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Recovery of spent coffee grounds phenolic compounds through optimized extraction processes

A. Loukri. N. Solomakou. P. Tsafrakidou. A-M. Michaelidou. I. Mourtzinos. A.M. Goula

Department of Food Science and Technology, School of Agriculture, Forestry and Natural Environment, Aristotle University, 541 24 Thessaloniki, Greece

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Coffee husk

World coffee consumption ↑ 1.3% from 2019-2020(≈9,852 millions kg 2019/2020) (ICO,2021)

Dry

processing

roasting

brewing

Wet

Coffee pulp

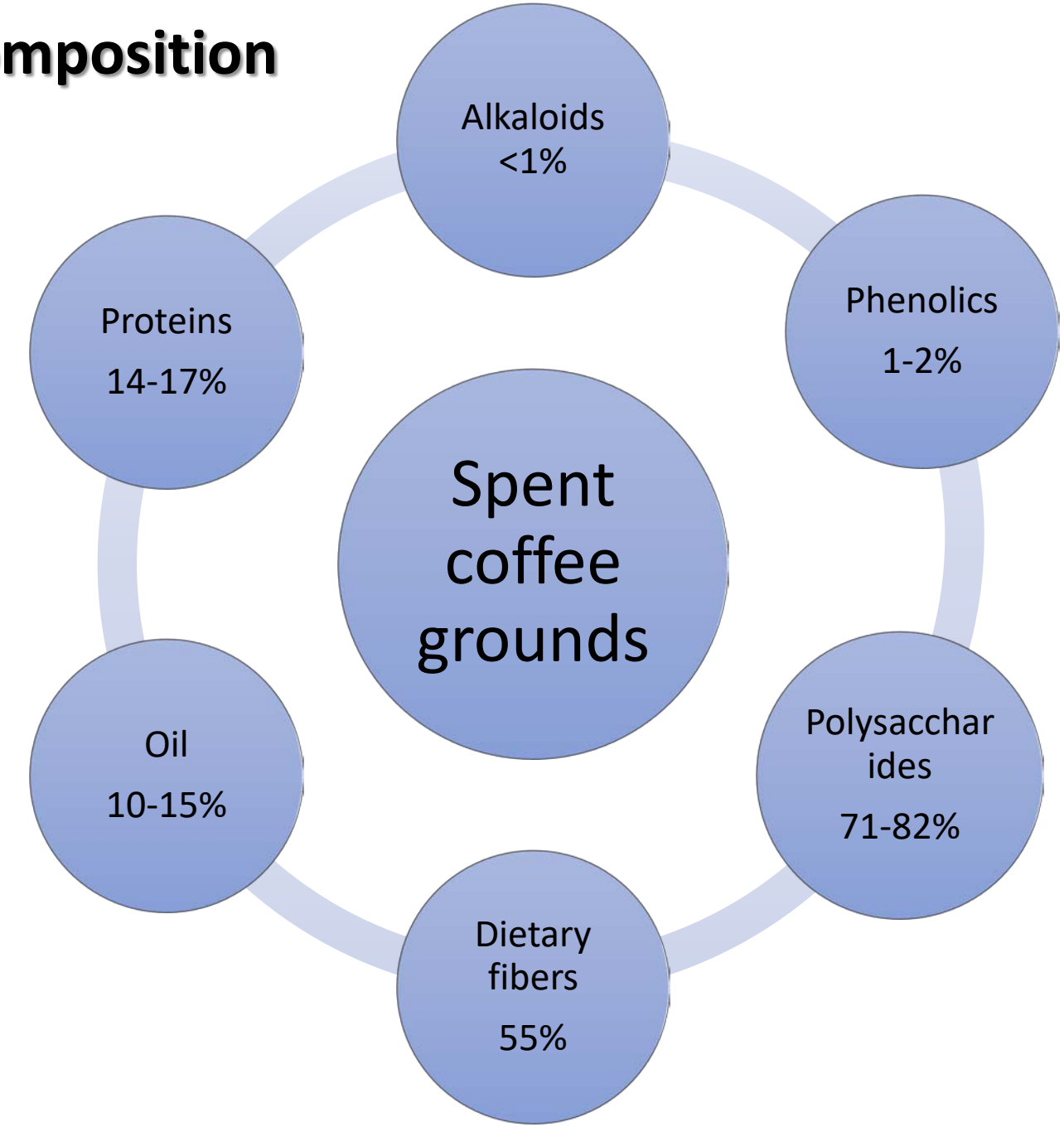
Silver skin

Spent coffee grounds

1 ton green coffee beans
=> 650 Kg Spent coffee grounds

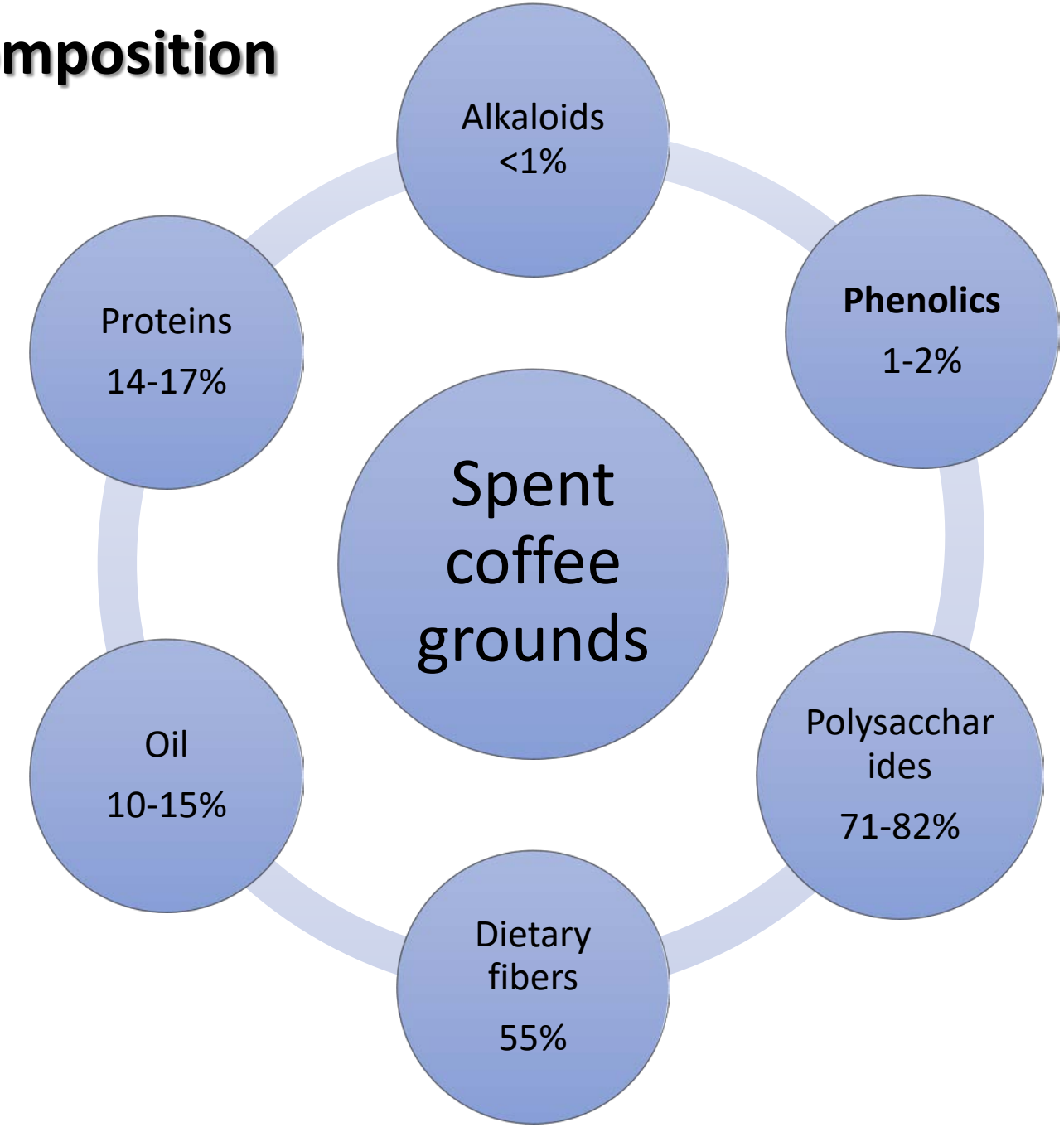
Murthy & Naidu, 2012

SCG composition



Gemechu,2021, Ramón-Gonçalves et al.,2019

SCG composition

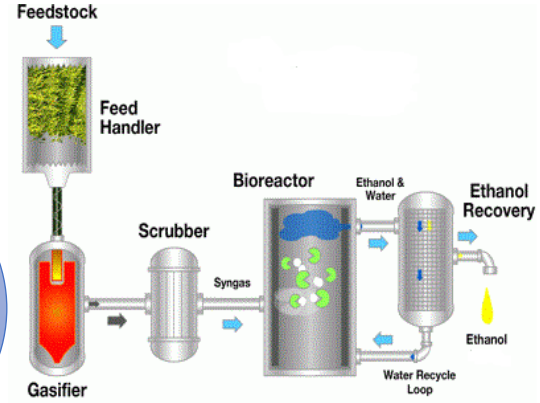


- **Chlorogenic acid**
- Caffeic acid
- Ellagic acid
- Ferulic acid
- Gallic acid
- Hydroxybenzoic acid
- Coumaric acid
- Pyrocatechinic acid
- Tannic acid
- Quercetin
- Catechin
- Epicatechin
- Rutin

Gemechu,2021, Ramón-Gonçalves et al.,2019

SCG management

Bioenergy-biofuel



Bioethanol

Foods

Circular

Spent coffee grounds

Economy

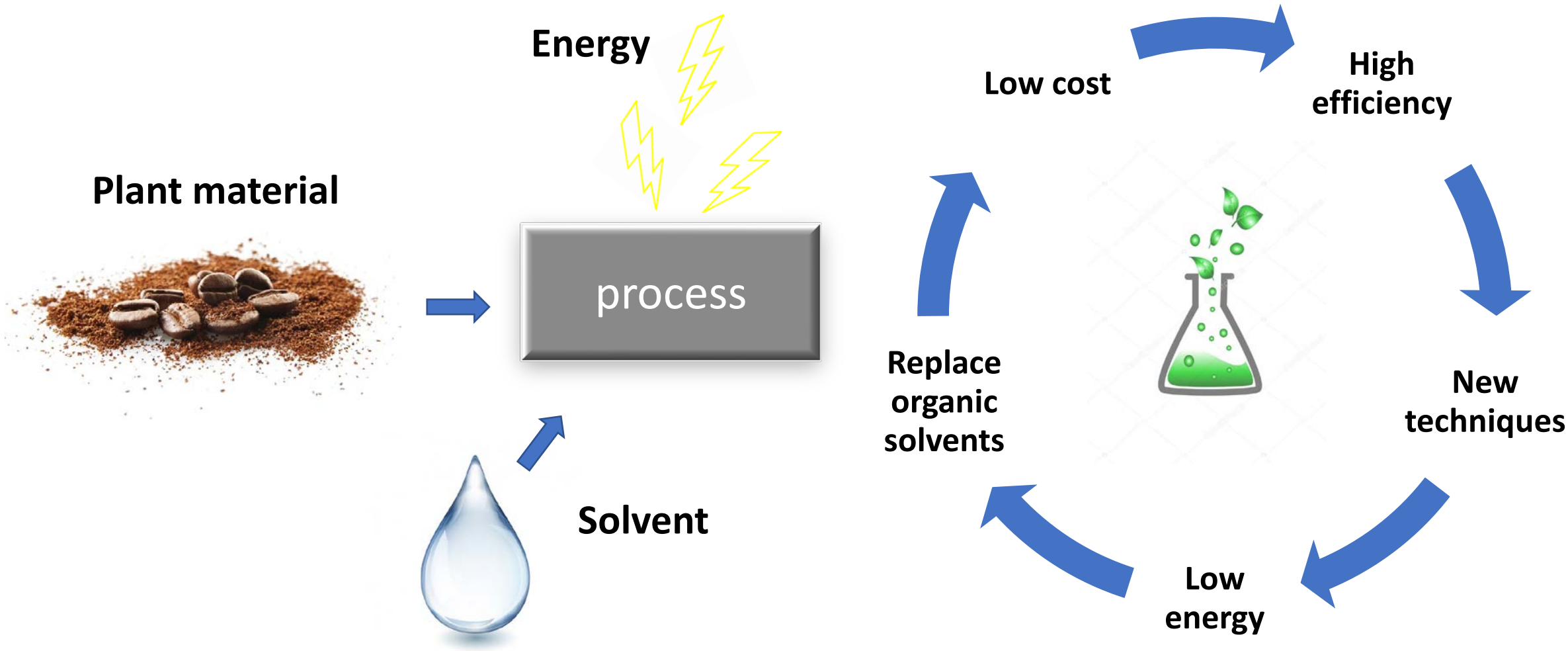
Fertilizer Animal feed

Food supplements , cosmetics

Packaging

Massaya et al., 2019, McNutt & He ,2019

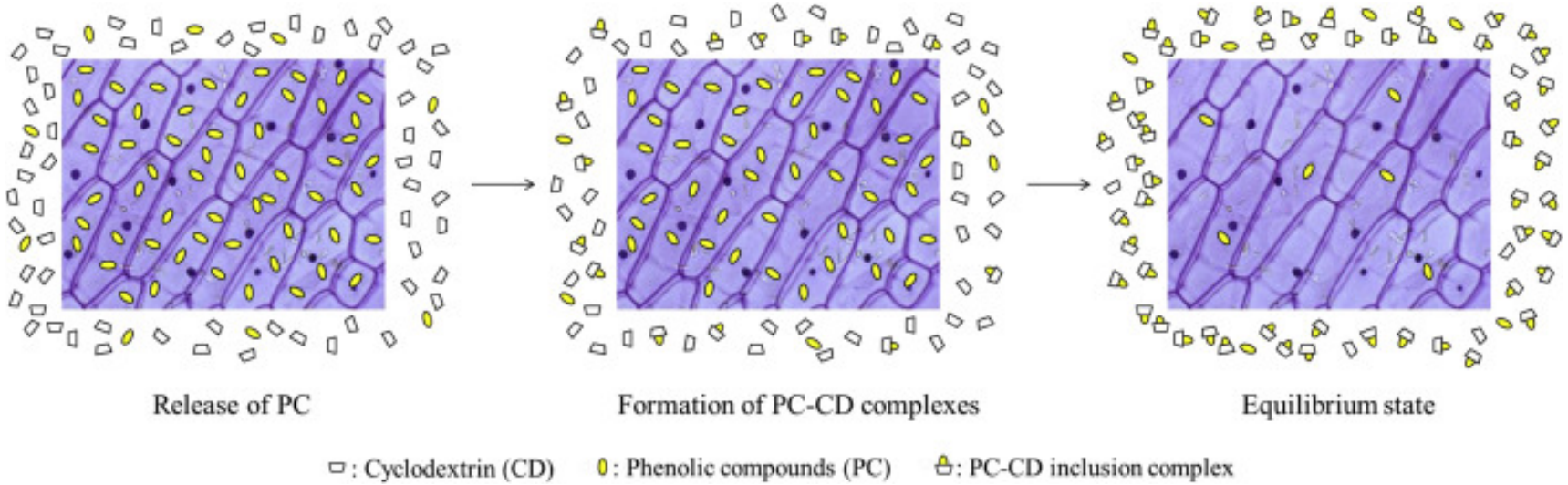
Extraction procedure



Chemat et al., 2019

Extraction with cyclodextrin

- Starch derivatives
- Hydrophilic surface and hydrophobic cavity



Increase extraction

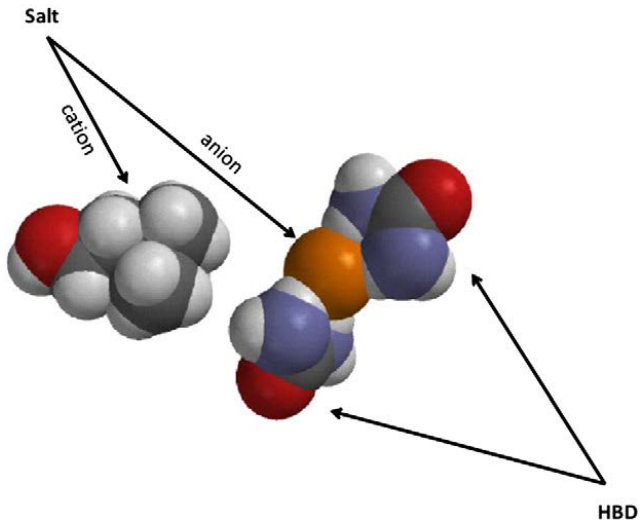
Enhance stability

Masking

Possible use as debittering agent in coffee

Loukri et al., 2020, Cai et al., 2018

Extraction with Deep eutectic solvents



HBA **HBD**

Choline chloride

Glycerol

Stirring

80°C

Colorless

High viscous

Water

Yoo et al., 2018, Mbous et al.,2017

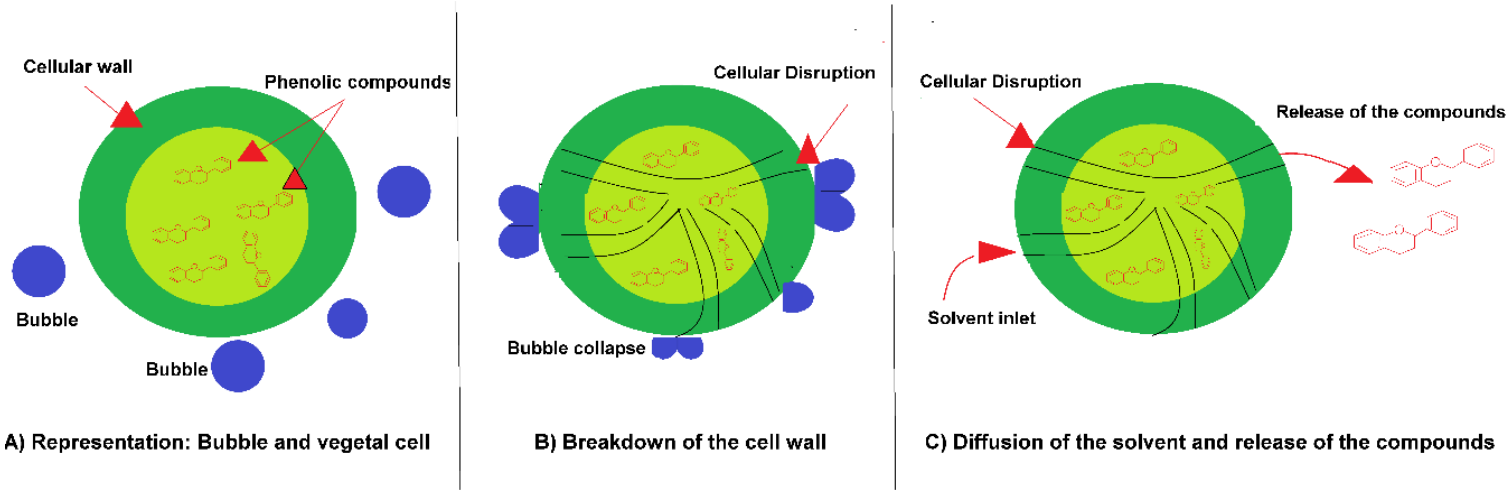
Ultrasound Assisted extraction a "green method"

acoustic cavitation

- Mechanical waves
- Compressions and reflections of the solvent

Medina-Torres

Formations of bubbles



Enhance extraction
Low time of extraction
Less energy consumption

Medina-Torres et al., 2017, Goula, 2013

Materials and methods

Spent coffee grounds *Coffea Arabica*

Dried => 10% moisture content
Particle size >50% 240 µm

Response surface analysis

Extraction yield (total phenolics mg/g dw)
(Folin–Ciocalteu)

Solvent type

- Aqua solutions of β-cd
- Aqua solutions of deep eutectic solvent
- Aqua solutions of Ethanol

Extraction method

- Ultrasound
- Heat and stirring

Experimental designs

Extraction with β-cyclodextrin

Variables	Levels				
	-1.68	-1	0	1	1.68
Cβ-cd (mg/mL)	1	4.55	9.75	14.95	18.5
L/S (mL/g)	5	16	33	49	60
T (°C)	20	28	40	52	60

20 experiments

Extraction with DES

Variables	Levels				
	-1.68	-1	0	1	1.68
CDES (% w/v)	35	47	65	83	95
L/S (mL/g)	5	16	33	49	60
T (°C)	20	28	40	52	60

20 experiments

Extraction with ethanol

Variables	Levels				
	-1.68	-1	0	1	1.68
EtOH (%v/v)	0	20	50	80	100
L/S (mL/g)	5	16	33	49	60
T (°C)	20	28	40	52	60

20 experiments

Ultrasound assisted extraction

	-2	-1	0	1	2
EtOH (%v/v)	0	25	50	75	100
L/S (mL/g)	5	18.75	32.5	46.25	60
T (°C)	20	30	40	50	60
A%	20	30	40	50	60

31 experiments

L/S Liquid to solid ratio

T Temperature

Cβ-cd Concentration of cyclodextrin in aqua solution

CDES Concentration of DES in aqua solution

EtOH Concentration of ethanol in aqua solution

A Altitude of the ultrasound waves

MINITAB (release 13.32)

Results

Regression analysis

Significance of main effects and interaction

Extraction with b-cyclodextrin

	Coef	P
Constant	2.9895	0.139
Cβ-cd	0.20568	0.135
L/S	0.12734	0.01
T	0.13825	0.061
Cβ-cd*Cβ-cd	-0.00487	0.222
L/S*L/S	-0.00096	0.029
T*T	-0.00059	0.428
Cβ-cd*L/S	-0.00109	0.506
Cβ-cd*T	-0.00142	0.529
L/S*T	0.0002	0.776

Extraction with DES

	Coef	P
Constant	-5.1894	0.596
CDES	-0.10137	0.426
L/S	0.48715	0.021
T	0.52729	0.109
CDES*CDES	-0.00055	0.448
L/S*L/S	-0.00423	0.016
T*T	-0.00523	0.083
CDES*L/S	0.00164	0.432
CDES*T	0.00382	0.198
L/S*T	-0.00491	0.135

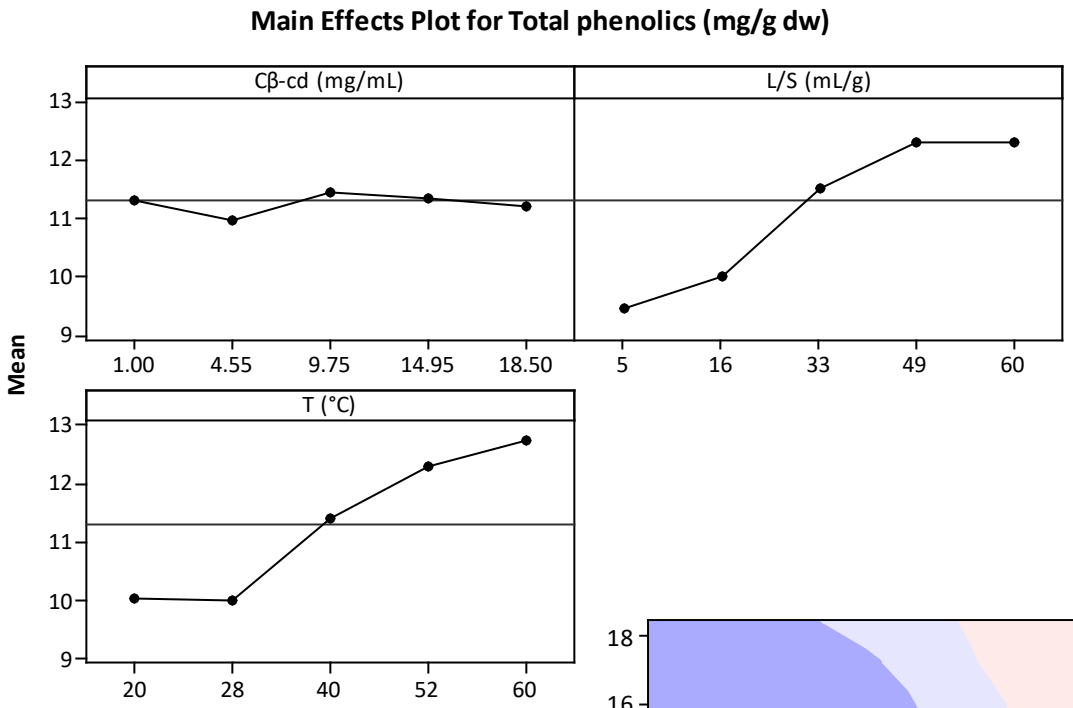
Extraction with Ethanol

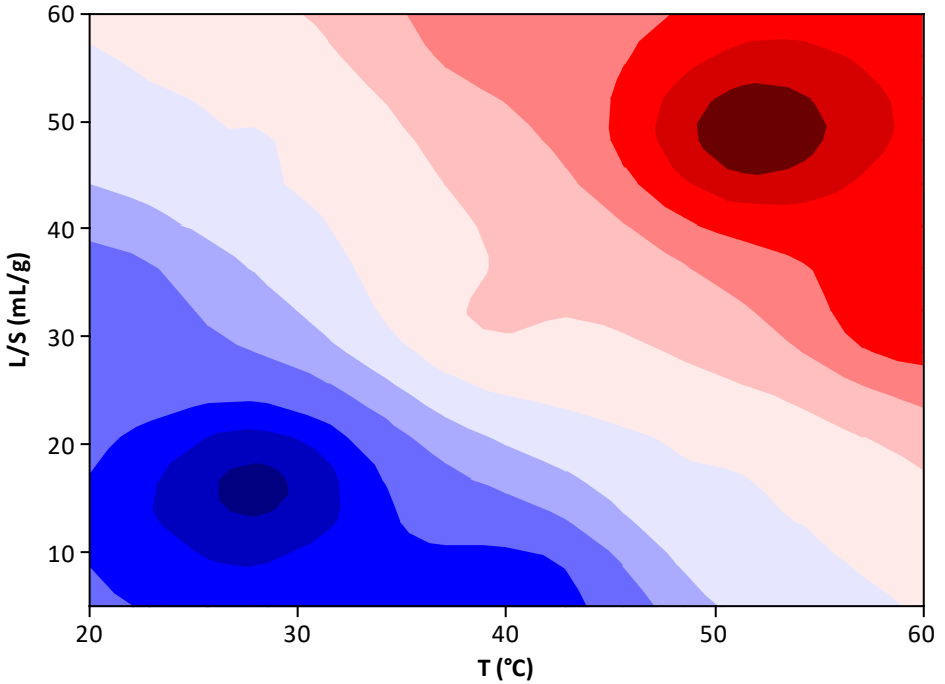
	Coef	T
Constant	-2.45271	0.625
L/S	0.49736	0.001
EtOH	0.18015	0.011
T	-0.03563	0.844
L/S*L/S	-0.00538	0
EtOH*EtOH	-0.003	0
T*T	0.00236	0.253
L/S*EtOH	0.00266	0.005
L/S*T	-0.00228	0.251
EtOH*T	0.00135	0.219

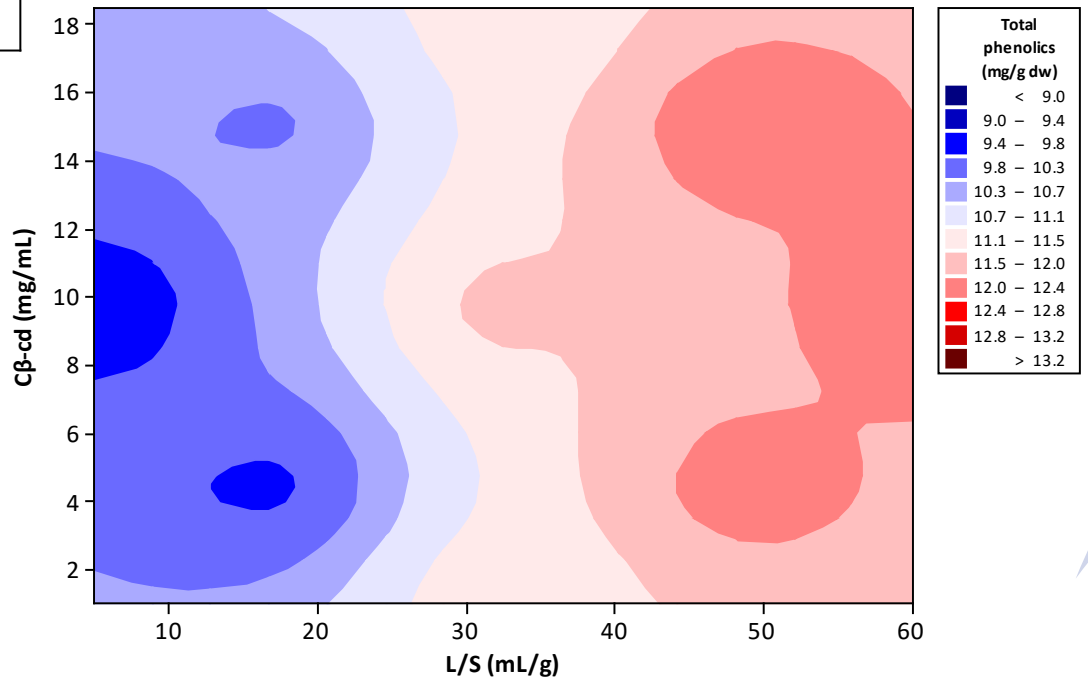
Ultrasound assisted extraction

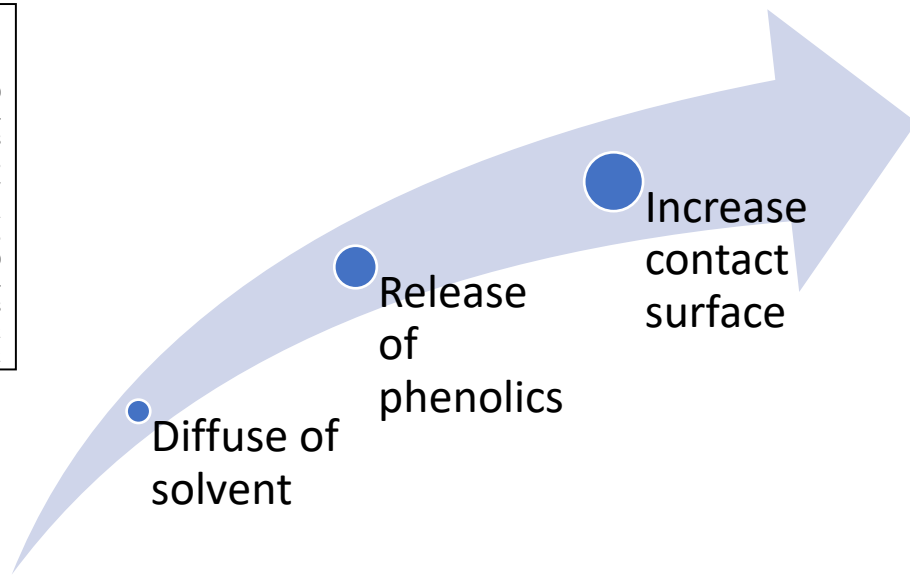
	Coef	P
Constant	374.100	0.774
T	-0.30434	0.367
EtOH	0.34788	0.008
L/S	0.34255	0.126
A	-0.33852	0.317
T*T	0.00254	0.427
EtOH*EtOH	-0.00296	0.000
L/S*L/S	-0.00367	0.041
A*A	0.00456	0.162
T*EtOH	0.00039	0.818
T*L/S	0.00312	0.318
T*A	0.00171	0.686
EtOH*L/S	-0.00229	0.077
EtOH*A	0.00082	0.631
L/S*A	-0.00046	0.882

Extraction with β-cyclodextrin

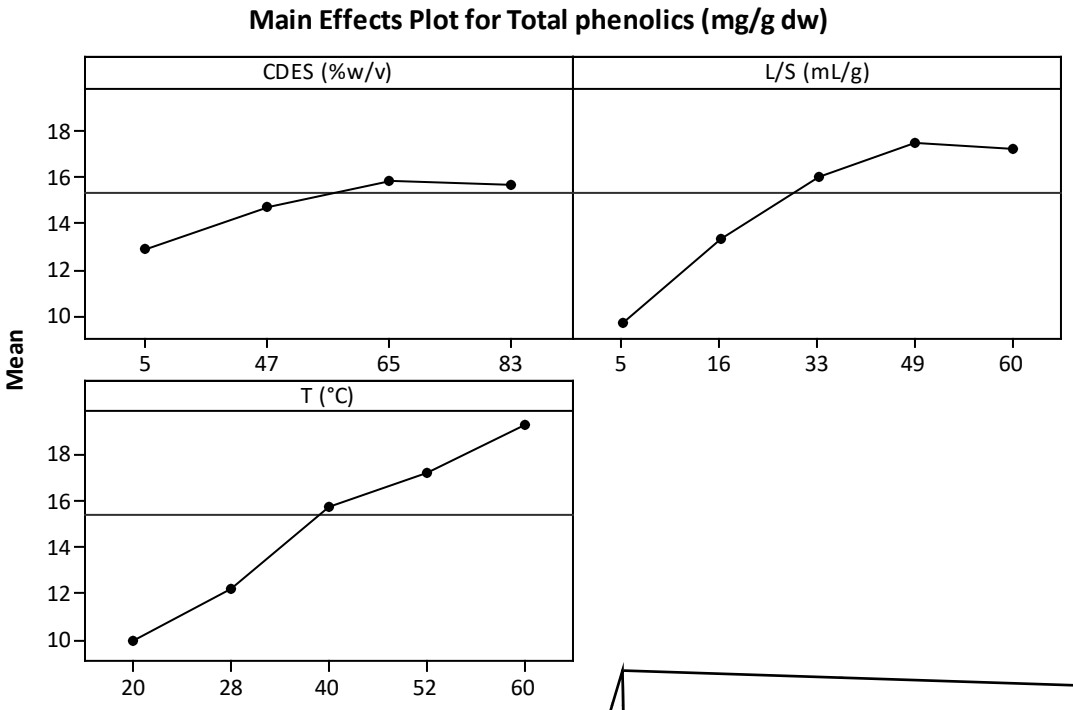


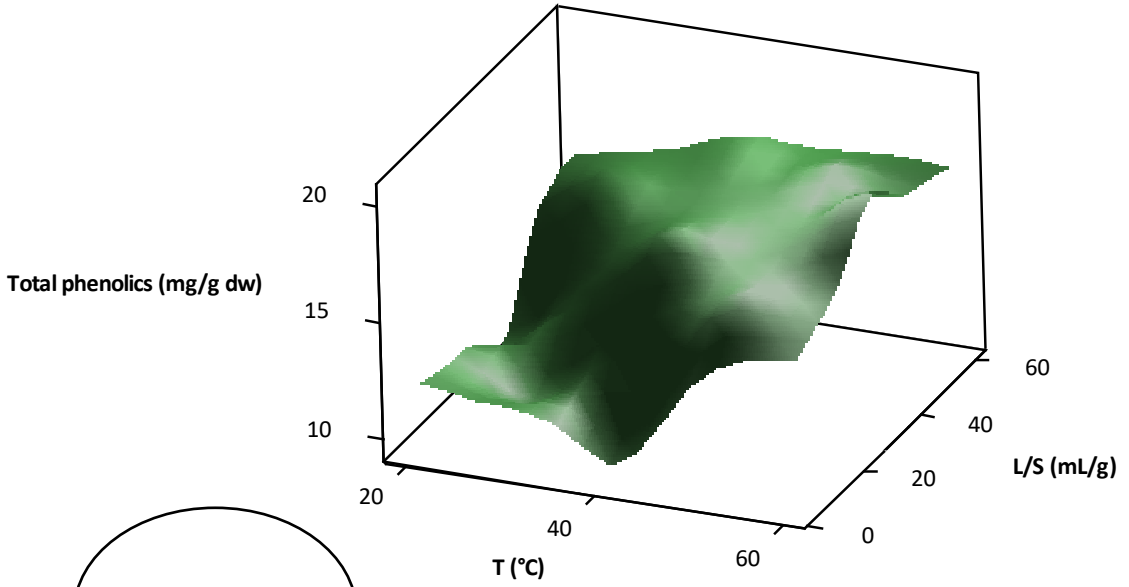


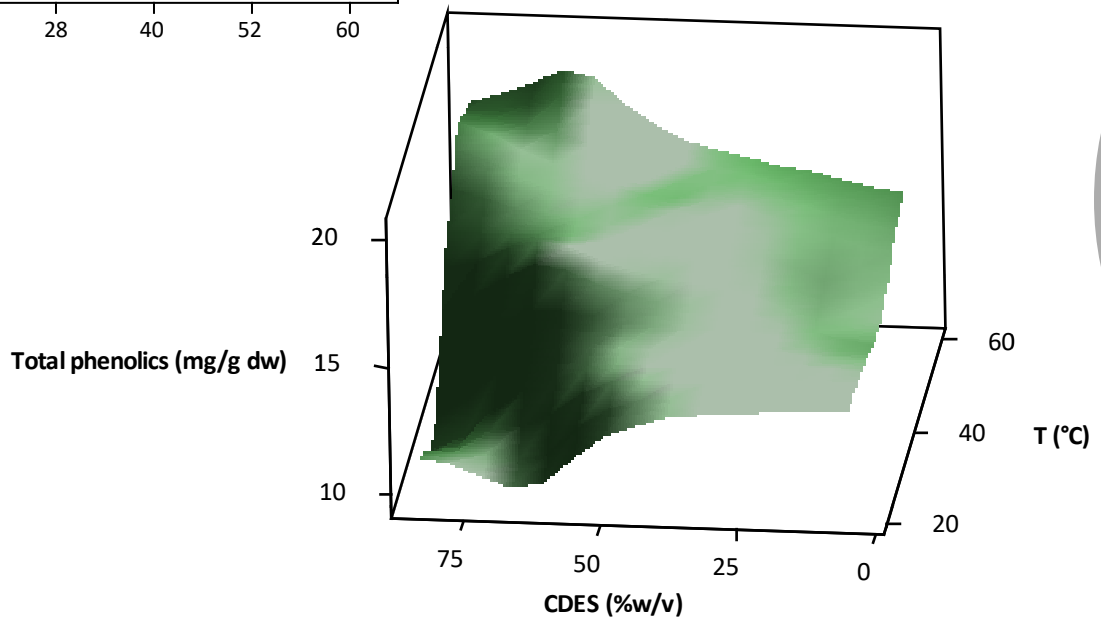


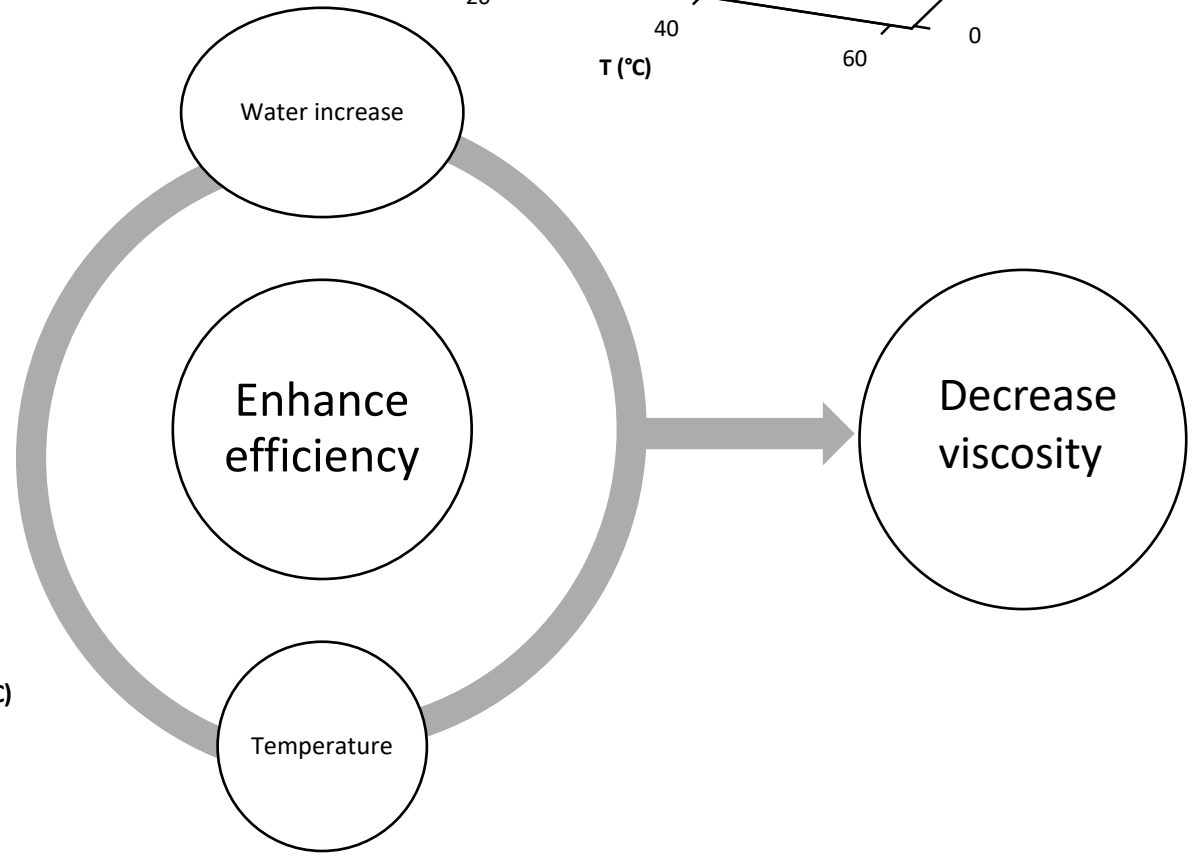


Extraction with DES

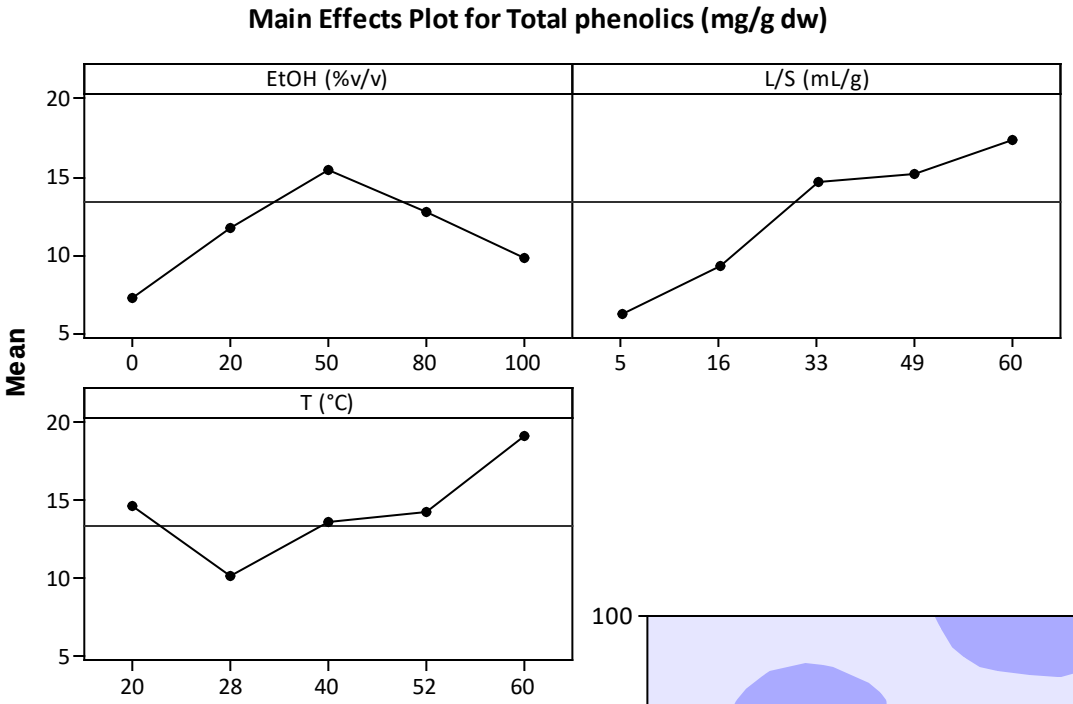


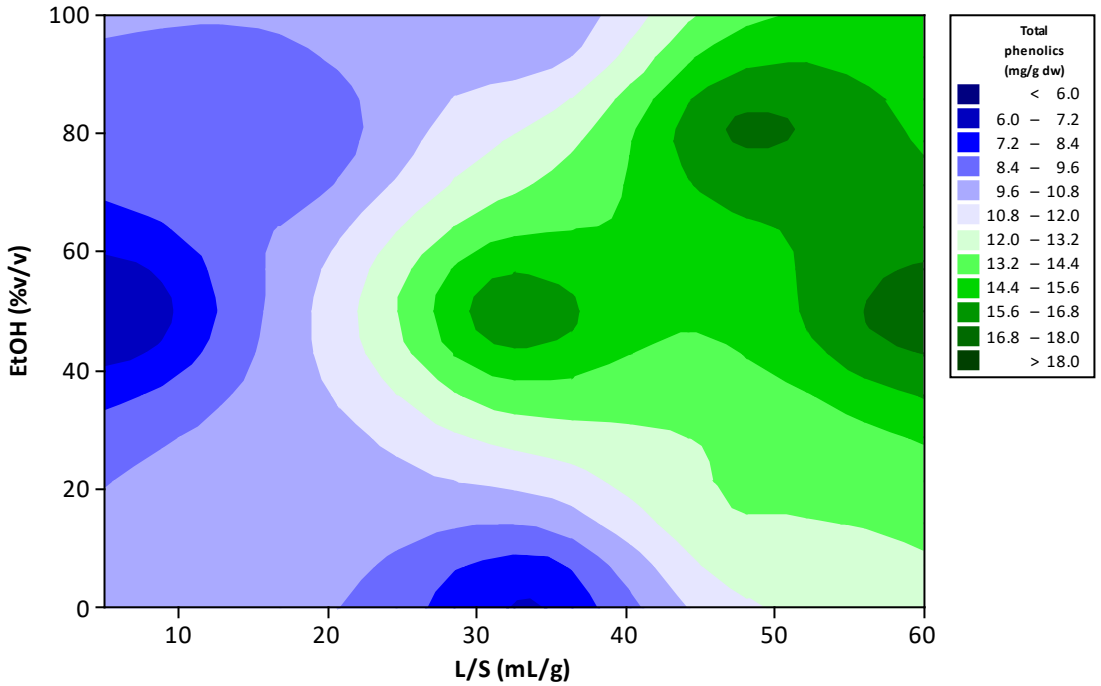


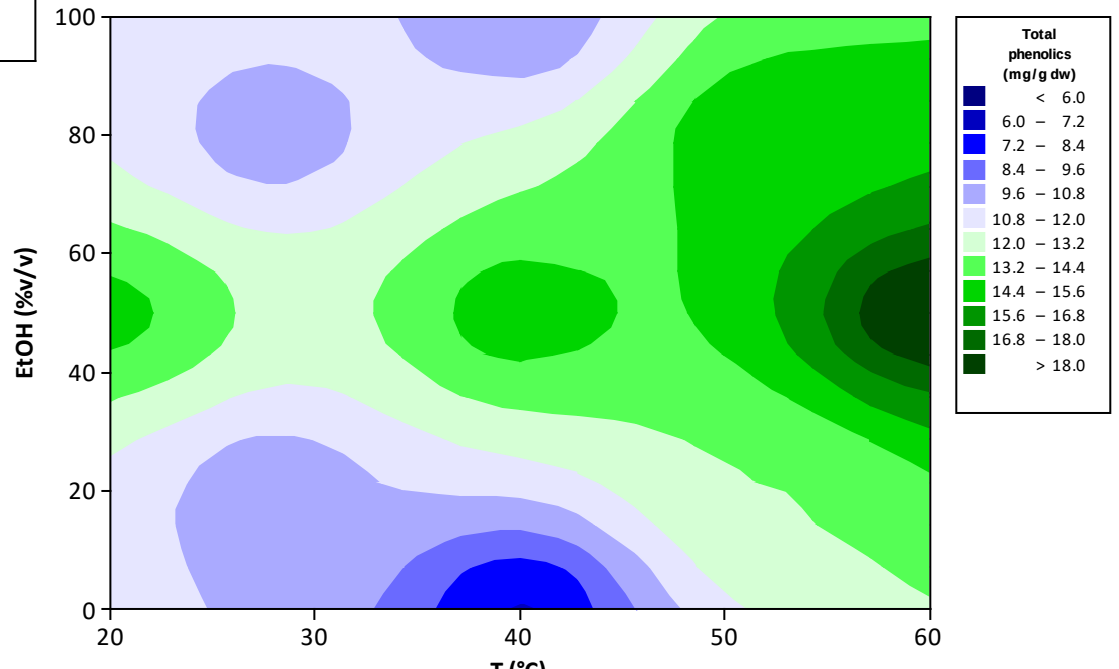


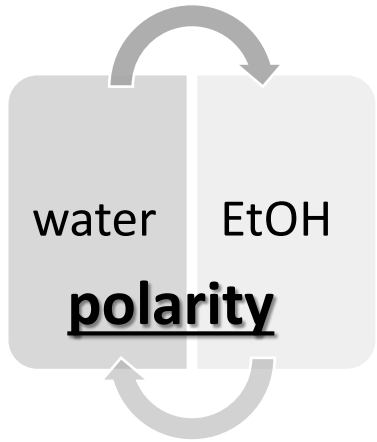


Extraction with Ethanol

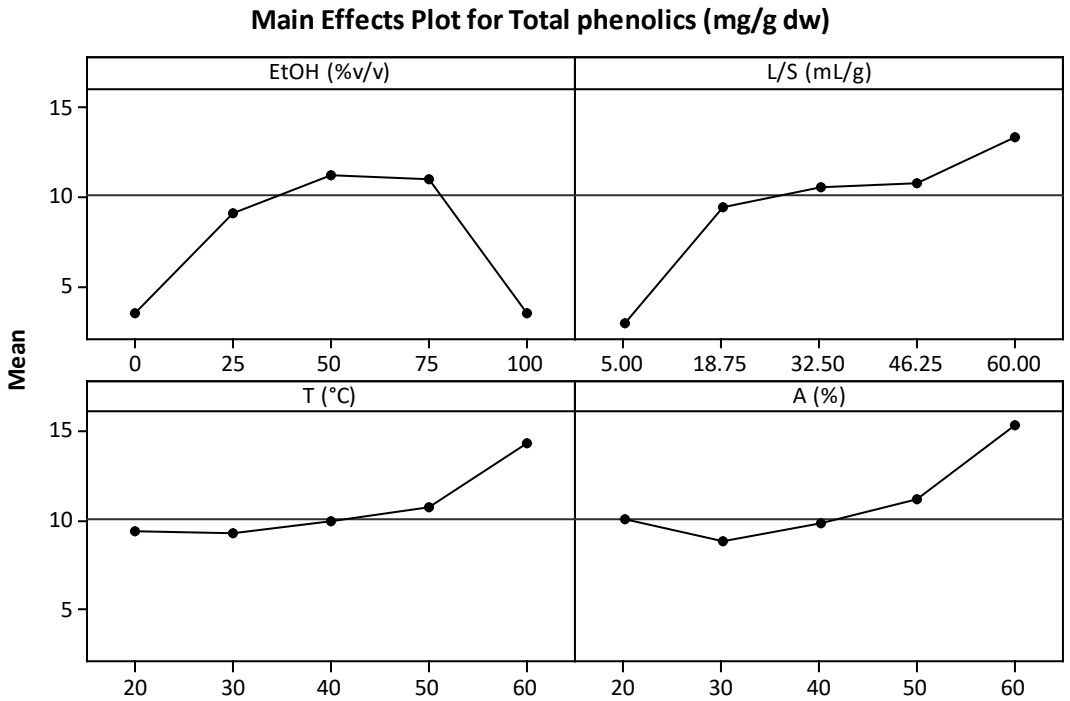


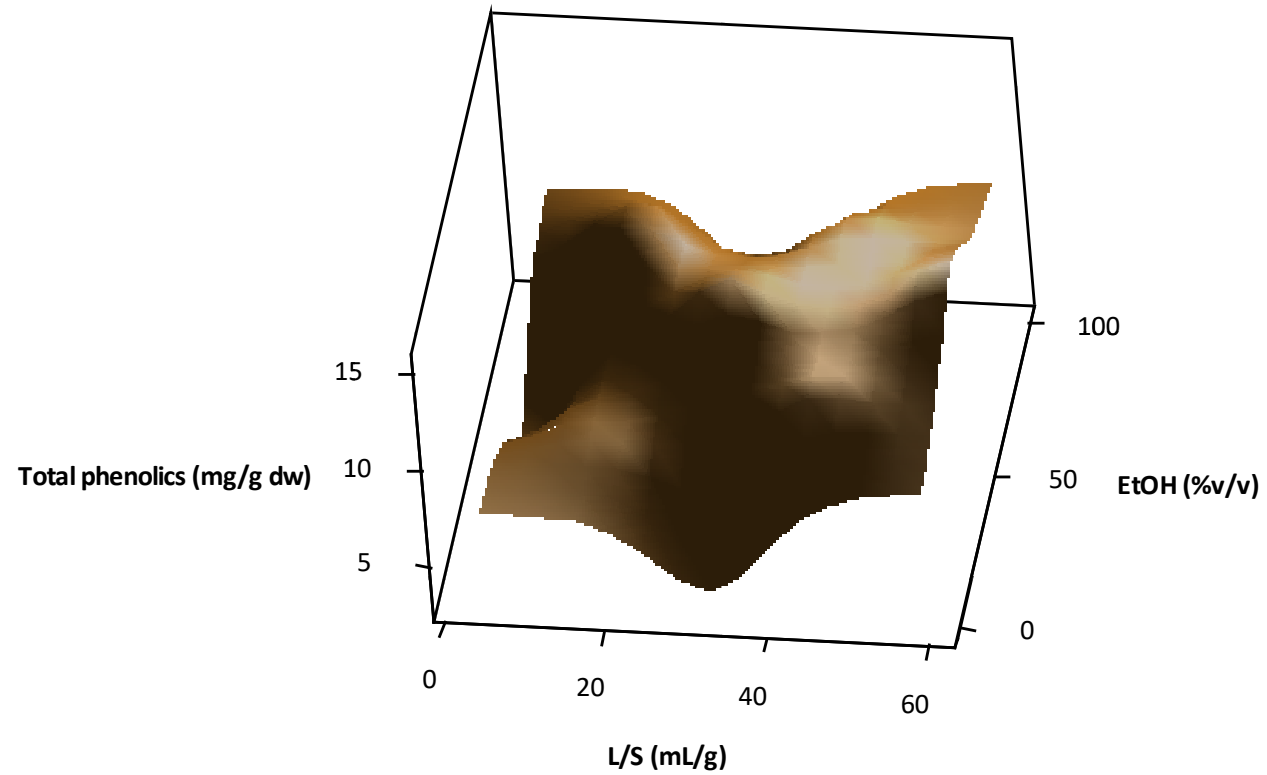


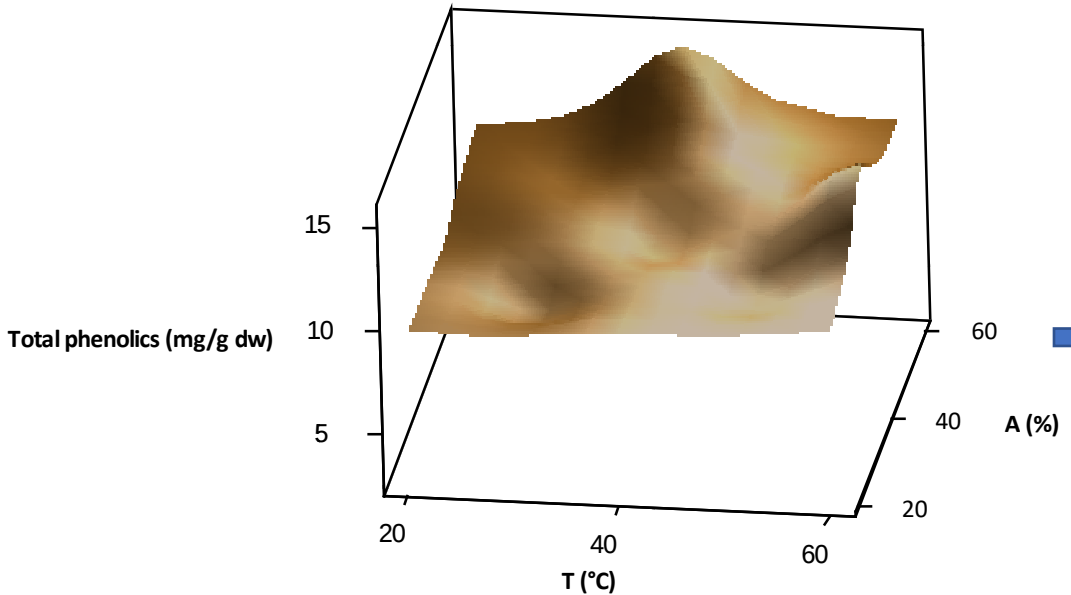




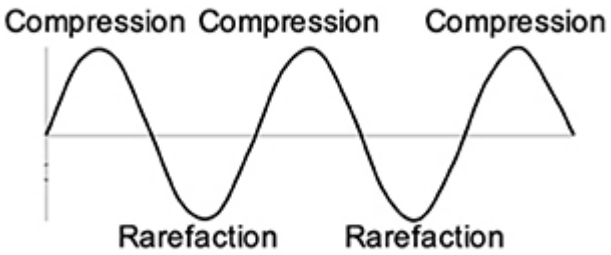
Ultrasound assisted extraction





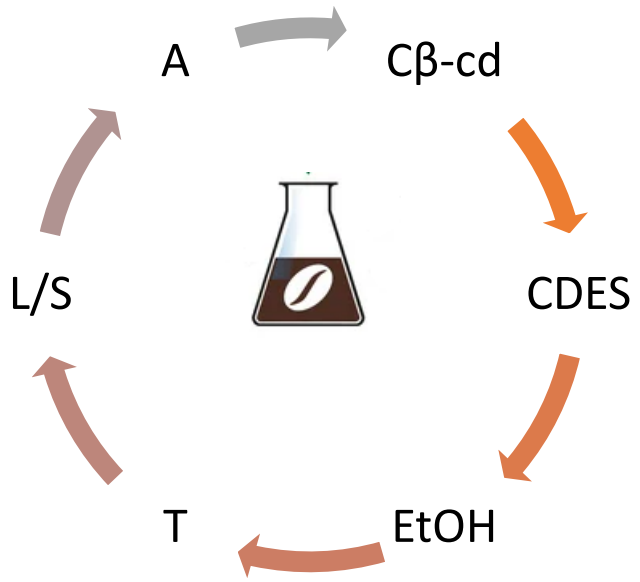


Rapture of the cells better penetration of solvent



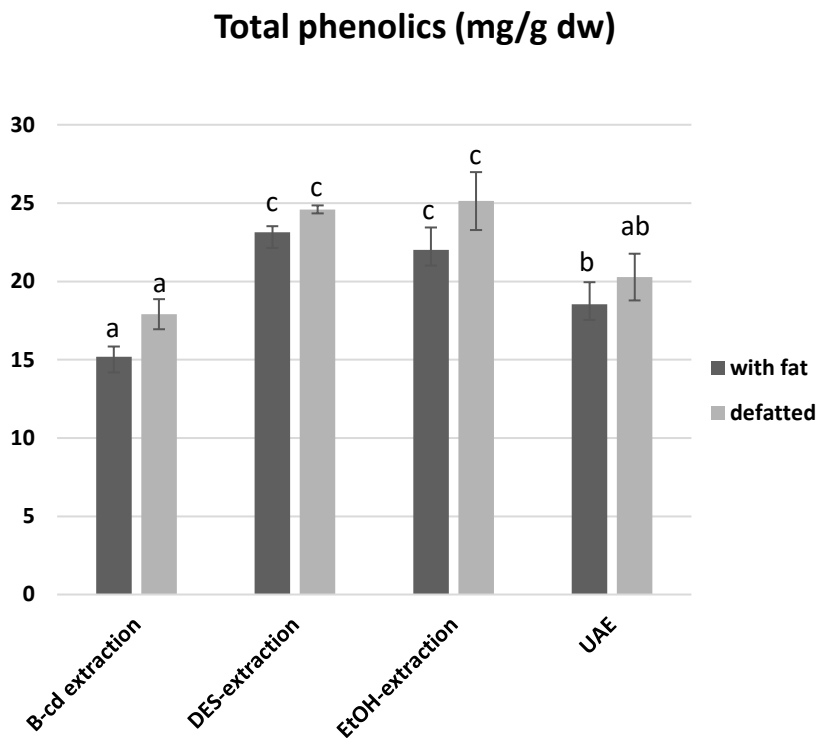
Optimum conditions

Type of extraction	Conditions	Experimented extraction yield (mg/g dw)	Predicted extraction yield (mg/g dw)
B-cyclodextrin	• **Cβ-cd** 5.7 mg/mL • **L/S** 60 mL/g • **T** 60°C	15.16±0.6	14.22
DES	• **CDES** 83 % w/v • **L/S** 39 mL/g • **T** 60°C	23.14 ±0.4	20.82
Ethanol	• **EtOH** 50% v/v • **L/S** 66 mL/g • **T** 60°C	22.01±1.4	21.42
Ultrasound	• **EtOH** 51 %v/v • **L/S** 53 mL/g • **T** 60 °C • **A** 60 %	18.54 ±1.4	20.77

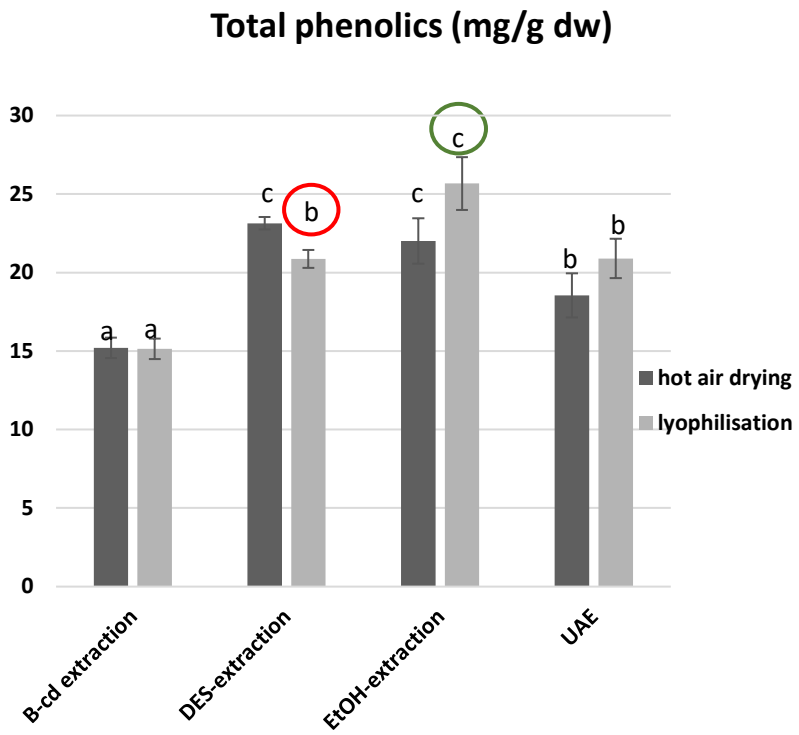


Effect of pretreatments on extraction yield (optimum conditions)

Effect of defatting



Effect of drying



Enhance extraction of polar compounds

Cell structure
Rate of degradation

Conclusions

4 experimental designs developed → To increase extraction yield

Aqua solutions of cyclodextrin had the lower efficiency compared to DES and EtOH

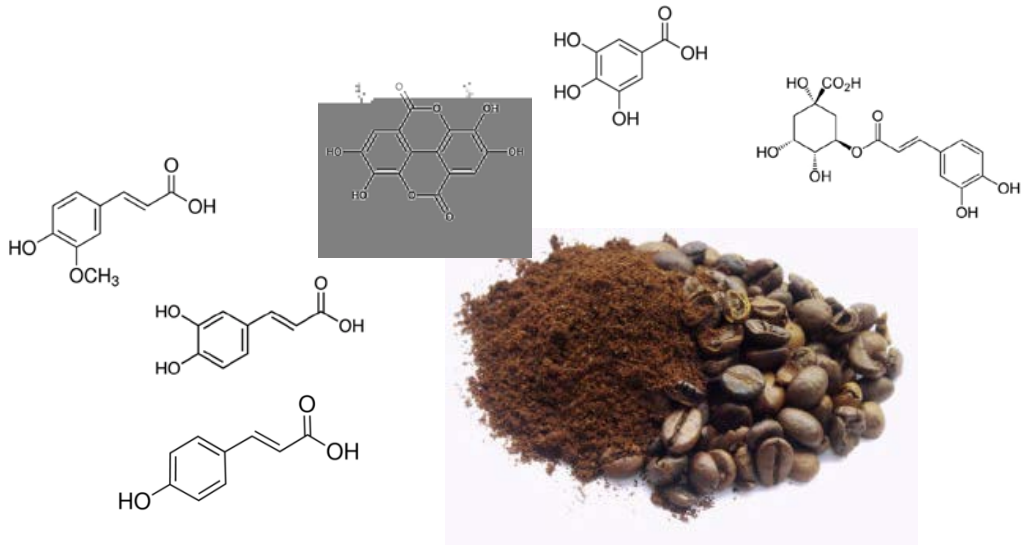
Ultrasound Assist extraction didn't enhance the recovery of phenolics, but decreased time of extraction by **75 %**

Removal of oil content enhanced the recovery in all cases

Extraction with Ethanol was the most efficient technique

Next step

Microwave assisted extraction



Coffee bio-yo

Optimizing the technofunctional and biofunctional attributes of a yoghurt-based product using Bacterial Cellulose Fiber from spent coffee grounds

≪Co-financed by the European Regional Development Fund of the European Union and Greek national funds through the Operational Program Competitiveness, Entrepreneurship and Innovation, under the call RESEARCH - CREATE - INNOVATE (project code:T2EDK-02242)≫

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Thank you for your attention