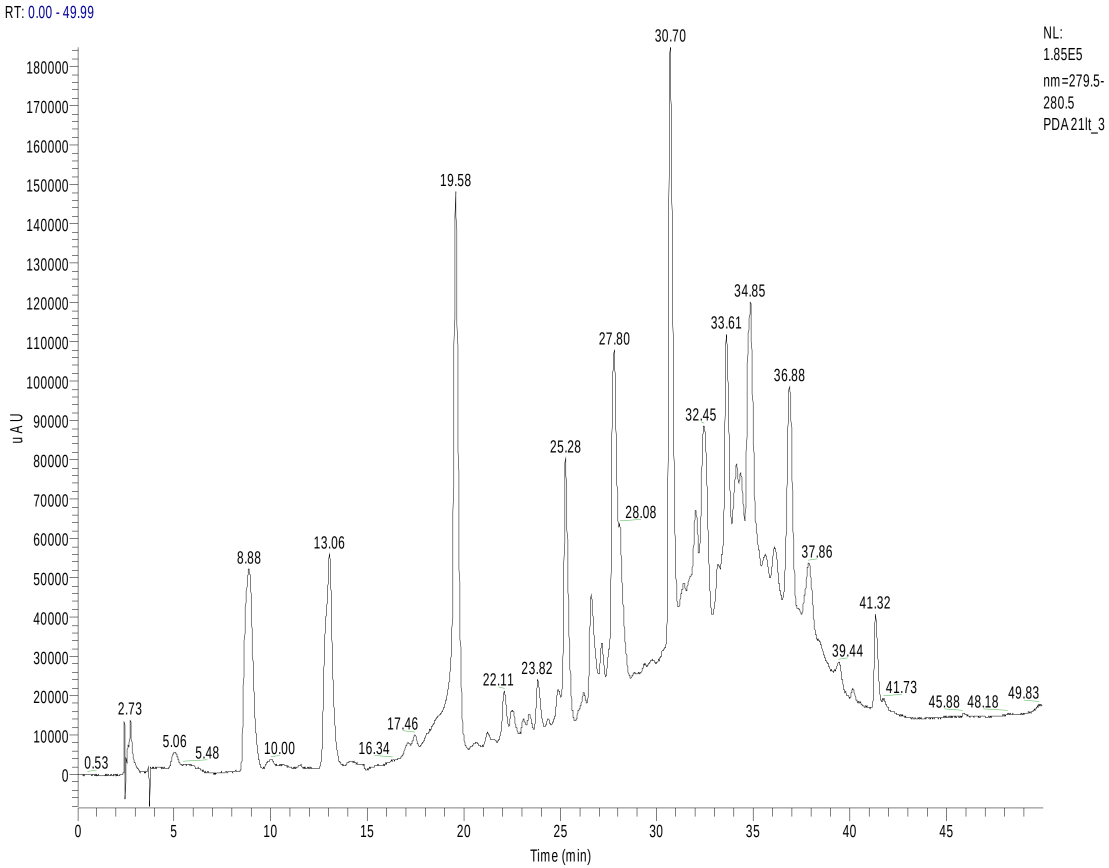
C_{CD} = 8% w/v, C_{gl} = 60% w/v, T = 80^{o}C

Prediction profiler displaying the overall desirability of the model, following adjustment of the independent variables at their optimal values



C_{CD} = 13% (w/v),
C_{gl} = 60% (w/v) ,
T = 80^{o}C.

LC – MS analysis



Peak	Rt (min)	λ_{max} (nm)	$[M+H]^+$	Other ions (*m/z*)	Compound
1	8.88	292	153	-	Unknown
2	13.06	258, 292	587	487, 325	Unknown
3	13.58	292	319	337	2-(3,4-dihydroxybenzoyl)-2,4,6-trihydroxybenzofuran-3(2H)-one
4	18.75	292, 514	449	-	Cyanidin 3-O-glucoside
5	22.30	254, 374, 522	535	441, 287	Malonylated cyanidin 3-O-glucoside derivative
6	26.61	292			Unknown
7	27.80	252, 320(s), 366, 384(s)	465	303	Quercetin glucoside
8	30.70	256, 318(s), 368, 3929(s)	303		Quercetin
9	32.45	256, 304, 368	765	603, 303	Quercetin glucoside / quercetin dehydrate adduct
10	33.61	248, 270(s), 304(s), 364	765	303	Quercetin glucoside / quercetin dehydrate adduct
11	34.85	248, 268(s), 304, 364	603	303	Quercetin dimmer
12	36.88	242, 272(s), 304, 362	603	303	Quercetin dimmer
13	37.86	240, 300, 360	601	303	Quercetin dehydrodimer

Incorporation in a yogurt matrix

Onion leaves
↓
Green extraction
↓
Natural colorant → (added after Heating, before Incubation)

Sweet whey
↓
Addition of ingredients
↓
Heating
↓
Incubation
↓
Cooling and storage

Flavor → (added after Heating, before Incubation)

Incorporation in yogurt

Quality Characteristics of yogurt					
Solids content %	Colour	Complex Viscosity (Pa.s)	Storage modulus, G' (Pa)	Loss modulus, G'' (Pa)	tanδ
15	L*69,79 a +13,98 b +11,70	156	10^3	$3x10^2$	0.3

Spectrophotometric test	**Value**
Total polyphenol yield (Y_{TP})	1157 mg gallic acid /L of extract
Antiradical activity (A_{AR})	1609 mM Trolox

Conclusions

- Development of a novel approach for more efficient extraction of polyphenols from onion solid wastes, leading to eco-friendly extracts and processes.

- Green-extraction techniques minimize the use of petrochemicals.

- Liquid extracts of plant polyphenols could become attractive and safe vehicles of these compounds to fortify food products or used as natural food colourants.

- Extracts should be also tested for their stability upon storage to maximize their effectiveness in providing functionality in a real food matrix.

Thank you for your attention