



# Supercritical CO<sub>2</sub> extraction of plant biologically active compounds



Stela Jokić, PhD

PTF<sub>S</sub>

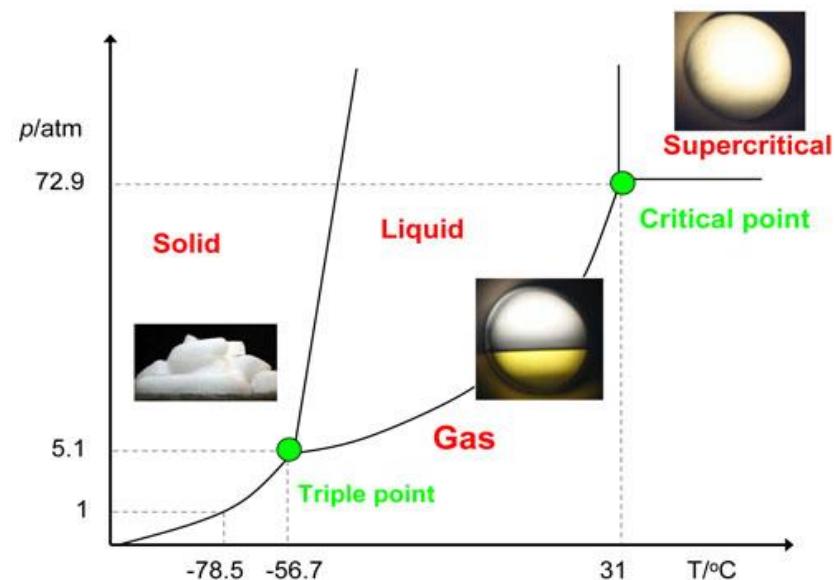
Šibenik, 11.04.2019.

# Supercritical fluid extraction (SFE)

- many advantages for natural plant materials

Gases	$T_c$ (K)	$P_c$ (MPa)
Carbon dioxide	304.17	7.38
Ethane	305.34	4.87
Methane	190.55	4.59
Ethylene	282.35	5.04
Propane	369.85	4.24
Nitrous oxide	309.15	7.28
Acetylene	308.70	6.24
Hydrogen	33.25	1.29
Nitrogen	126.24	3.39
Oxygen	154.58	5.04
Neon	44.40	2.65
Argon	150.66	4.86
Xenon	289.70	5.87

Critical Parameters of Different Solvents



Physical Parameters at Different Aggregation

	Density (g cm <sup>-3</sup> )	Diffusion coefficient (cm <sup>2</sup> s <sup>-1</sup> )	Viscosity (g cm <sup>-1</sup> s <sup>-1</sup> )
Gas	$10^{-4}$	0.1 – 0.4	$(1 - 3) \times 10^{-4}$
Liquid	0.6 – 1.6	$(0.2 - 2) \times 10^{-5}$	$(0.2 - 3) \times 10^{-2}$
Supercritical fluid	0.2 – 0.9	$(0.2-0.7) \times 10^{-3}$	$(1 - 9) \times 10^{-4}$



Contents lists available at [ScienceDirect](#)

## Food and Bioproducts Processing

journal homepage: [www.elsevier.com/locate/fbp](http://www.elsevier.com/locate/fbp)



# New perspective in extraction of plant biologically active compounds by green solvents



Marina Cuvjetko Bubalo<sup>a</sup>, Senka Vidović<sup>b</sup>, Ivana Radojčić Redovniković<sup>a,\*</sup>,  
Stela Jokić<sup>c</sup>

<sup>a</sup> University of Zagreb, Faculty of Food Technology and Biotechnology, 10000 Zagreb, Croatia

<sup>b</sup> University of Novi Sad, Faculty of Technology, Bulevar Cara Lazara 1, 21000 Novi Sad, Serbia

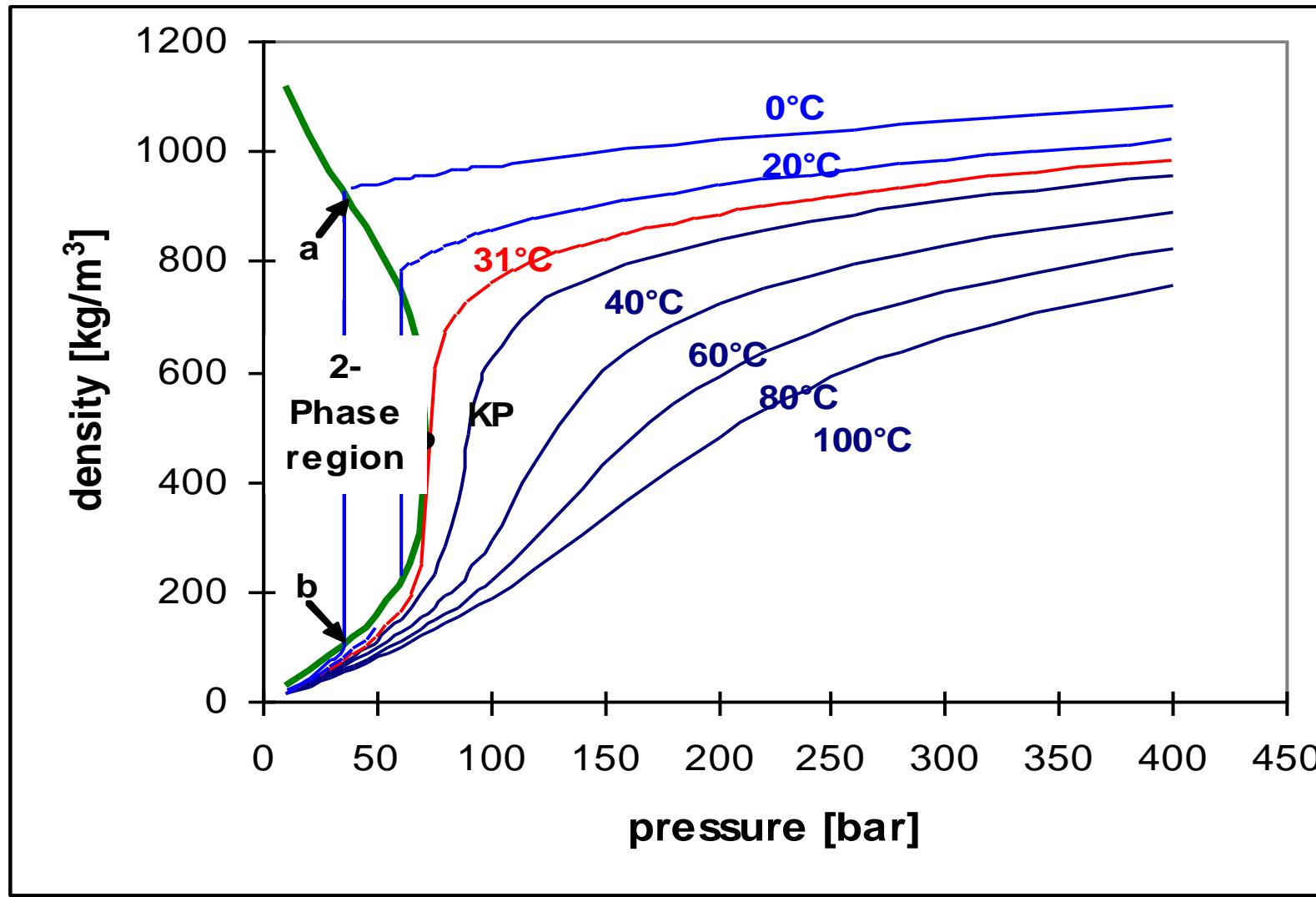
<sup>c</sup> University of Josip Juraj Strossmayer in Osijek, Faculty of Food Technology Osijek, Franje Kuhaca 20, 31000 Osijek, Croatia



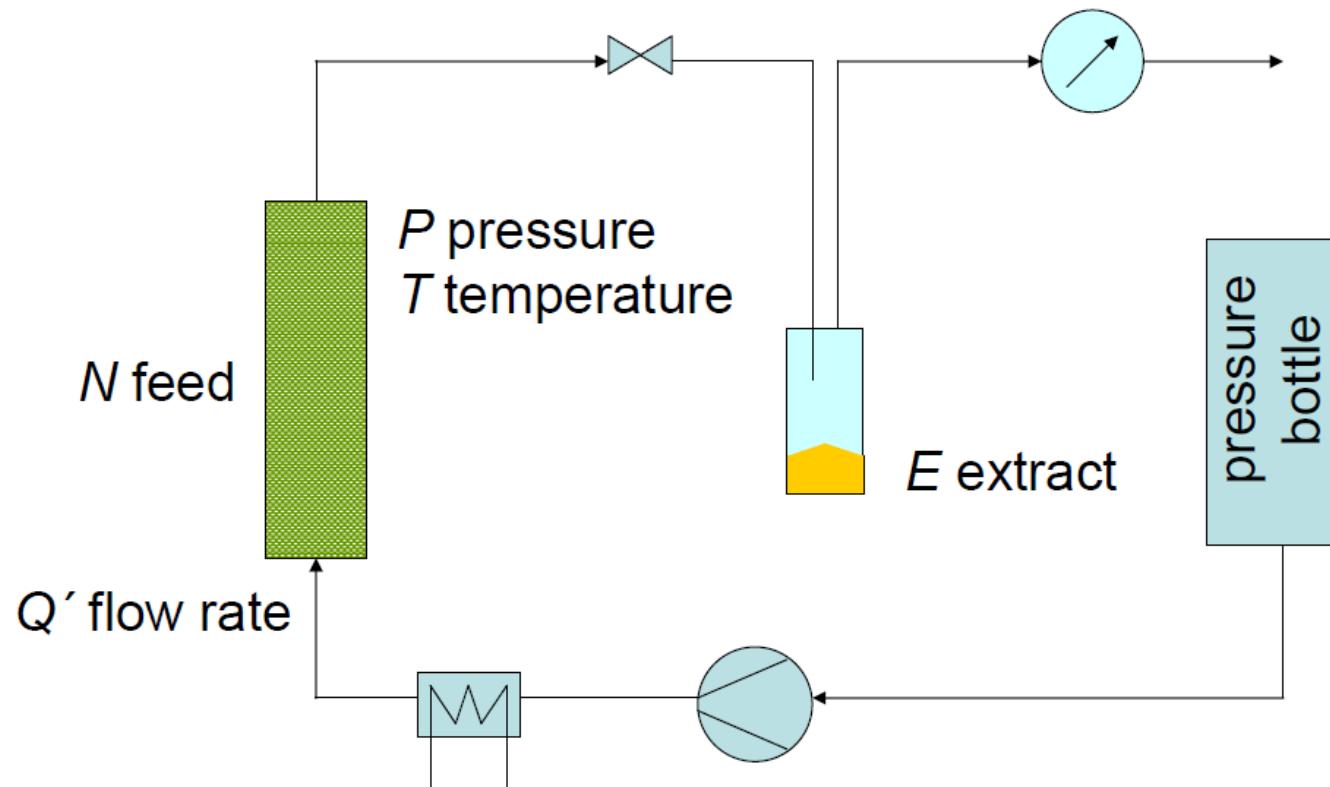
**Table 2 – Advantages and disadvantages of extraction processes.**

Extraction technique	Advantages	Drawbacks
SC-CO <sub>2</sub> extraction	<ul style="list-style-type: none"><li>Gentle treatment of heat-sensitive materials (its moderate critical temperature of 31.2 °C is a key issue for the preservation of bioactive compounds in extracts)</li><li>Solvent — free products</li><li>CO<sub>2</sub> as solvent does not cause environmental problems and is physiologically harmless, germicidal and not flammable.</li><li>CO<sub>2</sub> is a Generally recognized as safe (GRAS) solvent</li><li>CO<sub>2</sub> is inexpensive solvent</li><li>Due to low viscosity and relatively high diffusivity, supercritical CO<sub>2</sub> have enhanced transport properties than liquids, can diffuse easily through solid materials and can therefore give faster extraction rates.</li><li>Fragrances and aromas remain unchanged</li><li>Selective extraction and fractionated separation</li><li>Pure extracts by means of few process steps</li><li>Changeable solvating power (possibility of modifying the density of the fluid by changing its pressure and/or temperature)</li><li>High solubility for non/low polar substances (for example volatile compounds)</li><li>possibility of direct coupling with analytical chromatographic techniques such as gas chromatography (GC) or supercritical fluid chromatography (SFC)</li></ul>	<ul style="list-style-type: none"><li>High pressures</li><li>High investment cost (requires a careful business plan contemplating the cost/effective analysis of the desired compounds to be extracted)</li><li>Phase equilibrium of the solvent/solute system is complex, making design of extraction conditions difficult</li><li>High polar substances (sugars, amino acids, inorganic salts, proteins, ...) are insoluble</li><li>The use of high pressures leads to capital costs for plant, and operating costs may also be high so the number of commercial processes utilizing supercritical fluid extraction is relatively small, due mainly to the existence of more economical processes.</li></ul>

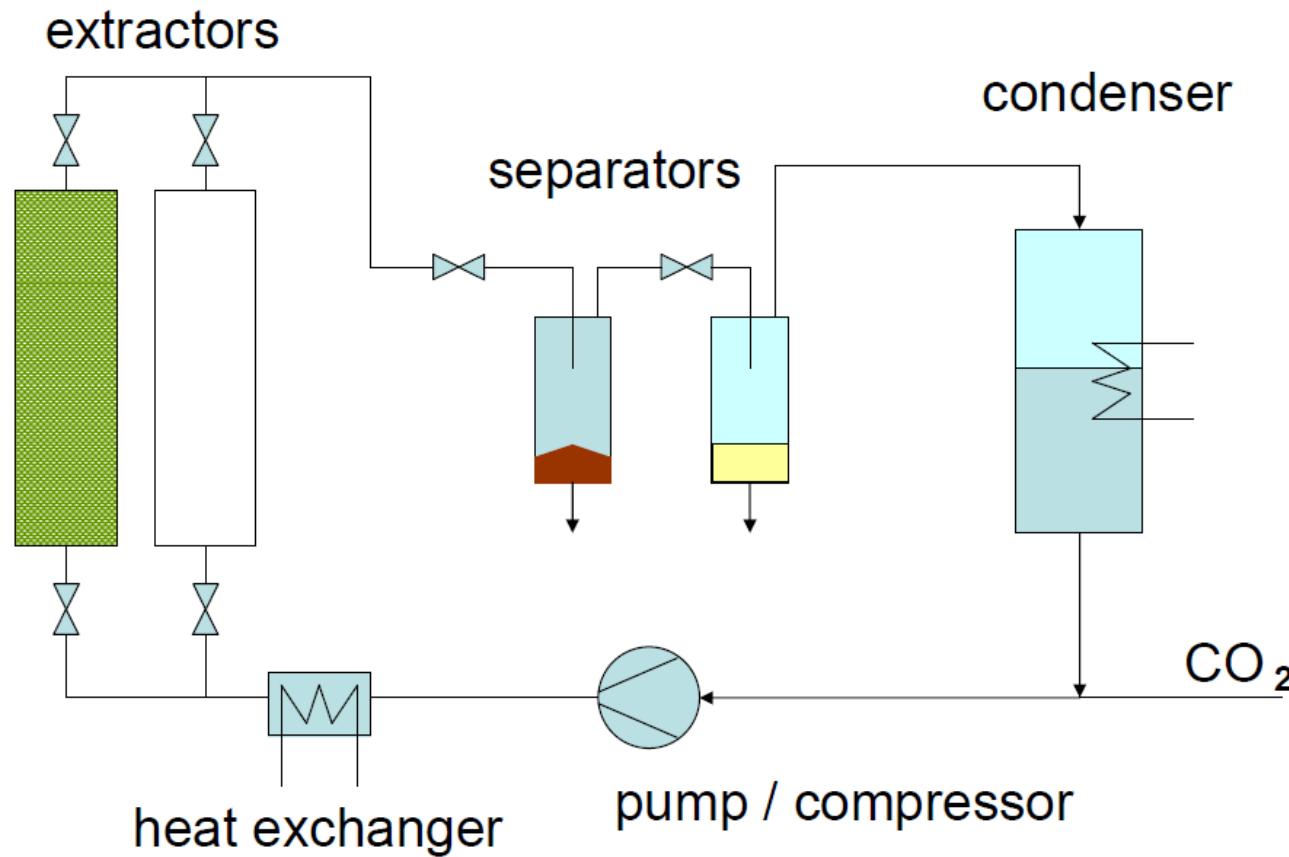
# Density Behaviour of CO<sub>2</sub>



## SFE from plants – laboratory



# SFE from plants - industry



**SUPERCritical CO<sub>2</sub> EXTRACTION PILOT PLANT DESIGN – TOWARDS IoT INTEGRATION****Goran Horvat, Krunoslav Aladić, Stela Jokić**

The interest in high pressure technology during last decades increased intensively. Supercritical Fluid Extraction (SFE) is a process that is growing in importance as an alternative to conventional separation processes. SFE uses environmentally friendly CO<sub>2</sub> as the extracting agent in the process because of its relatively low critical pressure (7.38 MPa), its low critical temperature (304 K), its non-dangerous character and low cost. During this process it is necessary to use high pressures in the procedure. The extractor vessel (pressure vessel) is the most important equipment of the system, where the supercritical conditions need to be established and the extraction occurs. Also other devices (separator vessel, heat exchangers, valves etc.) are necessary to be involved in the process due to used high pressures. Safety is the most important factor while dealing with SFE systems and the design of such equipment with full safety of process is very hard task. Therefore, to achieve the high desired safety level, a reliable control system must be designed as the control system and data communication segment. Various different process parameters such as CO<sub>2</sub> mass flow rate, extraction pressures and temperatures affect the extraction process and the quality of the extract; hence these parameters need to be precisely controlled and monitored during the extraction. A design of one supercritical CO<sub>2</sub> extraction laboratory-pilot plant and development of a remote control and its supervision system is presented in this paper. The developed SFE system (mechanical and electrical components) was compared with the existing commercial systems and its main advantages over the existing systems are presented. By enabling remote control and supervision the classical process control is joined with the concept of Internet of Things (IoT), where the information becomes omnipresent in the vast realm of Internet.

Preliminary communication



**Keywords:** embedded system; process control; supercritical fluid extraction; system construction.

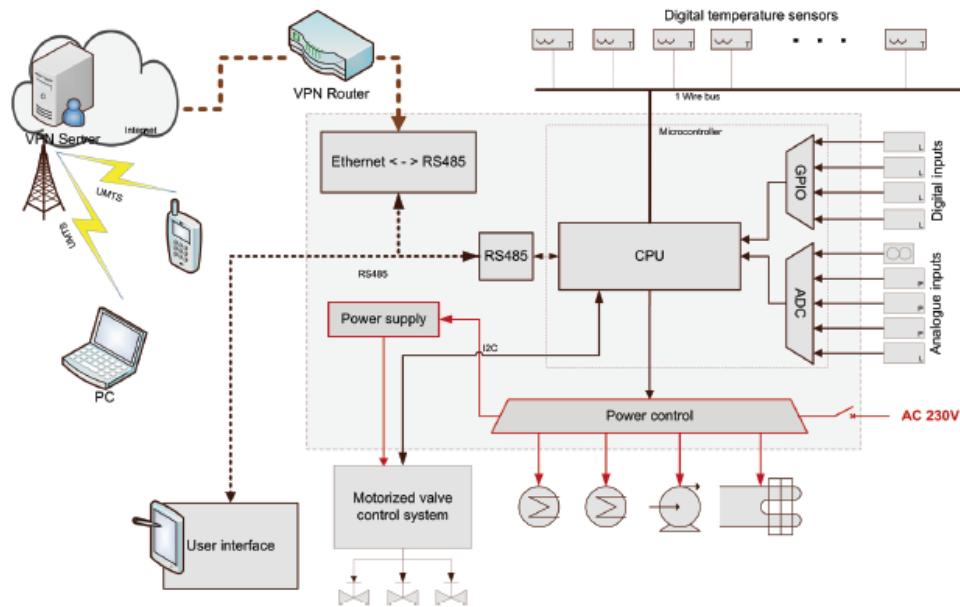


Figure 2 Block diagram of the supporting electronic system

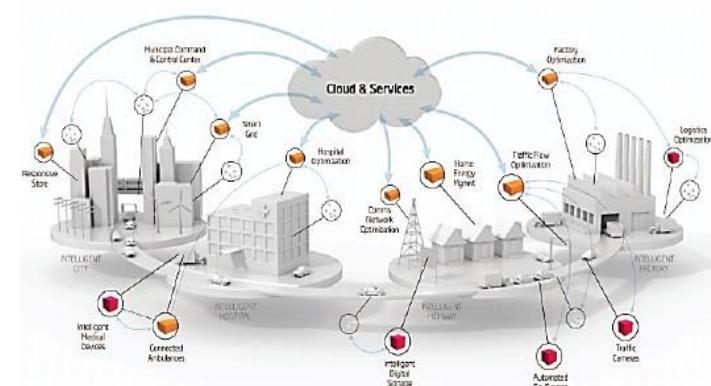
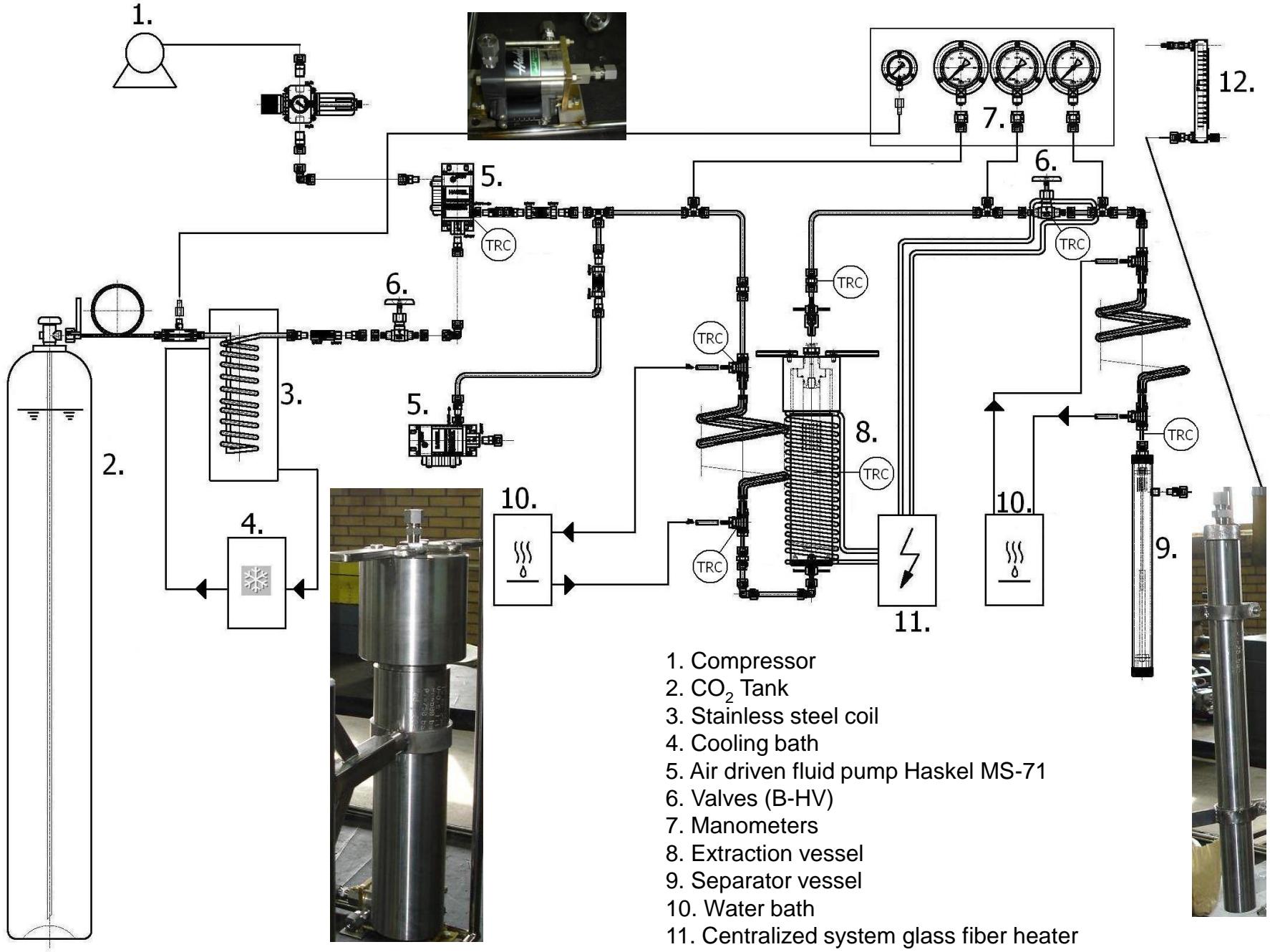
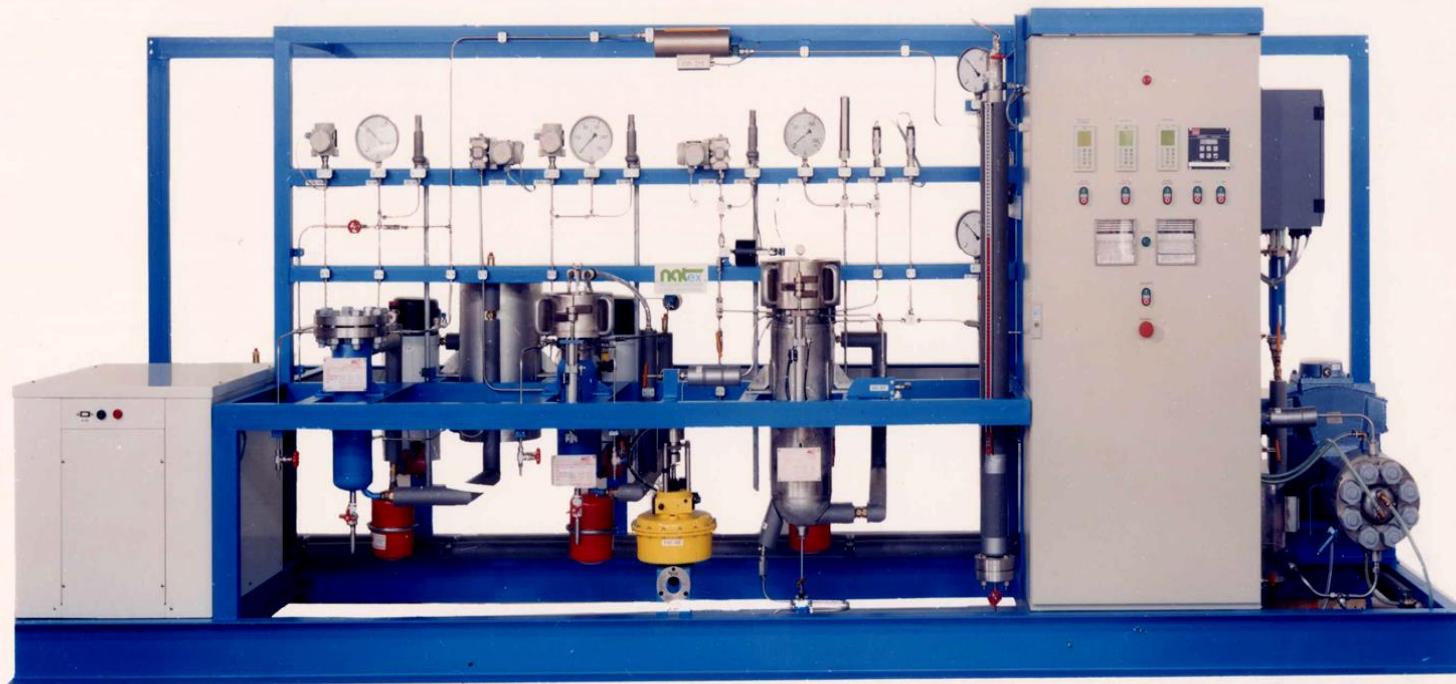


Figure 7 The concept of Internet of Things



1. Compressor
2. CO<sub>2</sub> Tank
3. Stainless steel coil
4. Cooling bath
5. Air driven fluid pump Haskel MS-71
6. Valves (B-HV)
7. Manometers
8. Extraction vessel
9. Separator vessel
10. Water bath
11. Centralized system glass fiber heater
12. Flow meter

## PRODUCT / PROCESS DEVELOPMENT & RESEARCH UNIT



**Extractor volume 5 litres, extraction pressure up to 1000 bar**

**High performance research unit for scientists and professionals,  
ready for "Plug & Work"**

# GERMANY Decaffeination of Tea



1988 turn-key, 3,000 t/annual

# ITALY Decaffeination of Coffee



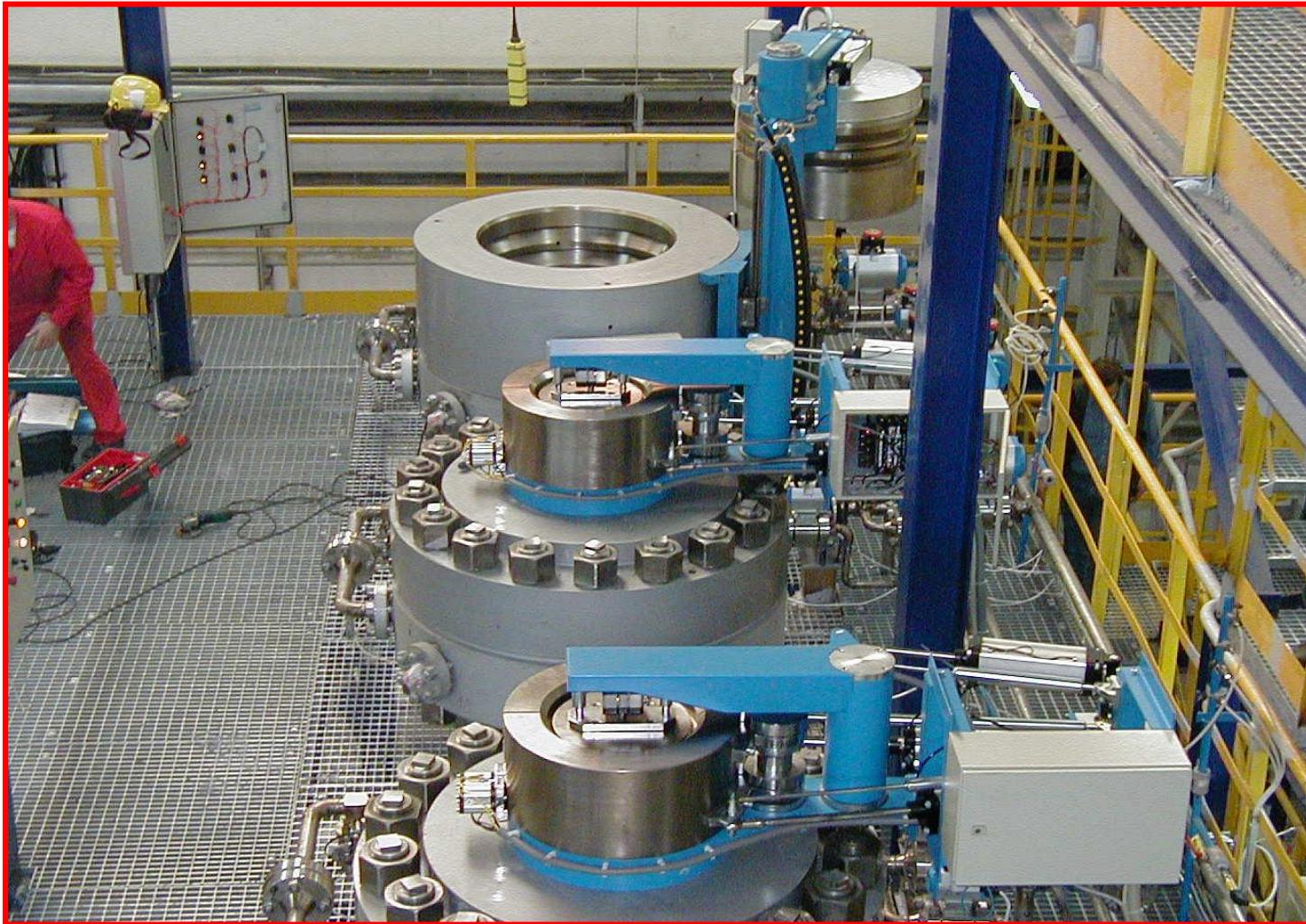
1992 Turn-key, 10,000 t/a

# TAIWAN Rice Treatment Plant



3 x 5,2 m<sup>3</sup>, 325 bar, Capacity 90 t per day

# POLAND Hops Extraction Plant



# NEW ZEALAND Extraction plant for hops and nutraceuticals



Multipurpose plant  
3 x 850 litres, 550 bar

# SOUTH KOREA Sesame Oil



3 x 2.500 litres  
550 bar

# Applications

Raw Material	Extract	Yield [%]	P [bar]	T [°C]	
<b>stimulants</b>					
beer	aroma		80 - 120	15 - 40	2
cocoa	theobromine	1.5	250 - 300	60 - 90	2
cocoa powder	cocoa butter and low fat powder	10 - 40	350 - 500	40 - 100	2
green coffee beans	decaffeinated coffee	0.8 - 2.5	220 - 320	60 - 90	1,2
tea	decaffeinated tea	2.8 - 3.8	250 - 300	45 - 75	1,2
tea leaves	aroma	1.8 - 2.4	100 - 300	50 - 60	2
tobacco leaves	total extract, aroma	2	100 - 300	20 - 80	1,2
wine	aroma, ethanol	10 - 12	80 - 120	15 - 40	1,2
<b>vegetable oils</b>					
currant seed	oil	26	250 - 300	20 - 40	2
ladies thistle seed	oil	32	250 - 300	40	2
palm oil distillate	tocopherol		220 - 270	40 - 80	2
pumpkin seed	oil	14	250 - 300	40	1,2
rape seed	oil	39.6	150 - 250	20-50	1
rape seed, press cake	oil	20 - 80	250 - 300	20 - 80	2
sesame seed, roasted	oil, flavour	43	250 - 450	20 - 80	2
sesame seed	oil	43	140	20 - 40	1
sunflower seed	oil	36	250 - 300	20 - 80	1,2
wheat germ	oil	13	250	40 - 60	1
amaranth	oil	5.2	250	40	1

# Applications

Raw Material	Extract	Yield [%]	P [bar]	T [°C]	
medical herbs					
arnica	total extract	5 - 6	150 - 300	20 - 60	1,2
calendula	oil	3.3	280	40 - 50	1
camomile	matricin, bisabolol	3.2	100 - 200	20 - 40	1
camomile	volatile oil, matricin	3 - 6	250 - 750	20 - 40	2
chrysanthemum	pyrethrines	3.2	60 - 150	20 - 50	1
chrysanthemum	pyrethrines	2 - 3	250 - 450	40 - 60	2
dandelion	total extract		100 - 400	25 - 60	1
eucalyptus	volatile oil, total extract	1.8 - 3	150 - 250	20 - 40	2
evening primrose	linolic acid	21 - 23	500 - 700	20 - 60	2
ginseng	pesticides		150 - 450	50 - 70	2
hayseed	total extract	1.2	200	20 - 40	2
marigold	total extract	7 - 9	150 - 600	20 - 50	2
parsley	volatile oil, total extract	13 - 18	450 - 500	60 - 70	2
peppermint leaf	volatile oil, total extract	2.5 - 3	150 - 250	20 - 40	2
peppermint leaf	menthol	2.8	100 - 130	25 - 40	1
ribwort leaf	total extract	2.2	150 - 250	20 - 40	2
St.Mary's thistle	total extract		100 - 400	25 - 60	1
valerian	total extract		100 - 400	25 - 60	1
wormwood leaf	total extract	2.7 - 3.5	150 - 250	20 - 40	2
yarrow leave	total extract	1.6	150 - 250	20 - 40	1,2

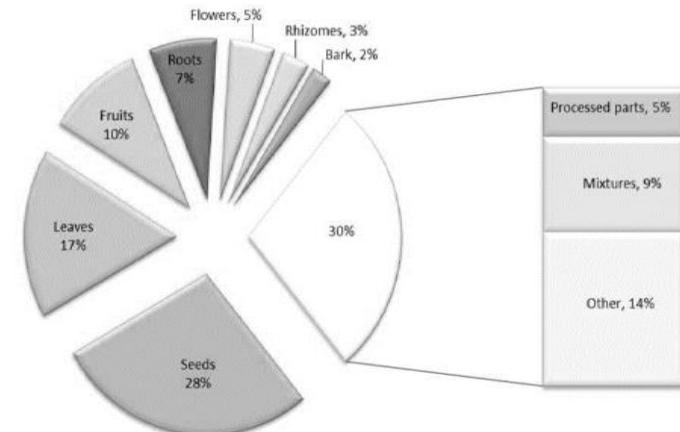
# Applications

Raw Material	Extract	Yield [%]	P [bar]	T [°C]	
spices, aromatics					
aniseed	total extract, volatile oil	8 - 10	150 - 300	20 - 40	1,2
caraway seed	total extract, volatile oil, carvone	7 - 18	150 - 700	20 - 60	1,2
cardamom	total extract	4 - 5.5	250 - 500	40 - 60	2
celery root	total extract	10	150 - 450	20 - 60	2
chilli	total extract, colour, capsaicin	3 - 12	400 - 500	50 - 70	2
cinnamon	eugenol	2.8	60-100	22 - 35	1
cinnamon bark	volatile oil	3	150 - 350	20 - 50	2
cinnamon leaf	total extract, volatile oil	3 - 4	150 - 250	20 - 40	2
clove	total extract	16 - 20	150 - 450	20 - 60	2
coriander	total extract, linalool	8.5 - 15	350 - 850	40 - 60	2
dill seed	total extract	1.5 - 2	500 - 700	20 - 60	2
fennel	fenchol	10 - 12	150 - 300	20 - 40	1,2
garlic	total extract	0.3 - 0.5	100 - 450	20 - 50	2
ginger	total extract	3 - 6	300 - 450	50 - 60	2
mace	total extract	20 - 38	250 - 450	50 - 60	2
marjoram green	total extract	1.2	150 - 700	20 - 70	2
mustard	total extract	12 - 18	450	60 - 80	2
nutmeg	total extract	25 - 30	350 - 500	60 - 80	2
onion dried	total extract	5 - 7	100 - 450	20 - 50	2
oregano	total extract	2 - 2.5	450 - 750	50 - 80	2
paprika sweet	total extract, aroma, pigments	6 - 15	100 - 700	20 - 80	2
peanut	aroma	1.5 - 2	150 - 450	40 - 80	2
pepper white	piperin	2.5 - 12	100 - 500	20 - 80	1,2

# Applications

Raw Material	Extract	Yield [%]	P [bar]	T [°C]	
<b>spices, aromatics</b>					
rosemary	total extract, antioxidants	5 - 8.5	350 - 800	50 - 90	2
sage	total extract, antioxidants		450 - 800	50 - 80	2
thyme	total extract, antioxidants		450 - 750	50 - 80	2
turmeric	total extract, curcumin	4 - 10	300 - 500	60 - 90	2
valerian roots	total extract	4	150 - 300	20 - 40	1,2
vanilla beans	total extract	3.5 - 8.5	100 - 450	20 - 60	2
wormwood	absinthin	3.5	100-150	25 - 40	1
<b>animal fat</b>					
animal body flour	animal fat	9 - 12	250 - 300	40	2
animal cadaver	fat	37.5	120	20	1
animal carcasses	animal fat	35 - 40	250 - 300	40	2
animal tankage	fat	9	120	20	1
pig brain	fat	30	100 - 300	25 - 40	1

Main families of compounds found in vegetable extracts obtained by supercritical fluids





## New perspective in extraction of plant biologically active compounds by green solvents

Marina Cvjetko Bubalo<sup>a</sup>, Senka Vidović<sup>b</sup>, Ivana Radojčić Redovniković<sup>a,\*</sup>, Stela Jokić<sup>c</sup>

**Table 4 – Supercritical CO<sub>2</sub> extraction of plant material (selected examples of plant origin).**

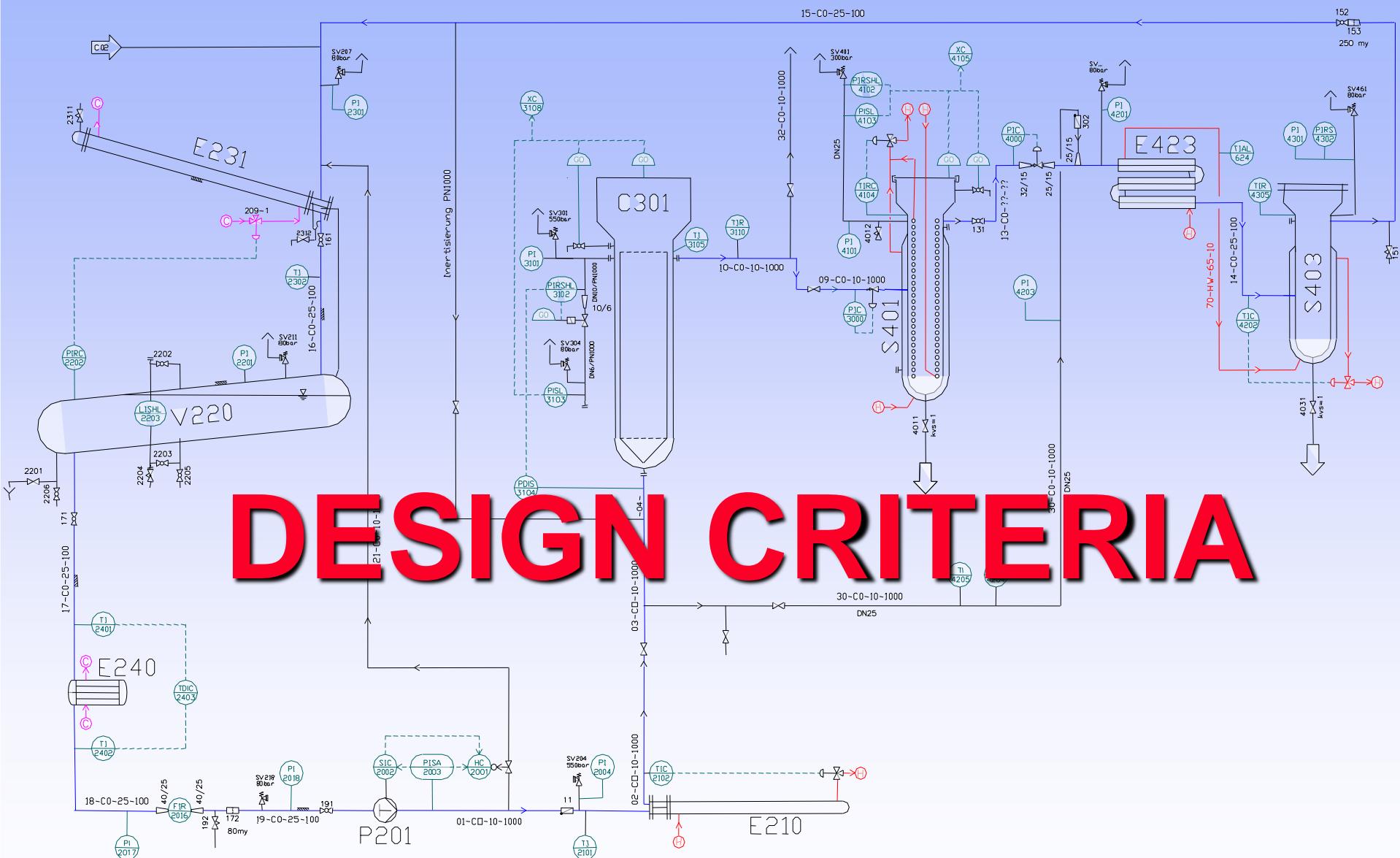
Material	Extract	Modifier	T (°C)	P (bar)	Reference
Almond	Oil, tocopherols		35–50	350–550	Leo et al. (2005)
Angelica root	Oil		40	120	Doneanu and Anitescu (1)
Aniseed	Essential oil		30	80–180	Rodrigues et al. (2003)
Annatto seed	Bixin		40–60	200–300	(Degnan et al. (1991))
Aloe vera leaves	α-Tocopherol	Methanol	32,41,50	350,400,450	Hu et al. (2005)
Apricot seed	Oil	Ethanol	40–70	300–600	Özkal et al. (2005)
Basil (leaves and flowers)	Total extract, linalool, eugenol, d-cadinene		40–60	100–300	Zeković et al. (2014)
Black pepper	Oleoresin, piperine		35–55	200–300	Dang and Phan (2014)
Borage seed	Oil		40–60	200–300	Kotnik et al. (2006)
Canola seed	Oil		40–60	200–250	Pedersetti et al. (2011)
Caraway seed	Carvone, limonene		32–75	75–300	Baysal and Starmans (199)
Carrots	β-Carotene		57	250	Subra et al. (1998)
Cashew nut shell	Oil		60	200–300	Patel et al. (2006)
Celery	Essential oil		40	100	Misić et al. (2008)
Chamomile	α-Bisabolol, chamazulene, matricine		30–40	100–250	Kotnik et al. (2007)
Cherry seed	Oil		40–60	180–220	Bernardo-Gil et al. (2001)
Chia seed	Oil		40–80	136–408	Uribe et al. (2011)
Chilli pepper	Capsaicinoids		40–80	100–400	Perva-Uzunalić et al. (2004)
Citrus peel	Naringin		60	95	Giannuzzo et al. (2003)
Cocoa beans	Methylxanthines (caffeine, theobromine)		70	200–400	Mohamed et al. (2002)
Cocoa shell	Theobromine		50–85	150–450	Rossi (1996)
Coffee husk	Caffeine, chlorogenic acid		40–60	100–300	Andrade et al. (2012)
Coriander seed	Oil		35	200–300	Illésa et al. (2000)
Corn germ	Oil		35–86	210–525	Rebolledo et al. (2012)
Cotton seed	Oil		60–80	350–550	Bhattacharjee et al. (2007)
Egg yolk	Phospholipids		40	517	Boselli and Caboni (2000)
Elderberry pomace	Anthocyanins	Ethanol, H <sub>2</sub> O	40	210	Seabra et al. (2010)
Eucalyptus	Essential oil		50	90	Della Porta et al. (1999)
Fennel	Essential oil, trans-anethole, fenchone		40	81	Simándi et al. (1999)
Flax seed	Oil		50–70	300–500	Özkal (2009)
Ginger	Oleoresin	Ethanol, isopropanol	25–35	200–250	Zancan et al. (2002)

			isopropanol		
Ginkgo leaves	Terpenes, flavonoids			60–110	242–312
Grape seed	Phenolic compounds	Methanol		35	455
Grape seed	Oil		40,50,60	200,300,400	Palma et al. (1999)
Grape skin	Resveratrol	Ethanol		40	150
Guarana seed	Caffeine	Water		40–70	Marti et al. (2001)
Hemp seed	Oil		40–60	300–400	Saldaña et al. (2002)
Hiprose	Total extract			35	250
Hop	Essential oil			40	150,300
Horsetail	Oleoresin			30,40	Illes et al. (1997)
Jojoba seed	Oil	Hexane		70–90	Aladić et al. (2015)
Lavender	Essential oil, monoterpenes, coumarin, herniarin			40–60	Salgin et al. (2004)
Lemongrass	Essential oil			23–50	Jerković et al. (2007)
Lovage	Essential oil			40–50	Carlson et al. (2001)
Marjoram leaves	Essential oil			40–50	Daukšas et al. (1999)
Marigold flowers	Total extract			20–40	Reverchon (1992)
Mate tea leaves	Caffeine, theophylline, theobromine			40–70	Campos et al. (2005)
Melon seed	Oil			40–80	Saldaña et al. (2000)
Neem seed	Nimbin			35–60	Nyam et al. (2011)
Nutmeg	Oil			23	Tonthubthimthong et al. (2001)
Olive pomace	Oil			40–50	Spricigo et al. (1999)
Orange peel	Essential oil			40	De Lucas et al., 2003
Oregano leaves	Flavonoids	Ethanol		100	Jerković et al. (2015)
Palm kernel	Oil			40–60	Cavero et al. (2006)
Paprika	Carotenoids, tocopherol, capsaicinoids			35–55	Zaidul et al. (2007)
Parsley seed	Oil			35–45	Cardoso De Oliveira et al. (2013)
Passion fruit seed	Oil			40–60	Mezzomo et al. (2010)
Peach seed	Oil	Ethanol		30–50	Bozan and Temelli (2003)
Poppy seed	Oil			50–70	

**Table 4 (Continued)**

Material	Extract	Modifier	T (°C)	P (bar)	Reference
Pumpkin seed	Oil		35–75	152–345	Mitra et al. (2009)
Rape seed	Oil		40–60	250–350	Yu et al. (2012)
Rice bran	γ-Oryzanol, tocols		40–60	276–414	Yoon et al. (2014)
Rosehip seed	Oil		40–80	150–450	Machmudah et al. (2007)
Rosemary	carnosic acid, carnosol		100	355	Tena et al. (1997)
Safflower seed	Oil		35–60	220–280	Han et al. (2009)
Sage	Carnosolic acid	Ethanol	100	250,350	Dauksas et al. (2001)
Sea buckthorn	Tocopherols, lycopene, β-carotene	Methanol, ethanol, 2-propanol	35–55	150–350	Kagliwal et al. (2011)
Sesame seeds	Oil		40–60	190–250	Corso et al. (2010)
Soybean seeds	Oil		40–60	300–500	Jokić et al. (2012)
Savory	Total extract, carvacrol		40–60	100–350	Vladić et al. (2016)
Thyme	Total extract, thymol, carvacrol		40	80–400	Zeković et al. (2000)
Tomato skin and seed	Carotenoids, tocopherols, sitosterols		40–80	300–460	Vagi et al. (2007)
Walnut	Oil	Ethanol	40–60	300–500	Salgin and Salgin (2006)
Wheat germ	Total extract, phenolics, tocopherols		40–60	148–602	Gelmez et al. (2009)
Yarrow	Essential oil		40–60	100	Bocevska and Sovová (2007)

# DESIGN CRITERIA



# Design Criteria

## Influence of

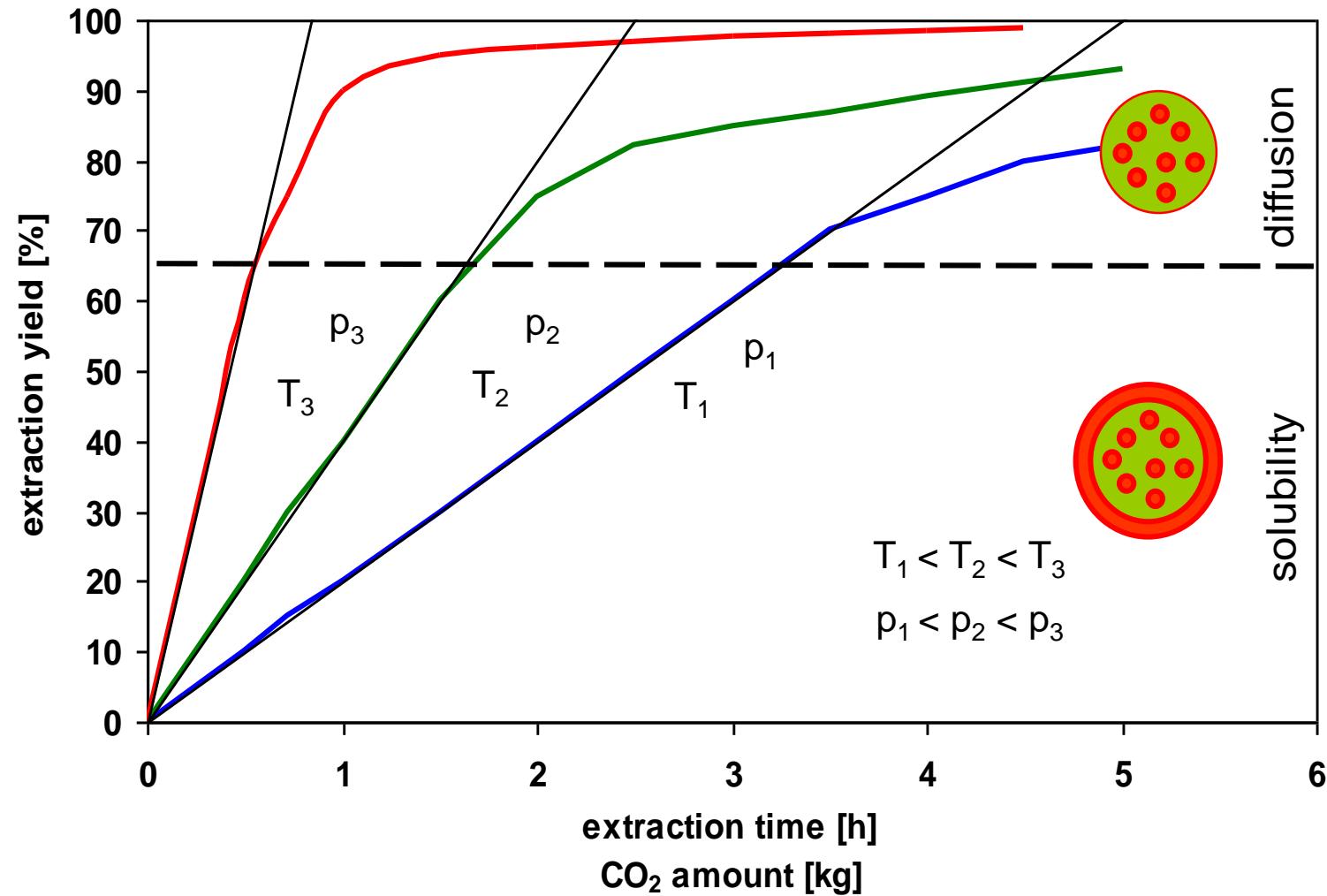
- raw material
  - pressure
  - temperature }      effect on **solubility**
  - particle size
  - ( $\text{CO}_2$  flow rate) → very often limited for Lab Scale Plants
    - > pressure → > solubility because of > fluid density
    - > temperature → 2 effects
      - > vapor pressure
      - < density of fluid
  - < particle size → shorter diffusion ways
    - breaking of shells
    - breaking cell walls
- optimal particle sizes: 0,4 – 0,8 mm**

**Lab Scale Plants**

# Modelling of Supercritical Fluid Extraction

- Before the installation of a plant, the modelling of the process is indispensable.
- The SFE curve modelling is important for process optimization and scale-up, helping the identification of extractor volume and extraction yield.
- The optimal process parameters (extraction time, solvent flow, pressure, temperature, particle size etc.) can be obtained from the model.
- For the mathematical description of the extraction curves different models can be used (the most commonly used model is Sovová model).

# Typical trend of extraction lines





ELSEVIER

Contents lists available at SciVerse ScienceDirect

## Food and Bioproducts Processing

journal homepage: [www.elsevier.com/locate/fbp](http://www.elsevier.com/locate/fbp)

|ChemE

# Effects of supercritical CO<sub>2</sub> extraction parameters on soybean oil yield

Stela Jokić<sup>a,\*</sup>, Bence Nagy<sup>b</sup>, Zoran Zeković<sup>c</sup>, Senka Vidović<sup>c</sup>, Mate Bilić<sup>a</sup>, Darko Velić<sup>a</sup>, Béla Simándi<sup>b</sup>

<sup>a</sup> Faculty of Food Technology, Osijek, F. Kuhaca 20, 31000 Osijek, Croatia

<sup>b</sup> Department of Chemical and Environmental Process Engineering, Budapest University of Technology and Economics, H-1521 Budapest, Hungary

<sup>c</sup> Faculty of Technology, Novi Sad, Bulevar Cara Lazara 1, 21000 Novi Sad, Serbia

# Sovová's model<sup>1</sup>

Sovová's model describes the yield in function of the time using 3 equations:

$$\frac{Y}{x_0} = \vartheta [1 - \exp(-Q)] \quad \vartheta < \frac{q}{Q}$$

$$\frac{Y}{x_0} = \vartheta - \frac{q}{Q} \exp[Q(Z_k - 1)] \quad \frac{q}{Q} \leq \vartheta < \vartheta_k$$

$$\frac{Y}{x_0} = 1 - \frac{1}{S} \ln \left\{ 1 + [\exp(S) - 1] \exp \left[ S \left( \frac{q}{Q} - \vartheta \right) \right] (1 - q) \right\} \quad \vartheta \geq \vartheta_k$$

$Q, S$  dimensionless model parameters [-]

$\tau$  minimal extraction time [s]

$q$  soluble material fraction on the surface of the particles [kg/kg]

$\vartheta = \frac{t}{\tau}$  dimensionless time [-]

$Z_k, \vartheta_k$  dimensionless length and time where and when the soluble material runs out from the surface [-]

# Parameters of Sovová's model

$$\vartheta_k = \frac{q}{Q} + \frac{1}{S} \ln \left\{ 1 - q [1 - \exp(S)] \right\} \quad Z_k = \frac{1}{S} \ln \left[ 1 + \frac{1}{q} \left\{ \exp \left[ S \left( \vartheta - \frac{q}{Q} \right) \right] - 1 \right\} \right]$$

$$\tau = \frac{m_s x_0}{\dot{m}_f y^*} \quad Q = \frac{m_s k_f a_p \rho_f}{\dot{m}_f (1 - \varepsilon) \rho_s} \quad S = \frac{m_s k_s a_p x_0}{\dot{m}_f (1 - \varepsilon) y^*}$$

$y^*$	solvability at extraction pressure and temperature	[kg/kg]
$x_o$	initial concentration of soluble material	[kg/kg]
$\rho_f \rho_s$	density of the fluid and solid phase	[kg/m <sup>3</sup> ]
$a_p$	specific surface area of the solid particles	[m <sup>2</sup> /m <sup>3</sup> ]
$k_f \ k_s$	mass transfer coefficient in the fluid and the solid phase	[m/s]
$m_s$	amount of the raw material	[kg]
$\dot{m}_f$	mass flow of the fluid	[kg/s]
$\varepsilon$	void fraction in the bed	[m <sup>3</sup> /m <sup>3</sup> ]

# Parameters of Sovová's model

$$\vartheta_k = \frac{q}{Q} + \frac{1}{S} \ln \left\{ 1 - q [1 - \exp(S)] \right\} \quad Z_k = \frac{1}{S} \ln \left[ 1 + \frac{1}{q} \left\{ \exp \left[ S \left( \vartheta - \frac{q}{Q} \right) \right] - 1 \right\} \right]$$

$$\tau = \frac{m_s x_0}{\dot{m}_f y^*} \quad Q = \frac{m_s k_f a_p \rho_f}{\dot{m}_f (1 - \varepsilon) \rho_s} \quad S = \frac{m_s k_s a_p x_0}{\dot{m}_f (1 - \varepsilon) y^*}$$

$y^*$	solvability at extraction pressure and temperature	[kg/kg]
$x_0$	initial concentration of soluble material	[kg/kg]
$\rho_f$ $\rho_s$	density of the fluid	
$a_p$	specific surface	
$k_f$ $k_s$	mass transfer coefficient	
$m_s$	amount of the raw material	[kg]
$\dot{m}_f$	mass flow of the fluid	[kg/s]
$\varepsilon$	void fraction in the bed	[m <sup>3</sup> /m <sup>3</sup> ]

Solvability was estimated from an empirical equation  
or  
from the initial slope of the extraction curves

# Parameters of Sovová's model

$$\vartheta_k = \frac{q}{Q} + \frac{1}{S} \ln \left\{ 1 - q [1 - \exp(S)] \right\} \quad Z_k = \frac{1}{S} \ln \left[ 1 + \frac{1}{q} \left\{ \exp \left[ S \left( \vartheta - \frac{q}{Q} \right) \right] - 1 \right\} \right]$$

$$\tau = \frac{m_s x_0}{\dot{m}_f y^*} \quad Q = \frac{m_s k_f a_p \rho_f}{\dot{m}_f (1 - \varepsilon) \rho_s} \quad S = \frac{m_s k_s a_p x_0}{\dot{m}_f (1 - \varepsilon) y^*}$$

$y^*$	solvability at extraction pressure and temperature	[kg/kg]
$x_o$	initial concentration of soluble material	[kg/kg]
$\rho_f, \rho_s$	density of the fluid and solid phase	[kg/m <sup>3</sup> ]
$a_p$	The initial concentration of soluble material was estimated by traditional solvent Soxhlet-extraction using <i>n</i> -hexane as solvent.	[m <sup>2</sup> /m <sup>3</sup> ]
$k_f$	phase	[m/s]
$m$		[kg]
$\dot{m}_f$	mass flow of the fluid	[kg/s]
$\varepsilon$	void fraction in the bed	[m <sup>3</sup> /m <sup>3</sup> ]

# Parameters of Sovová's model

$$\vartheta_k = \frac{q}{Q} + \frac{1}{S} \ln \left\{ 1 - q [1 - \exp(S)] \right\} \quad Z_k = \frac{1}{S} \ln \left[ 1 + \frac{1}{q} \left\{ \exp \left[ S \left( \vartheta - \frac{q}{Q} \right) \right] - 1 \right\} \right]$$

$$\tau = \frac{m_s x_0}{\dot{m}_f y^*} \quad Q = \frac{m_s k_f a_p \rho_f}{\dot{m}_f (1 - \varepsilon) \rho_s} \quad S = \frac{m_s k_s a_p x_0}{\dot{m}_f (1 - \varepsilon) y^*}$$

$y^*$	solvability at extraction pressure and temperature	[kg/kg]
$x_o$	initial concentration of soluble material	[kg/kg]
$\rho_f \rho_s$	density of the fluid and solid phase	[kg/m <sup>3</sup> ]
$a_p$	specific surface area of the solid particles	[m <sup>2</sup> /m <sup>3</sup> ]
$k_f \ k_s$	mass transfer coefficient	
$m_s$	amount of the raw material	
$\dot{m}_f$	mass flow of the fluid	
$\varepsilon$	void fraction in the bed	[m <sup>3</sup> /m <sup>3</sup> ]

The density of the carbon dioxide was calculated from the Bender equation of state at the pressure and temperature of the extraction.

# Parameters of Sovová's model

$$\vartheta_k = \frac{q}{Q} + \frac{1}{S} \ln \left\{ 1 - q [1 - \exp(S)] \right\} \quad Z_k = \frac{1}{S} \ln \left[ 1 + \frac{1}{q} \left\{ \exp \left[ S \left( \vartheta - \frac{q}{Q} \right) \right] - 1 \right\} \right]$$

$$\tau = \frac{m_s x_0}{\dot{m}_f y^*} \quad Q = \frac{m_s k_f a_p \rho_f}{\dot{m}_f (1 - \varepsilon) \rho_s} \quad S = \frac{m_s k_s a_p x_0}{\dot{m}_f (1 - \varepsilon) y^*}$$

$y^*$	solvability at extraction pressure and temperature	[kg/kg]
$x_o$	initial concentration of soluble material	[kg/kg]
$\rho_f$ $\rho_s$	density of the fluid and solid phase	[kg/m <sup>3</sup> ]
$a_p$	specific surface area of the solid particles	[m <sup>2</sup> /m <sup>3</sup> ]
$k_f$ $k_s$		[m/s]
$m_s$		[kg]
$\dot{m}_f$		[kg/s]
$\varepsilon$	void fraction in the bed	[m <sup>3</sup> /m <sup>3</sup> ]

The density of the raw material was measured by  
Hofsäss air pycnometer.

# Parameters of Sovová's model

$$\vartheta_k = \frac{q}{Q} + \frac{1}{S} \ln \left\{ 1 - q [1 - \exp(S)] \right\} \quad Z_k = \frac{1}{S} \ln \left[ 1 + \frac{1}{q} \left\{ \exp \left[ S \left( \vartheta - \frac{q}{Q} \right) \right] - 1 \right\} \right]$$

$$\tau = \frac{m_s x_0}{\dot{m}_f y^*} \quad Q = \frac{m_s k_f a_p \rho_f}{\dot{m}_f (1 - \varepsilon) \rho_s} \quad S = \frac{m_s k_s a_p x_0}{\dot{m}_f (1 - \varepsilon) y^*}$$

$y^*$	solubility at extraction pressure and temperature	[kg/kg]
$x_0$	initial concentration of soluble material	[kg/kg]
$\rho_f \rho_s$	density of the fluid and solid phase	[kg/m <sup>3</sup> ]
$a_p$	specific surface area of the solid particles	[m <sup>2</sup> /m <sup>3</sup> ]

$k_f \ k_s$  mass transfer coefficient in the fluid and the solid phase [m/s]

$m_s$

$\dot{m}_f$

$\varepsilon$

The specific surface area was estimated from the surface-volume diameter of the particles, what was determined from sieve experiments using the Rosin–Rammler–Bennett particle size distribution model.

# Parameters of Sovová's model

$$\vartheta_k = \frac{q}{Q} + \frac{1}{S} \ln \left\{ 1 - q [1 - \exp(S)] \right\} \quad Z_k = \frac{1}{S} \ln \left[ 1 + \frac{1}{q} \left\{ \exp \left[ S \left( \vartheta - \frac{q}{Q} \right) \right] - 1 \right\} \right]$$

$$\tau = \frac{m_s x_0}{\dot{m}_f y^*} \quad Q = \frac{m_s k_f a_p \rho_f}{\dot{m}_f (1 - \varepsilon) \rho_s} \quad S = \frac{m_s k_s a_p x_0}{\dot{m}_f (1 - \varepsilon) y^*}$$

$y^*$   
 $x_o$   
 $\rho_f$   
 $a_p$   
 $k_f$     $k_s$   
 $m_s$   
 $\dot{m}_f$   
 $\varepsilon$

The mass transfer coefficient in the fluid phase was estimated from a Sherwood correlation.

[kg/kg]  
[kg/kg]  
[kg/m<sup>3</sup>]  
[m<sup>2</sup>/m<sup>3</sup>]  
[m/s]  
[kg]  
[kg/s]  
[m<sup>3</sup>/m<sup>3</sup>]

# Parameters of Sovová's model

$$\vartheta_k = \frac{q}{Q} + \frac{1}{S} \ln \left\{ 1 - q [1 - \exp(S)] \right\} \quad Z_k = \frac{1}{S} \ln \left[ 1 + \frac{1}{q} \left\{ \exp \left[ S \left( \vartheta - \frac{q}{Q} \right) \right] - 1 \right\} \right]$$

$$\tau = \frac{m_s x_0}{\dot{m}_f y^*} \quad Q = \frac{m_s k_f a_p \rho_f}{\dot{m}_f (1 - \varepsilon) \rho_s} \quad S = \frac{m_s k_s a_p x_0}{\dot{m}_f (1 - \varepsilon) y^*}$$

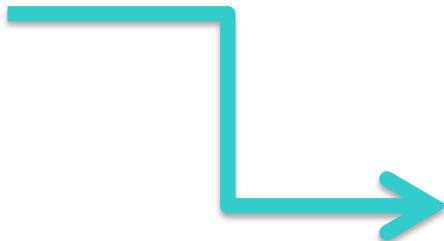
$y^*$	solvability at extraction pressure and temperature	[kg/kg]
$x_o$	initial concentration of soluble material	[kg/kg]
$\rho_f \rho_s$	density of the fluid and solid phase	[kg/m <sup>3</sup> ]
$a_p$	specific surface	
$k_f \ k_s$	mass transfer coefficient	
$m_s$	amount of the raw material	
$\dot{m}_f$	mass flow of the fluid	[kg/s]
$\varepsilon$	void fraction in the bed	[m <sup>3</sup> /m <sup>3</sup> ]

The void fraction in the bed was calculated from the density of the raw material and the bulk density.

# Scale up

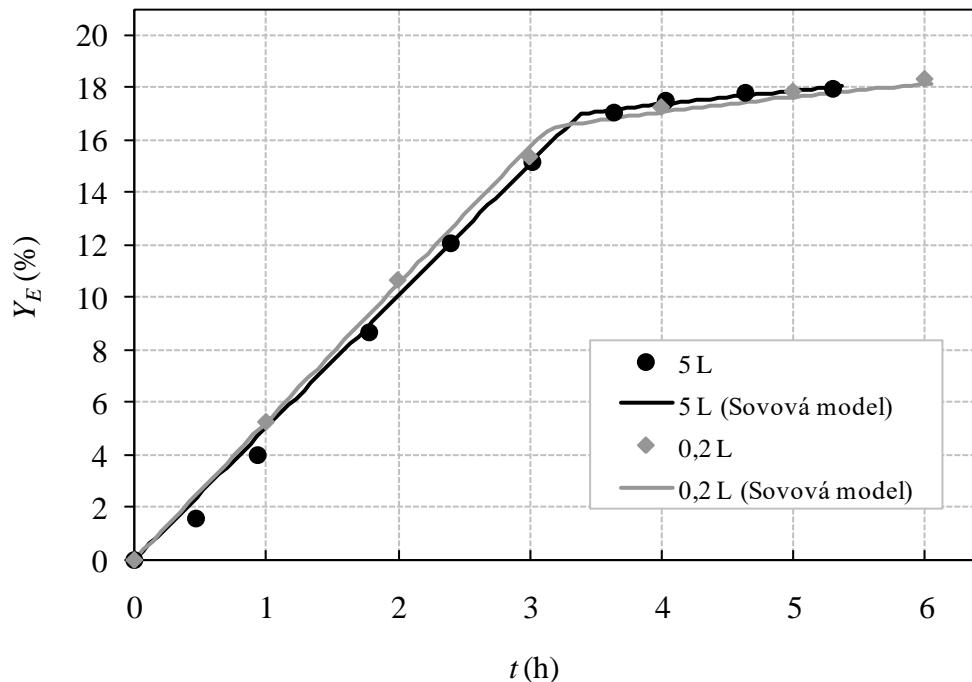
No.	$m_s$	$\dot{m}_f$	$k_f$	$\tau$	$Q$	$S$	$q$	$k_s$
	kg	kg/h	m/s	s	-	-	kg/kg	m/s
0.2 L								
5 L								

**PROPOSALS**



$$\frac{H_{\text{packed column}}}{D_{\text{packed column}}} = \text{const.}$$

$$\frac{\dot{m}_f}{m_s} = \text{const.}$$

**Table 2 – Parameters used and calculated values obtained by lab scale and pilot scale apparatus.**

Experiment <sup>a</sup>	$m_s$ (kg)	$\dot{m}_f$ (kg/h)	$k_f$ (m/s)	$\tau$ (s)	Q	S	$q$ (kg/kg)	$k_s$ (m/s)	AARD %
0.2 L	0.130	0.436	$3.61 \times 10^{-6}$	13,756	57.50	0.874	0.752	$2.92 \times 10^{-9}$	1.9
5 L	1.914	6.413	$1.38 \times 10^{-5}$	14,366	220.8	0.865	0.787	$2.77 \times 10^{-9}$	8.3

<sup>a</sup> Extraction conditions:  $P_E = 400$  bar,  $T_E = 40^\circ\text{C}$ ,  $d_0 = 0.383$  mm.

# Scale-up Procedure

## Challenges in SFE technology:

- Feasible designs for scale-up are necessary for commercialization;
- Modeling for scale-up not always available;
- Continuing instrumental and equipment improvements;
- Continuing research and development for applications and new material design.

## Geometric proposal:

$$\frac{h_{\text{packed column}}}{d_{\text{packed column}}} = \text{constant}$$

## Problems in SFE technology:

- The literature on scaling-up of SFE processes is limited to mathematical expressions for a preliminary estimation of both investment and operating costs;
- One of the problems found in scale-up studies is the use of small vessels for determining extraction curves, which influences the results because of the extract loss in the tubes walls of the equipment.

# Scale-up Procedure

Scale-up assays can be performed according to four proposals presented by Clavier and Perrut (2004):

**Proposal 1:** for processes where the solubility is the limiting kinetics mechanism, the ratio solvent mass/raw material mass should be maintain constant between small and large scales as given in following equation (Eq. 1):

$$\frac{\dot{m}_f}{m_s} = \text{constant} \quad (1)$$

**Proposal 2:** for processes where the diffusion mechanism (particularly internal diffusion) controls the extraction, the ratio CO<sub>2</sub> flow rate/mass of raw material as given (Eq. 2):

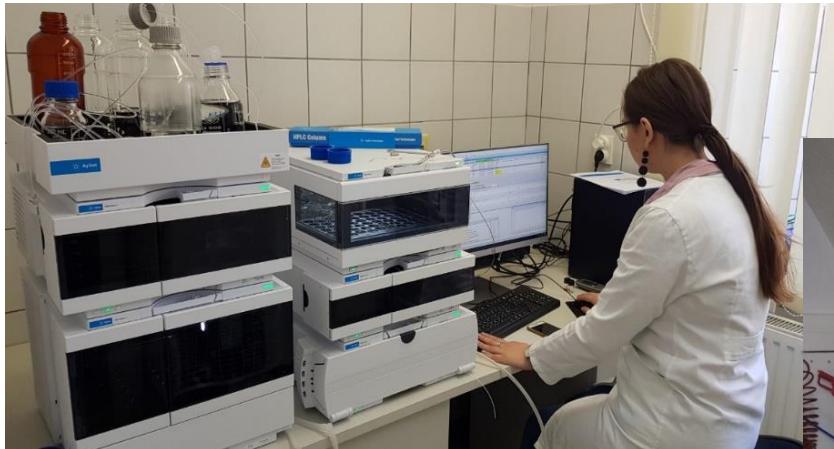
$$\frac{Q_f}{m_s} = \text{const.} \quad (2)$$

**Proposal 3:** for processes where diffusion and solubility are limiting mechanisms, both ratios (Eq. 1 and Eq. 2) should be maintained constant between small and large scales.

**Proposal 4:** three factors should be maintained constant between small and large scale: solvent mass/raw material mass, CO<sub>2</sub> flow rate/mass of raw material and the *Reynolds* number (*Re*).

# *EXAMPLES at*

PTF  
OS





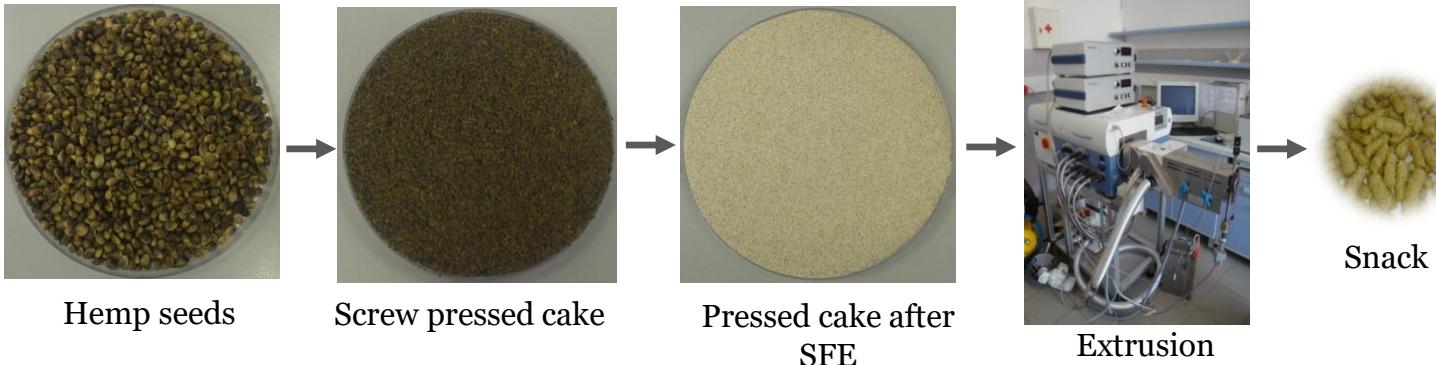
*Cannabis sativa* L.  
(Fedora 17 type)

Supercritical CO<sub>2</sub> extraction of hemp (*Cannabis sativa* L.) seed oil

Krunoslav Aladić<sup>a</sup>, Kristjan Jarni<sup>b</sup>, Tina Barbir<sup>c</sup>, Senka Vidović<sup>d</sup>, Jelena Vladić<sup>d,\*</sup>, Mate Bilić<sup>e</sup>, Stela Jokić<sup>e</sup>



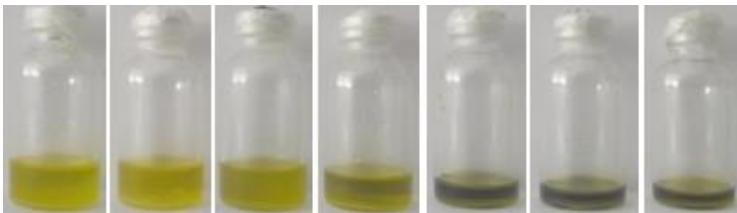
## „Zero waste” approach



- RESEARCH:



SFE



Collected hemp seed oil during the SFE process

- RESEARCH:



Screw pressed cake (nozzle ID 12, 9 and 6 mm)

SFE



Cake residual oil



HRZZ project: Application of food industry by-products in development of functional and environmentally friendly extruded food products and additives (2014-2018)



SFE production of edible and essential oils:



Plant extracts:



# *Helichrysum italicum* L.



No.	Compound	RI	%
1	4-Methylhexan-3-one	< 900	0.1
2	Nonane	900	0.1
3	$\alpha$ -Pinene	940	13.7
4	$\alpha$ -Fenchene	953	0.5
5	$\beta$ -Pinene	982	0.6
6	$\alpha$ -Terpinene	1022	0.6
7	Limonene	1034	3.1
8	Isobutyl 2-methylbut-2-enoate (Isobutyl angelate)**	1055	0.3
9	$\gamma$ -Terpinene	1064	0.6
10	$\alpha$ -Terpinolene	1092	0.2
11	Linalool	1105	0.7
12	Isopropyl 2-methylbut-2-enoate**	1158	0.8
13	Terpinene-4-ol	1184	0.4
14	$\alpha$ -Terpineol	1197	0.2
15	Decanal	1209	0.1
16	Nerol	1241	0.5
17	Linalyl acetate	1262	0.1
18	(Z)-hex-3-enyl tiglate	1282	0.1
19	Hexyl 3-methylbut-2-enoate (Hexyl angelate)**	1289	0.4
20	Undecan-2-one	1297	0.1
21	Neryl acetate	1369	5.4
22	$\alpha$ -Ylangene	1375	0.2
23	$\alpha$ -Copaene	1380	2.9
24	Italicene	1407	4.5
25	cis- $\alpha$ -Bergamotene	1419	1.2
26	trans-Caryophyllene	1424	4.7
27	trans- $\alpha$ -Bergamotene	1440	1.1
28	Geranyl propionate	1479	1.9
29	$\gamma$ -Selinene	1479	2.4
30	$\gamma$ -Curcumene	1485	23.2
31	$\beta$ -Selinene	1492	9.9
32	$\alpha$ -Selinene	1499	6.7
33	$\delta$ -Cadinene	1528	1.7
34	Nerolidol	1570	0.3
35	Guaiol	1603	0.8
36	$\alpha$ -Gurjunene	1640	0.7
37	$\alpha$ -Cadinol	1650	0.2
38	7-Epi-Amiteol	1665	1.3
39	$\alpha$ -Bisabolol	1691	0.4
40	Hexahydrofarnesyl acetone	1849	0.1
<b>Total identified</b>			92.8%

\*\* - correct isomer is not identified, RI – retention indices determined relative to *n*-alkanes (C<sub>9</sub>-C<sub>25</sub>), % - the chromatogram area percentages

Separation Science and Technology



ISSN: 0149-6395 (Print) 1520-5754 (Online) Journal homepage: <http://www.tandfonline.com/loi/sst20>

Optimization of supercritical CO<sub>2</sub> extraction of dried *Helichrysum italicum* flowers by response surface methodology: GC-MS profiles of the extracts and essential oil

Igor Jerković, Marina Rajić, Zvonimir Marijanović, Mate Bilić & Stela Jokić

# HELICHRYSUM ITALICUM L.

No.	Compound	RI	Run 4 7.93 MPa 50 °C [%]	Run 11 10 MPa 60 °C [%]	Run 8 15 MPa 35.86 °C [%]	Run 1 15.00 MPa 50 °C [%]	Run 3 20 MPa 40°C [%]	Run 12 22.07 MPa 50 °C [%]
1	2-Methylbutanoic acid	<900	-	0.1	-	0.1	0.1	-
2	Nonane	<900	-	-	-	0.1	-	-
3	α-Pinene	940	t	-	t	0.1	-	-
4	Hexanoic acid	990	-	-	-	0.1	-	-
5	α-Terpinene	1022	t	-	-	t	0.1	-
6	p-Cymene	1030	-	-	-	0.1	-	-
7	β-Phellandrene	1035	-	-	-	-	0.1	-
8	Limonene	1034	-	0.1	t	t	-	t
9	1,8-Cineole	1036	-	-	-	t	-	-
10	γ-Terpinene	1064	-	-	-	0.3	-	-
11	α-Terpinolene	1092	-	0.1	-	0.1	-	-
12	Linalool	1105	t	0.1	t	0.3	0.1	t
13	Nonanal	1112	-	-	-	t	-	-
14	Terpinen-4-ol	1184	-	0.1	0.1	0.1	0.1	0.2
15	α-Terpineol	1197	0.1	0.1	0.1	0.1	0.1	0.2
16	Decanal	1209	-	0.1	-	0.1	0.1	-
17	Nerol	1241	-	0.1	t	0.1	0.1	0.2
18	Neryl acetate	1369	0.1	0.4	0.3	0.4	0.3	0.2
19	α-Ylangene	1375	-	0.1	0.1	0.1	0.1	0.2
20	α-Copaene	1380	0.1	0.3	0.1	0.3	0.1	0.2
21	Italicene	1407	0.1	0.3	0.3	0.3	0.1	0.2
22	cis-α-Bergamotene	1419	0.1	0.1	0.1	0.1	0.1	0.5
23	trans-Caryophyllene	1424	0.2	0.5	0.4	0.8	0.7	0.3
24	Aromadendrene	1443	-	-	-	0.5	-	-
25	α-Humulene	1457	0.1	0.1	0.1	0.1	0.1	0.2
26	Alloaromadendrene	1464	0.1	-	-	0.3	-	-
27	γ-Curcumene	1485	0.7	-	-	0.3	-	1.3
28	Ar-Curcumene	1487	0.4	5.0	3.3	4.7	2.9	1.6
29	β-Selinene	1492	0.2	0.1	-	0.1	-	1.3
30	α-Selinene	1499	0.2	1.2	0.7	0.9	0.6	0.5
31	4-Methyl-2,6-bis(1,1-dimethylethyl)phenol	1515	0.1	-	-	0.5	0.3	0.2
32	8-Cadinene	1528	0.1	0.3	0.3	0.3	0.1	0.2
33	α-Calacorene	1548	-	0.1	0.1	0.1	-	0.2
34	Spathulenol	1584	-	-	-	0.5	-	-
35	Dodecanoic acid	1587	-	1.3	-	-	-	-
36	2-Ethyl-5-hydroxy-3,6-dimethylpyran-4-one*	1613	0.4	-	0.9	1.4	0.8	-
37	7-Epi-Amiteol*	1665	0.2	1.3	0.4	0.9	0.6	0.5
38	α-Bisabolol	1692	-	0.4	-	-	-	-
39	Tetradecanoic acid	1775	-	1.1	-	0.7	0.7	-
40	Eicosane	2000	-	0.8	7.4	7.1	-	-
41	1-[2-(3-Hydroxyprop-1-en-2-yl)-2,3-dihydro-1-benzofuran-5-yl]ethanone* (12-Hydroxytremetone; Bitalin A)	2043	9.2	16.0	6.1	19.7	23.0	12.9
42	Heneicosane	2100	-	1.1	-	0.4	-	-
43	1-[2-(3-Acetylprop-1-en-2-yl)-2,3-dihydro-1-benzofuran-5-yl]ethanone* (12-Acetoxytremetone)	2114	3.7	13.3	3.7	8.3	12.1	12.7
44	1-[2-(2-Methyl-2,3-dihydroxypropyl)-2,3-dihydro-1-benzofuran-5-yl]ethanone*	2121	17.1	16.9	19.7	25.2	20.4	17.2
45	1-[2-(3-Hydroxy-2-(1-hydroxyprop-1-en-2-yl)-2,3-dihydro-1-benzofuran-5-yl]ethanone* (Gnaphaliol)	2158	2.4	4.4	5.3	2.4	4.9	2.9
46	Docosane	2200	3.8	2.1	26.6	4.5	3.7	10.1
47	Isobutyl bitalin A*	2283	6.5	9.0	4.3	4.1	4.4	4.8
48	Tricosane	2300	-	4.6	-	0.4	-	5.6
49	1-[2-(Acetylprop-1-en-2-yl)-3-hydroxy-2,3-dihydro-1-benzofuran-5-yl]ethanone*	2309	10.3	7.0	6.4	6.7	6.5	7.1
50	Diisooctyl phthalate	>2300	-	-	-	1.4	4.1	6.3
51	Squalene	>2300	35.6	-	-	-	-	-
	Total identified		91.8%	88.8%	86.8%	95.1%	87.4%	87.8%

SFE

# MATRICARIA CHAMOMILLA

lab



Molnar et al. Chemistry Central Journal (2017) 11:78  
DOI 10.1186/s13065-017-0308-y

Chemistry Central Journal

RESEARCH ARTICLE

Open Access



Comparison of various techniques for the extraction of umbelliferone and herniarin in *Matricaria chamomilla* processing fractions

Maja Molnar, Nikolina Mendešević, Drago Šubarić, Ines Banjari and Stela Jokić\*

pilot



# SATUREJA MONTANA



SC-CO<sub>2</sub> extraction

Mathematical modeling of SC-CO<sub>2</sub> process

Chemical analysis of extracts

Hierarchical cluster analysis of compounds

Compound	Pressure (bar)										
	100	125	150	175	200	225	250	275	300	325	350
<b>Monoterpene hydrocarbons</b>											
$\beta$ -Myrcene	0.14	0.35	0.28	0.46	0.08	0.08	0.05	ni	0.06	ni	ni
$\alpha$ -Terpinene	0.40	0.47	0.47	0.60	0.21	0.17	0.21	0.17	0.13	ni	0.21
$\gamma$ -Terpinene	0.48	1.11	1.37	2.04	1.20	1.24	1.01	1.01	0.79	1.12	0.78
p-Cymene	7.13	7.83	8.79	10.24	5.00	5.33	4.77	4.34	3.64	5.08	4.23
m-Cymene	ni <sup>a</sup>	ni	ni	ni	0.44	0.52	0.32	ni	ni	ni	ni
<b>Oxygenated monoterpenes</b>											
Eucalyptol	0.70	0.68	0.74	0.83	0.33	ni	ni	0.23	0.24	0.31	ni
Trans-sabinene hydrate	0.80	0.79	0.56	0.75	0.27	0.36	0.36	0.35	0.30	0.40	0.16
Cis-sabinene hydrate	0.26	0.29	0.33	0.25	0.12	0.16	0.16	0.19	0.19	0.24	0.19
Linalool	0.51	0.70	0.70	0.52	0.47	0.40	0.34	0.30	0.39	0.36	0.34
Borneol	2.42	2.00	1.99	2.14	1.34	1.29	1.42	1.29	1.44	1.52	1.42
Terpinene 4-ol	0.70	0.45	0.55	0.56	0.45	0.44	0.43	0.29	0.47	0.33	0.57
$\alpha$ -Terpineol	0.08	0.08	ni	ni	ni	0.08	0.08	0.16	0.18	0.17	ni
Carvone	0.82	0.87	1.01	0.63	0.69	0.85	0.54	ni	0.53	0.75	0.87
Carvacrol	67.58	60.70	57.43	54.30	71.34	72.69	73.21	77.36	76.67	79.38	76.10
<b>Sesquiterpenes</b>											
Trans-caryophyllene	2.80	2.45	2.79	2.92	2.03	2.04	2.26	2.10	1.97	2.06	2.08
$\alpha$ -Amorphen	0.55	0.60	0.67	0.66	0.32						
$\beta$ -Bisabolene	0.89	0.98	0.95	1.10	0.66						
$\gamma$ -Cadinene	0.59	0.57	0.61	0.68	0.39						
$\delta$ -Cadinene	1.02	1.06	1.09	1.18	0.68						
Caryophyllene oxide	1.58	1.63	1.54	1.74	0.95						
<b>Aliphatics</b>											
Heptacosane	0.59	1.51	1.66	1.94	0.95						
Nonacosane	2.74	7.29	8.84	8.92	7.20						
<b>Total</b>	92.77	92.40	92.39	92.44	95.10						

J. of Supercritical Fluids 117 (2016) 89–97



Contents lists available at ScienceDirect

The Journal of Supercritical Fluids

journal homepage: [www.elsevier.com/locate/supflu](http://www.elsevier.com/locate/supflu)



Winter savory: Supercritical carbon dioxide extraction and mathematical modeling of extraction process

Jelena Vladić<sup>a</sup>, Zoran Zeković<sup>a</sup>, Stela Jokić<sup>b</sup>, Sandra Svilović<sup>c</sup>, Strahinja Kovačević<sup>a</sup>, Senka Vidović<sup>a,\*</sup>



# VITEX AGNUS-CASTUS



## SC-CO<sub>2</sub> extraction



J. of Supercritical Fluids 123 (2017) 50–57

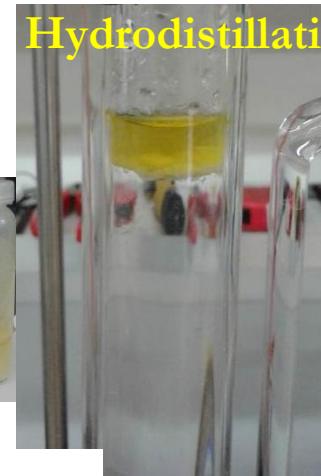
Contents lists available at ScienceDirect

The Journal of Supercritical Fluids



Journal homepage: [www.elsevier.com/locate/supflu](http://www.elsevier.com/locate/supflu)

## Hydrodistillation



SC-CO<sub>2</sub> extraction of *Vitex agnus-castus* L. fruits: The influence of pressure, temperature and water presoaking on the yield and GC-MS profiles of the extracts in comparison to the essential oil composition

Stela Jokić <sup>a,\*</sup>, Igor Jerković <sup>b</sup>, Marina Rajić <sup>c</sup>, Krinoslav Aladić <sup>d</sup>, Mate Bilić <sup>a</sup>,  
Senka Vidović <sup>e</sup>

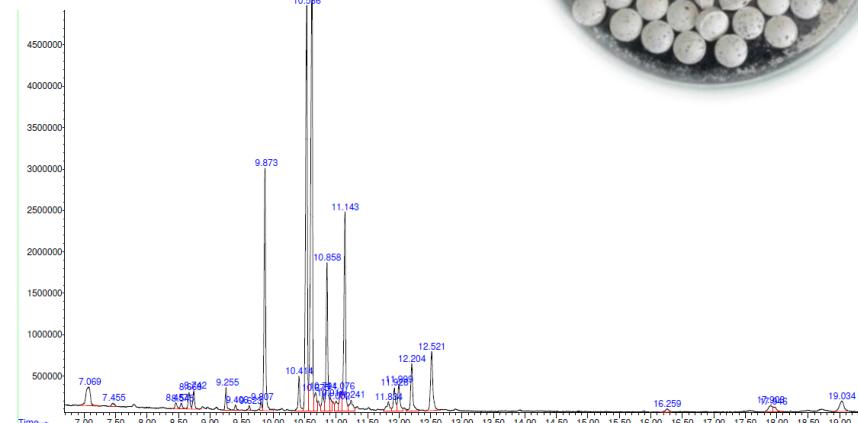


TIC: PROBA2.D\data.ms

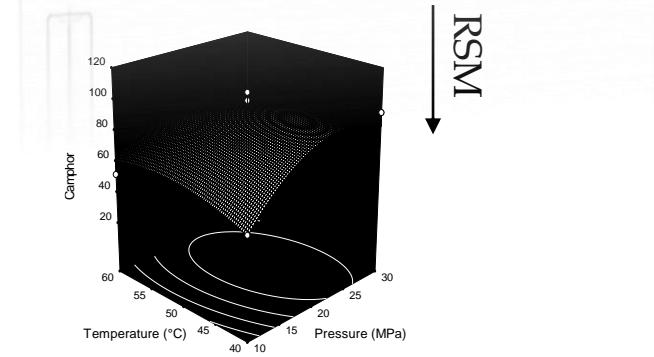
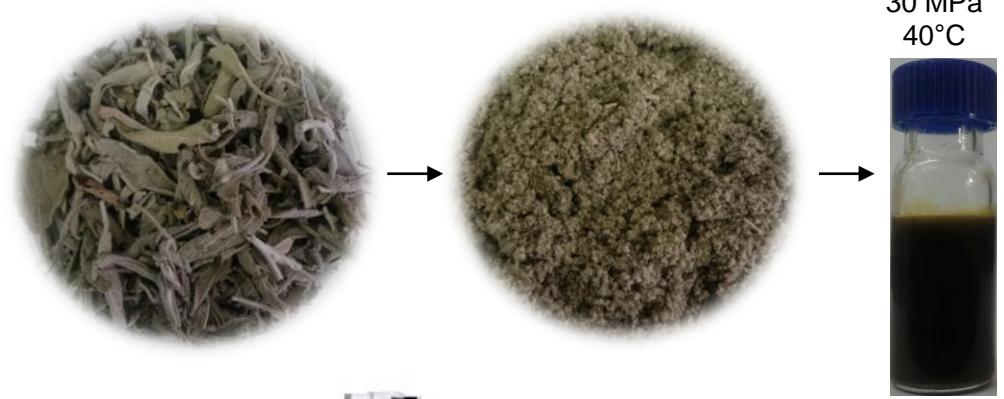
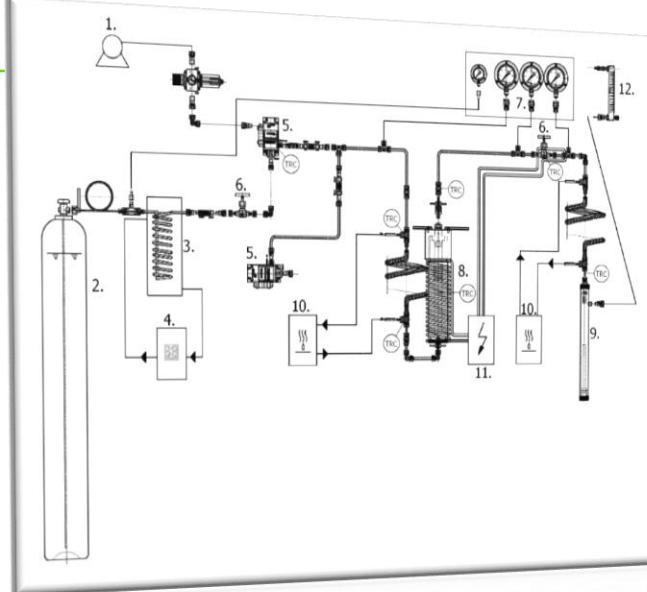


Essential oil

Essential oil



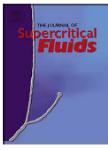
# *Salvia officinalis* L.



The Journal of Supercritical Fluids 133 (2018) 253–262



Contents lists available at ScienceDirect  
The Journal of Supercritical Fluids  
journal homepage: [www.elsevier.com/locate/supflu](http://www.elsevier.com/locate/supflu)

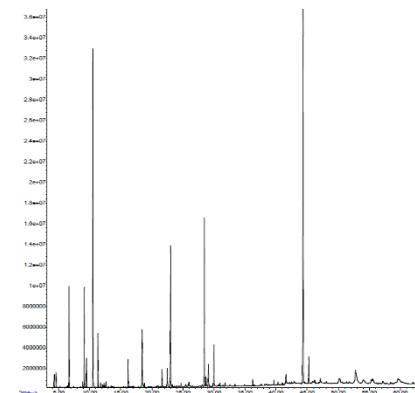


Optimization of supercritical CO<sub>2</sub> extraction of *Salvia officinalis* L. leaves targeted on Oxygenated monoterpenes,  $\alpha$ -humulene, viridiflorol and manool

Stela Jokić<sup>a,\*</sup>, Maja Molnar<sup>a</sup>, Martina Jakovljević<sup>a</sup>, Krunoslav Aladić<sup>b</sup>, Igor Jerković<sup>c</sup>



CrossMark





## Extraction of bioactive phenolics from black poplar (*Populus nigra* L.) buds by supercritical CO<sub>2</sub> and its optimization by response surface methodology

Piotr Kuś <sup>a,\*</sup>, Igor Jerković <sup>b</sup>, Martina Jakovljević <sup>c</sup>, Stela Jokić <sup>c</sup>

<sup>a</sup> Department of Pharmacognosy, Wrocław Medical University, ul. Borowska 211a, 50-556 Wrocław, Poland

<sup>b</sup> Department of Organic Chemistry, Faculty of Chemistry and Technology, University of Split, R. Boškovića 35, 21000 Split, Croatia

<sup>c</sup> Faculty of Food Technology, Josip Juraj Strossmayer University of Osijek, Franje Kuhača 20, 31000 Osijek, Croatia



Journal of Pharmaceutical and Biomedical Analysis 158 (2018) 15–27

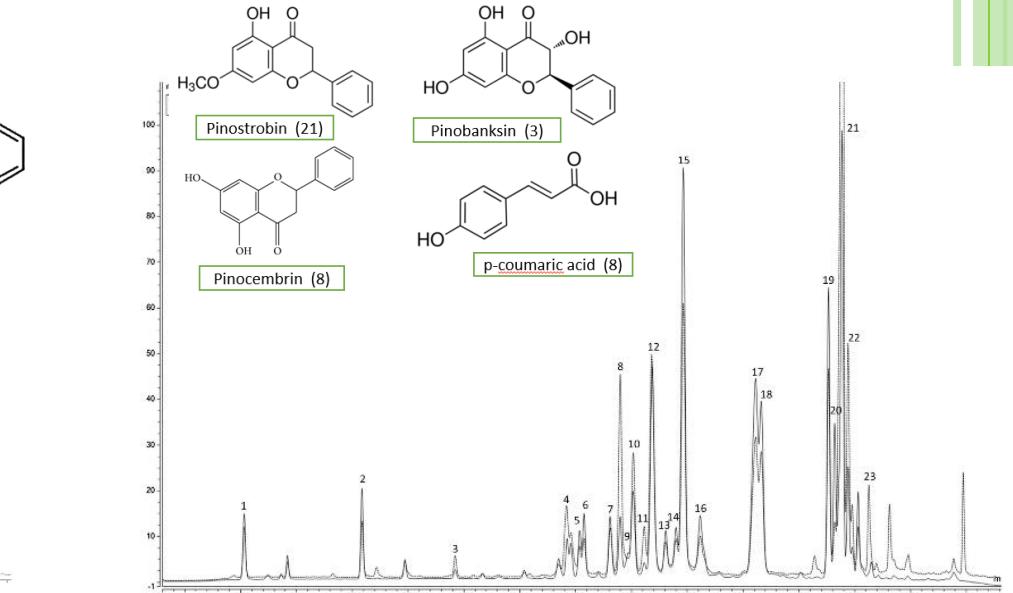
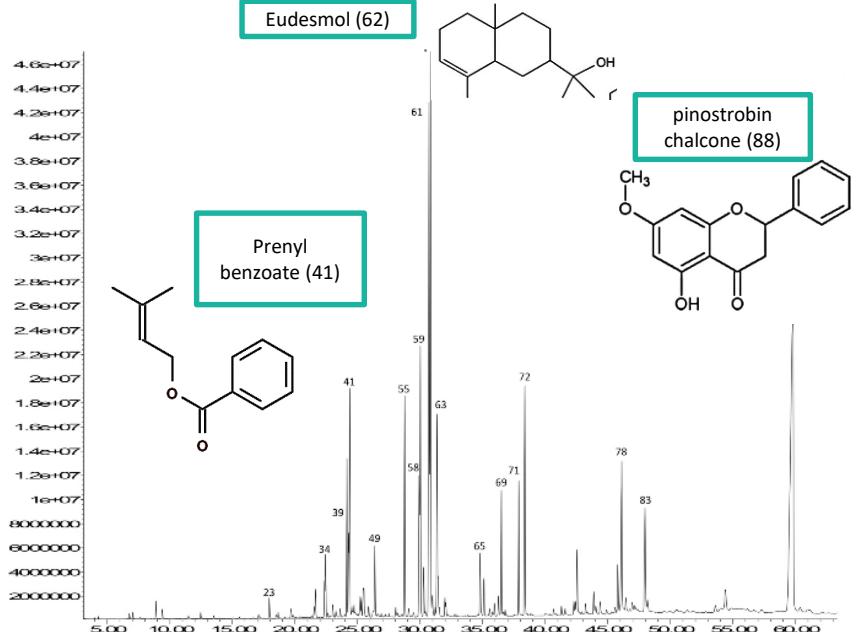


ELSEVIER



## Development of supercritical CO<sub>2</sub> extraction of bioactive phytochemicals from black poplar (*Populus nigra* L.) buds followed by GC-MS and UHPLC-DAD-QqTOF-MS

Piotr M. Kuś <sup>a,\*</sup>, Piotr Okińczyk <sup>a</sup>, Martina Jakovljević <sup>b</sup>, Stela Jokić <sup>b</sup>, Igor Jerković <sup>c,\*</sup>



# FOOD INDUSTRY BY-PRODUCTS





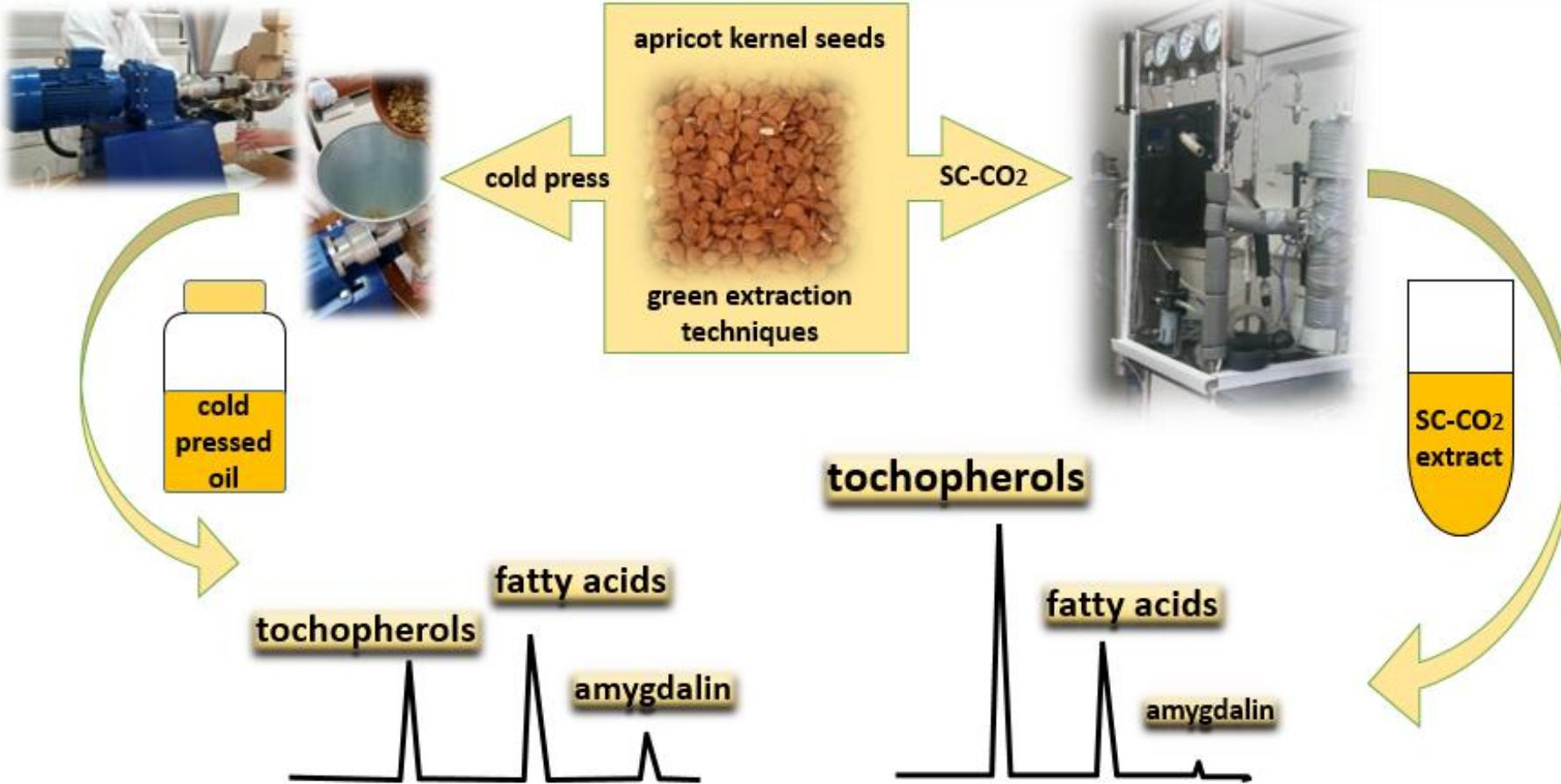
## RESEARCH ARTICLE

Apricot Kernel Oils

European Journal of  
Lipid Science and Technology  
[www.ejlst.com](http://www.ejlst.com)

# Recovery of Tocopherols, Amygdalin, and Fatty Acids From Apricot Kernel Oil: Cold Pressing Versus Supercritical Carbon Dioxide

Nika Pavlović, Senka Vidović, Jelena Vladić, Ljiljana Popović, Tihomir Moslavac, Snježana Jakobović, and Stela Jokić\*

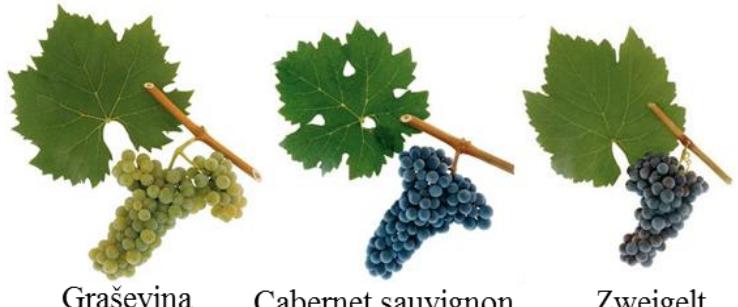




Original article

## Optimisation of supercritical CO<sub>2</sub> extraction of grape seed oil using response surface methodology

Stela Jokić,<sup>1</sup> Marco Bijuk,<sup>1</sup> Krunoslav Aladić,<sup>2</sup> Mate Bilić<sup>1</sup> & Maja Molnar<sup>1\*</sup>



Graševina

Cabernet sauvignon

Zweigelt

### Seed preparation

- Seeds screened from pomace
- Seeds screened from pomace and washed
- Seeds screened from pomace after drying

Grape seeds



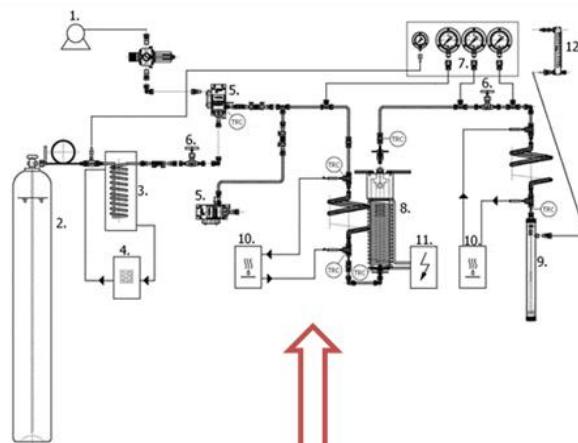
### Drying

- Natural drying
- Chamber drying

Grounded grape seeds



### Supercritical CO<sub>2</sub> extraction



Deffated meal



Grape seed oil

# On going research



***NEW Installation Research Projects: „Application of innovative techniques of the extraction of bioactive components from by-products of plant origin” (2018-2023)***  
*Principal Investigator: Stela Jokić*  
**(Budget: 1.607.708,72 HRK)**

PTF  
OS





# ByProExtract



**Cocoa shell**



**Citrus peel**



a) Dust



**Tobacco waste**

b) Midrib



c) Scrap



# Thank you for your attention



This work has been supported by Croatian Science Foundation under the project “Application of innovative techniques of the extraction of bioactive components from by-products of plant origin”  
(UIP-2017-05-9909)