Innovative Food Product Development Cycle: Frame for Stepping Up Research Excellence of FINS

European Commission

Conventional vs Novel extraction technologies

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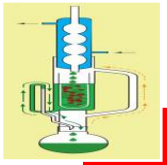
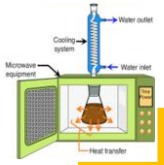
Extraction technologies



Classical (SLE/LLE)

- Methanol
- Ethanol
- Water
- Acid/Alkali
- etc



Combination

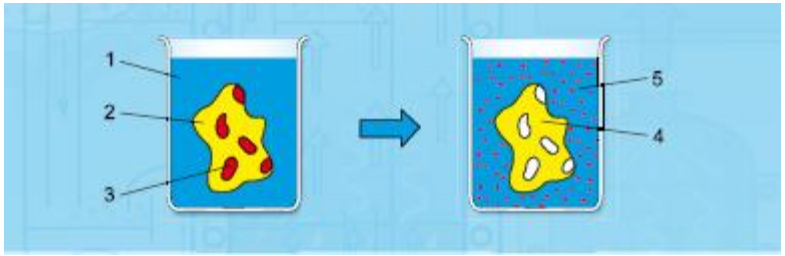
- Solvent +NE
- NE+NE
  - Enzyme +Ultrasound
  - Ultrasound +Microwave
  - Any suitable combinations

Novel extraction (NE)

- Enzymes
- Ultrasound
- Supercritical
- Pressurized liquid
- Negative pressure cavitation
- High pressure
- Pulsed electric field
- Microwave

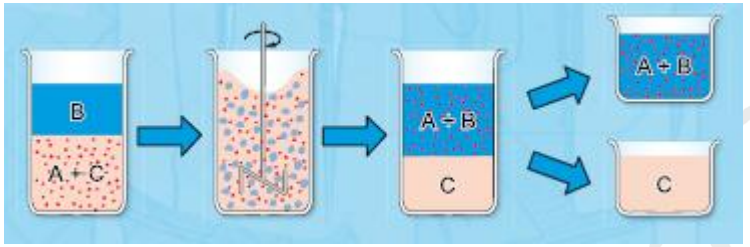
Conventional extraction techniques

Solid-liquid extraction



__1__ solvent, __2__ extraction material (solid carrier phase with transition component), __3__ transition component, __4__ depleted solid carrier phase, __5__ solvent with dissolved transition component

Liquid-liquid extraction



When the initial mixture __(A+C)__ and the solvent __(B)__ are mixed, the transition component __(A)__ is transferred into the solvent.
After settling, two phases are obtained: the extract __(A+B)__ and the carrier liquid __(C)__.

Selected example

Extractable Biomolecule	Substrate	Yield
Pectin	Apple pomace, Citrus peel, Sugar beet, Sunflower heads, wastes from tropical fruits	10%–15%, 20%–30%
Flavanones	Citrus peels and residues from segments and seeds after pressing	
Total and soluble dietary fibres	Apple pomace	72% and 10%
Phenolic compounds	Apple pomace	33%
γ-oryzanol	Rice bran	1527–4164 mg/kg
β-glucans	Barley bran	
Lignins	Flaxseeds	
Phenolic acids	Wheat brans	
Lycopene and β-carotene	Tomato pomace	50%

Example of some extracted bioactive compounds by different solvents

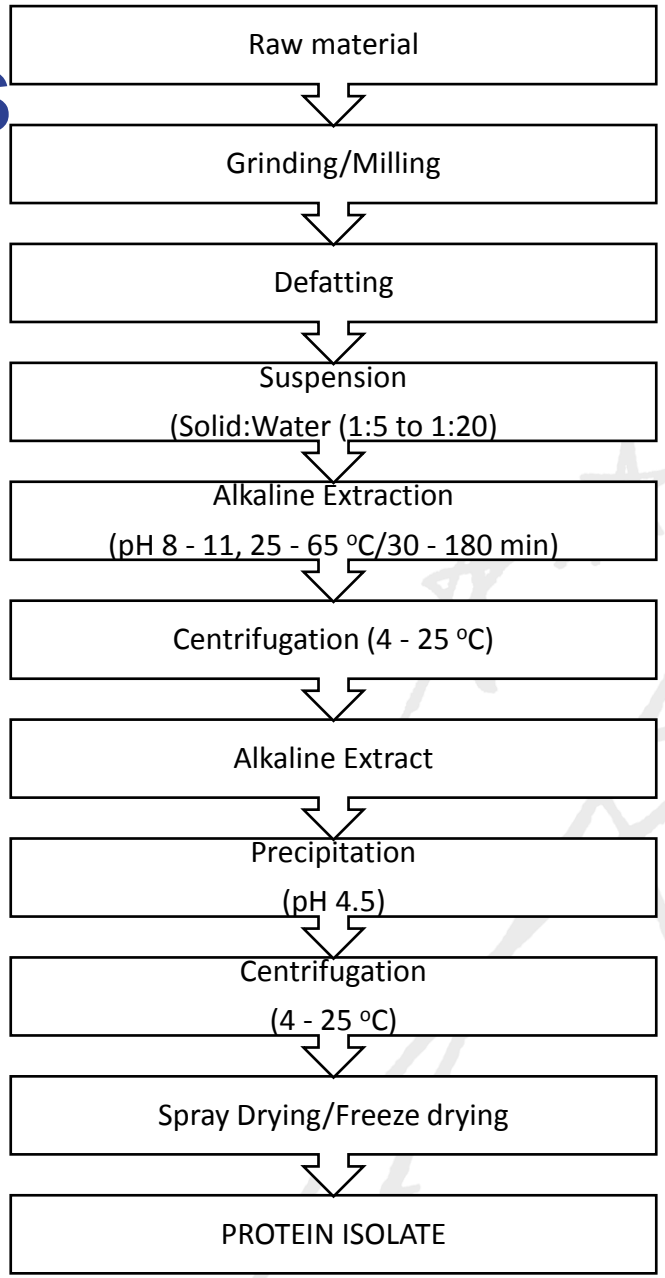
Water	Ethanol	Methanol	Chloroform	Dichloromethanol	Ether	Acetone
Anthocyanins	Tannins	Anthocyanin	Terpenoids	Terpenoids	Alkaloids	Flavonoids
Tannins	Polyphenols	Terpenoids	Flavonoids		Terpenoids	
Saponins	Flavonol	Saponins				
Terpenoids	Terpenoids	Tannins				
	Alkaloids	Flavones				
		Polyphenols				

Extraction of proteins

Technique employed for range of raw materials including grains, marine and meat sources

Challenge

Removal of salt from final product



Drivers for novel extraction technologies in the food industry

- *Regulation*
- *Food Safety and Shelf life extension*
- *Nutrient and Sensory aspects*
- *Consumer and Processor Acceptability*
- *Technology advances*
- *Environmental impact*
- *Hurdle concept*

Clean and green extraction technologies

Principle of clean and green extraction techniques

- **Principle 1:** Innovation by selection of varieties and use of renewable plant resources.
- **Principle 2:** Use of alternative solvents and principally water or agro-solvents.
- **Principle 3:** Reduce energy consumption by energy recovery and using innovative technologies.
- **Principle 4:** Production of co-products instead of waste to include the bio- and agro-refining industry.
- **Principle 5:** Reduce unit operations and favour safe, robust and controlled processes.
- **Principle 6:** Aim for a non denatured and biodegradable extract without contaminants.

Chemat (2012) Int. J. Mol. Sci., 13(7), 8615-8627

Novel extraction techniques

- Enzyme assisted extraction
- Microwave assisted extraction
- Ultrasound assisted extraction
- Supercritical fluid extraction
- Pressurized liquid extraction
- Negative pressure cavitation

Alternative solvents for green extraction

Solvent	Extraction Technique (Application)	Solvent Power			Health & Safety	Cost	Environmental Impact
		Polar	Weakly Polar	Non-Polar			
Solvent-free	Microwave Hydrodiffusion and Gravity (antioxidants, essential oils)	+++	+		+++	+	+++
	Pulse Electric Field (antioxidants, pigments)	+++	+		+++	+	+++
Water	Steam distillation (essential oils)	++	+		+	++	+
	Microwave-assisted distillation (essential oils)	+++	+++	+	+	+	++
	Extraction by sub-critical water (Aromas)	+	++		+	+	+
CO_2	Supercritical fluid extraction (decaffeination of tea and coffee)	–	+	+++	+	+	+
Ionic liquids	Ammonium salts (Artemisinin)	–	+	+++	–	–	++
Agrosolvents	Ethanol (pigments and antioxidants)	+	+	–	–	++	+
	Glycerol (polyphenols)	+	+	–	–	+	+
	Terpenes such as d-limonene (fats and oils)	–	–	++	–	+	+
Petrochemical solvents	n-Hexane (fats and oils)	–	+	+++	–––	++	–––

Enzyme assisted extraction

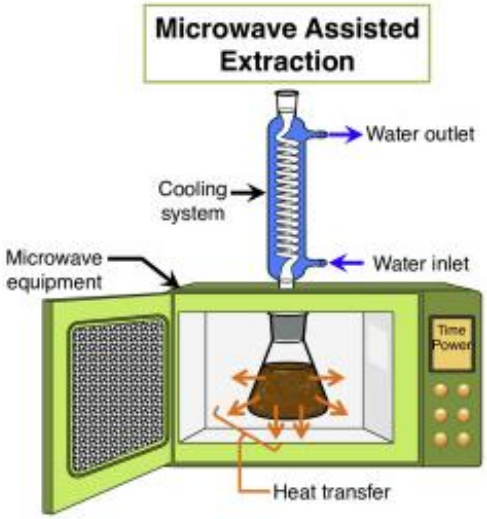
Application of enzyme assisted extraction for bioactives

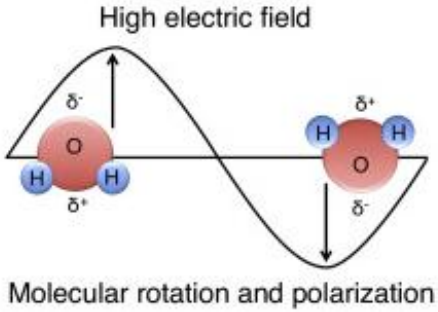
Raw material	bioactive compound	enzymes used for extraction
Undaria pinnatifida	Fucoxanthin	Alginase lyase enzymes, temperature of 37 °C and pH of 6.2
Sargassum horneri	Antioxidant rich extracts	Carbohydrases and proteases
Brown seaweed species	Antioxidant rich extracts	Carbohydrases and proteases

Enzymes, pH and temperature employed for enzyme-assisted extraction of bioactives from seaweeds

Enzyme	temperature (°C)	pH	enzyme composition
Viscozyme	50	4.5	arabanase, cellulase, β-glucanase, hemi-cellulase and xylanase
Cellucast	50	4.5	group of enzymes catalyzing the breakdown of cellulose into glucose, cellobiose and higher glucose polymer
Termamyl	60	6.0	heat-stable α-amylase
Ultraflo	60	7.0	heat-stable multi-active β-glucanase
Neutrase	50	6.0	endoprotease
Flavourzyme	50	7.0	endoprotease and exopeptidase activities
Alcalase	50	8.0	α-endoprotease

Microwave assisted extraction

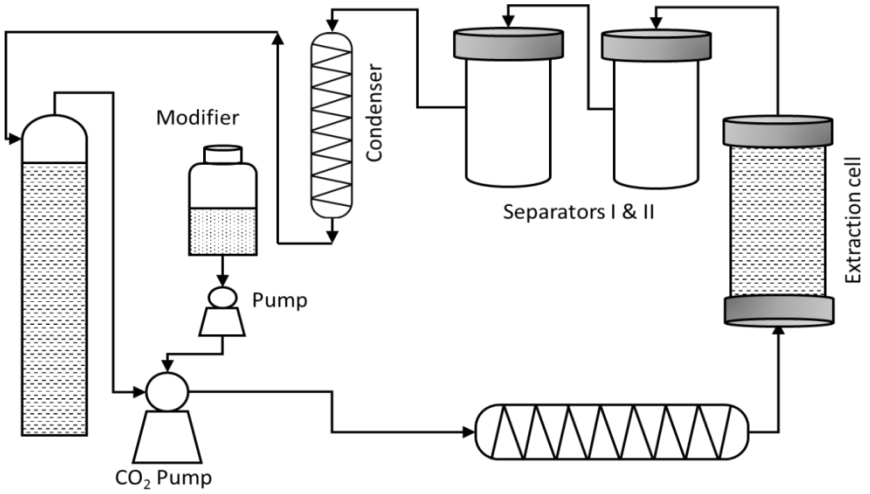


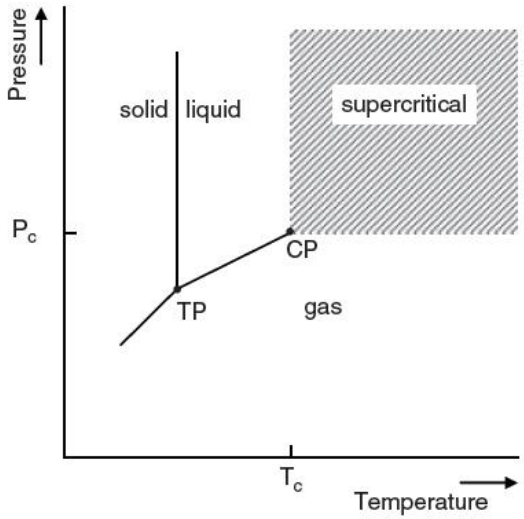


Molecular rotation and polarization

Marine algae	bioactive compound	conditions
Dunaliella tertiolecta	carotenoids	temperature of 56 °C and atmospheric pressure conditions
Fucus vesiculosus	fucoidan – sulphated polysaccharides	pressure of 200-800 kPa, extraction time 1–31 min, and alga/water ratio of 1/25 to 5/25 g ml^{-1}
Porphyra (Nori) and Palmaria (Dulse), Undaria pinnatifida (Wakame), Himanthalia elongata (Sea spaghetti) and Laminaria ochroleuca (Kombu), Ulva Rigida (Sea Lettuce)	Iodine	temperature of 200 °C, power of 1,000 W, holding time of 0-5 min

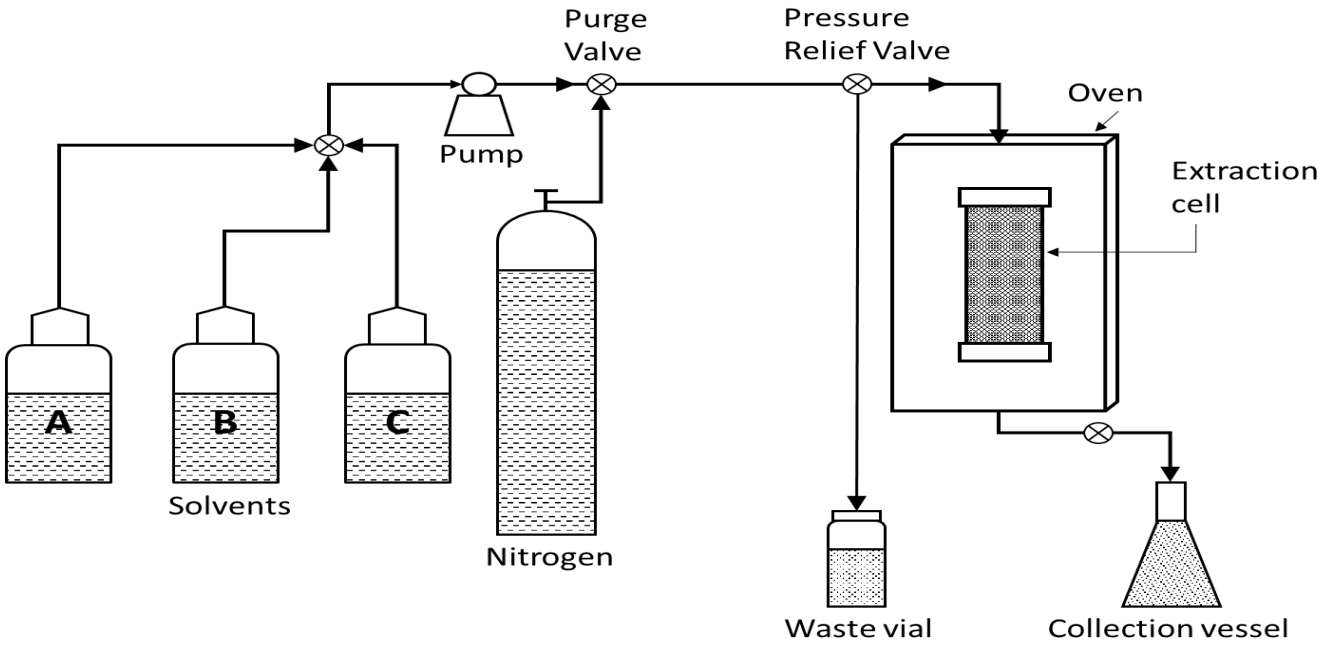
Supercritical fluid extraction





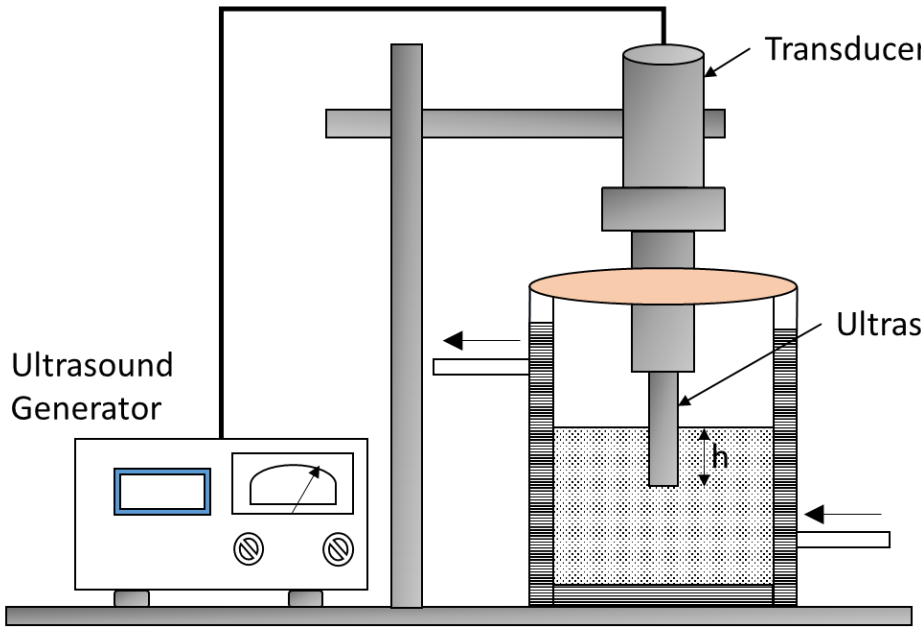
Marine algae	bioactive compound	conditions
Haemato coccuspluvialis	astaxanthin	ethanol along with acids were used as solvents for extraction
Scenedesmus almeriensis	carotenoids	pressure of 40 MPa and temperature of 60 °C
Dunaliella salina	chlorophyll	methanol as solvent
Hypneacharoides sp.	PUFA	temperature ranges from 40 to 50 °C and pressure from 24.1 and 37.9 MPa
Dunaliella salina	β-carotene	pressure of 30 MPa and temperature of 40 °C
Sargassum muticum	polyphenols	extractions were performed using CO_2 modified with 12% ethanol at 15.2 MPa pressure and 60 °C during 90 min

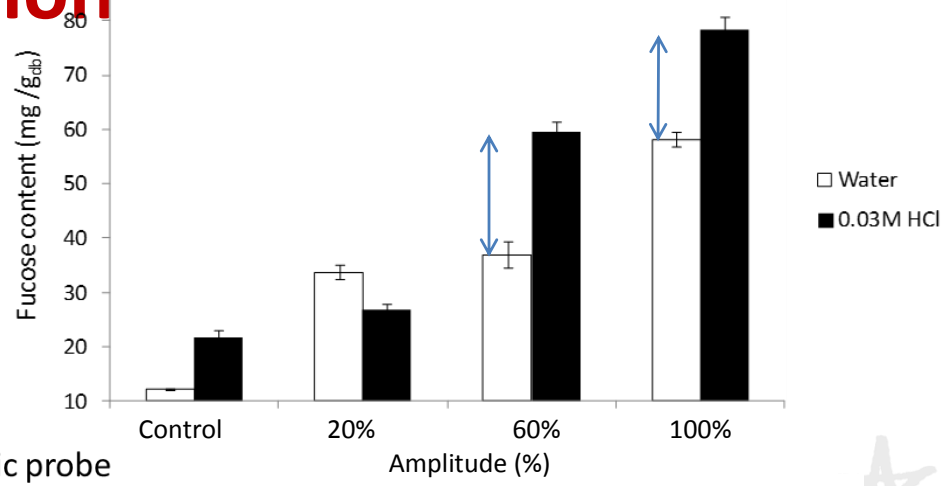
Pressurized liquid extraction

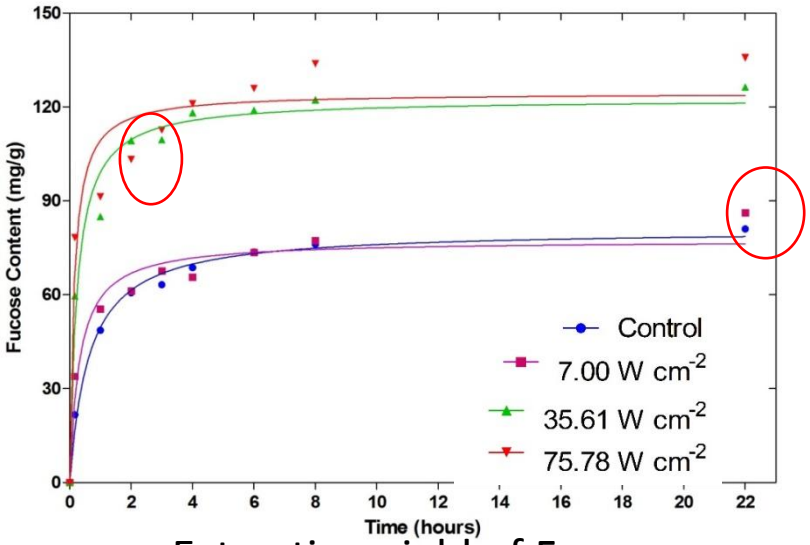


Marine algae	bioactive compound	conditions
Eisenia bicyclis	fucoxanthin	temperature 110 °C and 90% ethanol concentration
C. ellipsoidea	zeaxanthin	temperature and time for extraction were 115.4 °C and 23.3 min
Dunaliella salina	bioactive phenols	temperature of 40, 100 and 160 °C and time of 5, 17.5 and 30 min
Himanthalia elongate	bioactive phenols	temperature of 50, 100, 150 and 200 °C for 20 min
Undaria pinnatifida	antioxidants	water as solvent
Sargassum muticum	polyphenols	pressure of 10.3 MPa at 120 °C temperature for 6 min

Ultrasound Assisted Extraction

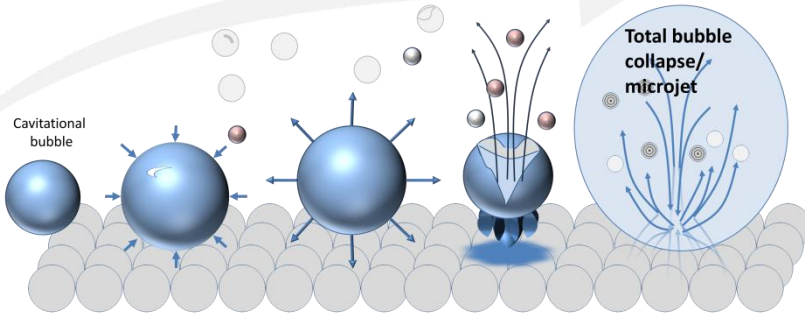




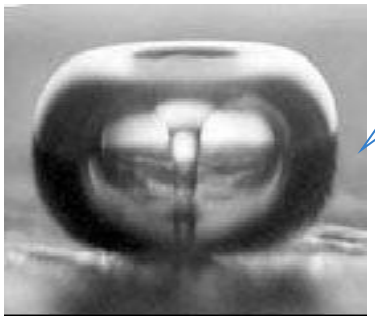


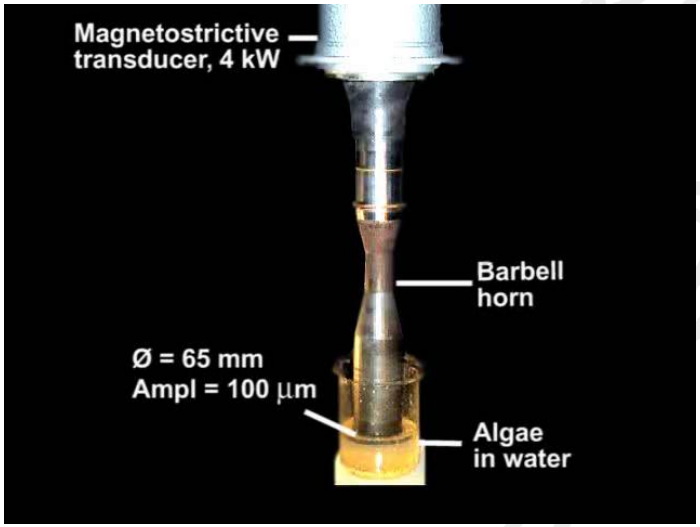
Extraction yield of Fucose

Mechanisms of action

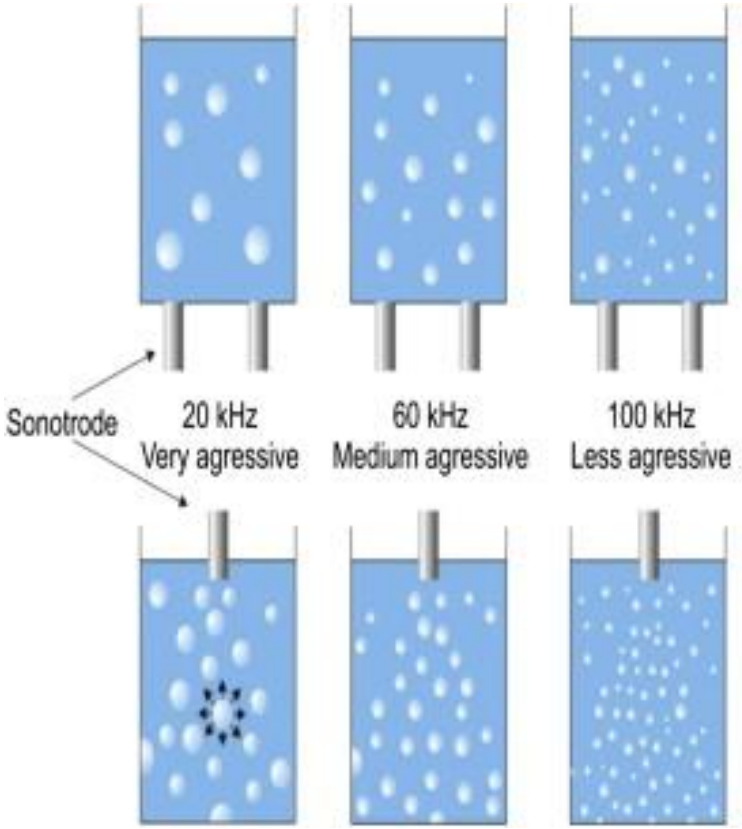


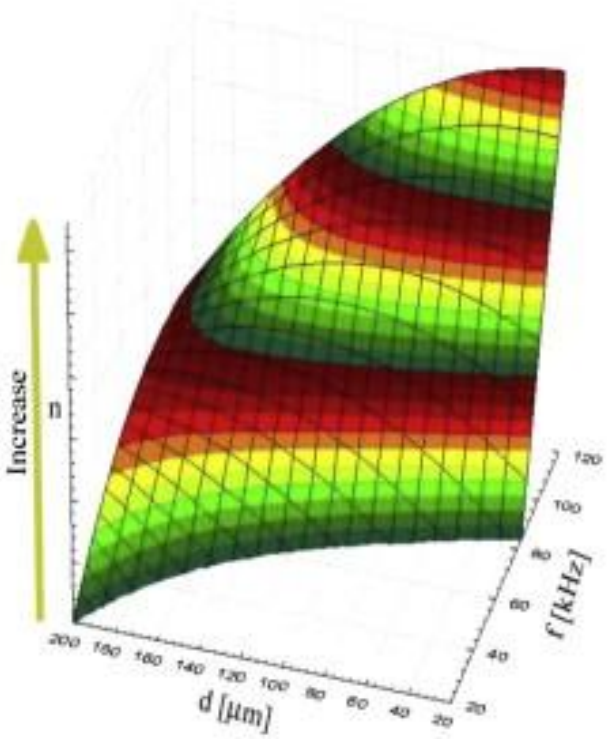
High speed micro-jet



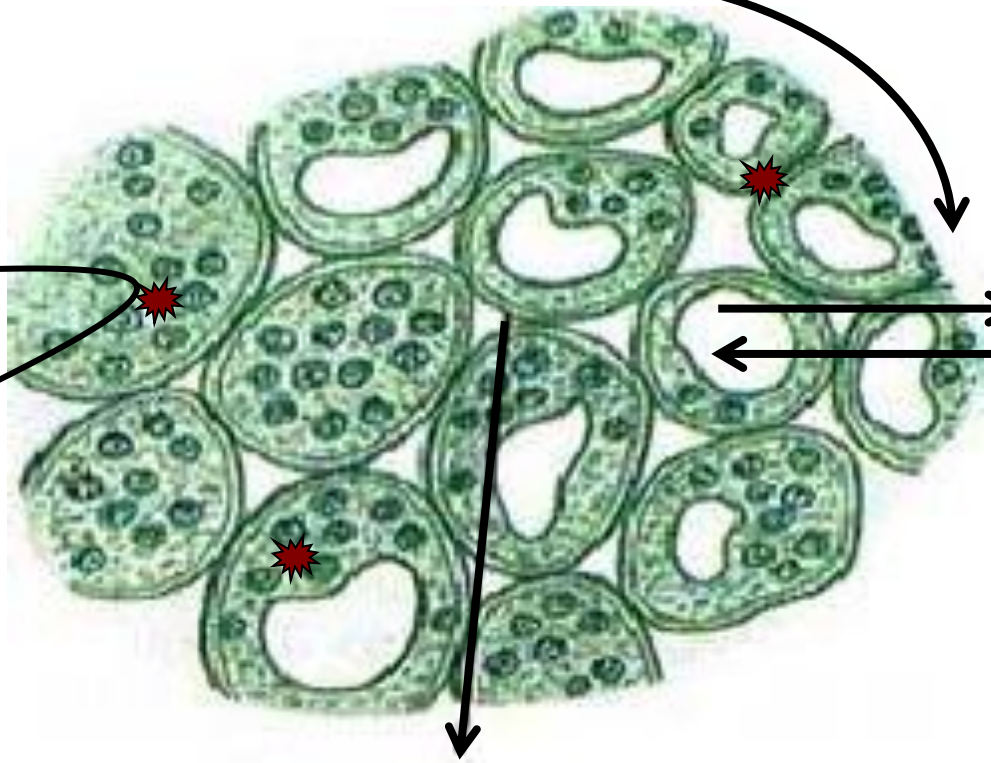


Size of the cavitational bubbles in dependence of ultrasounds frequency





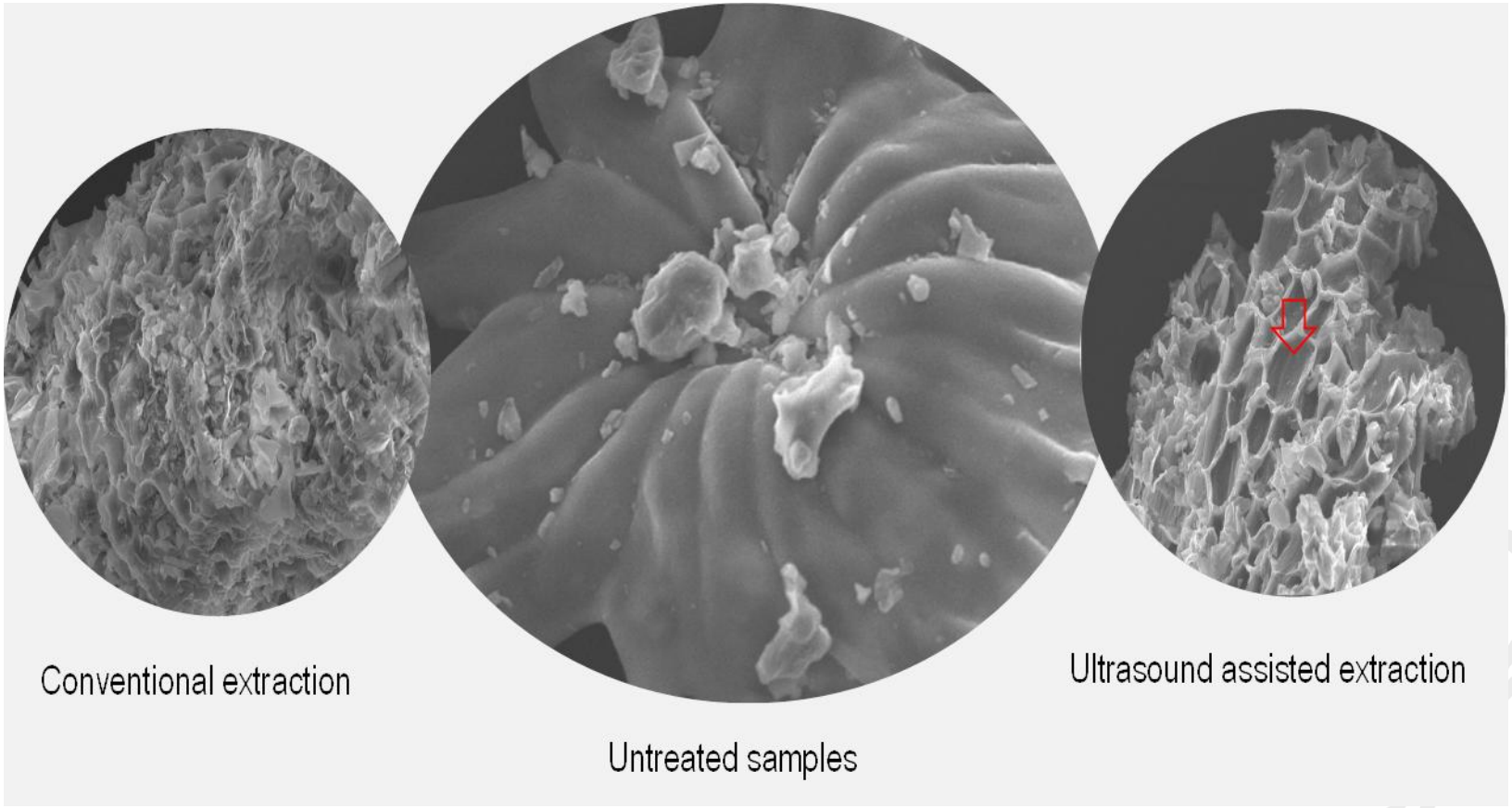
Enhanced mass transfer to substrate surface

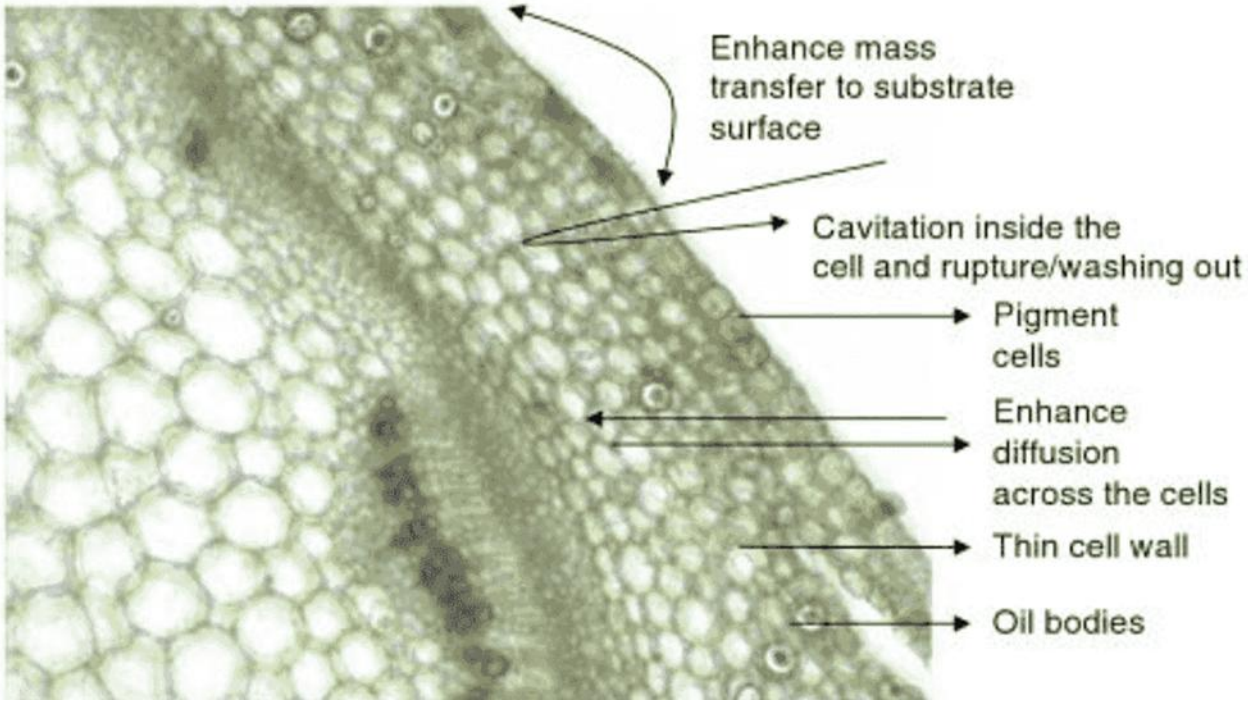


Enhanced diffusion Across the cells

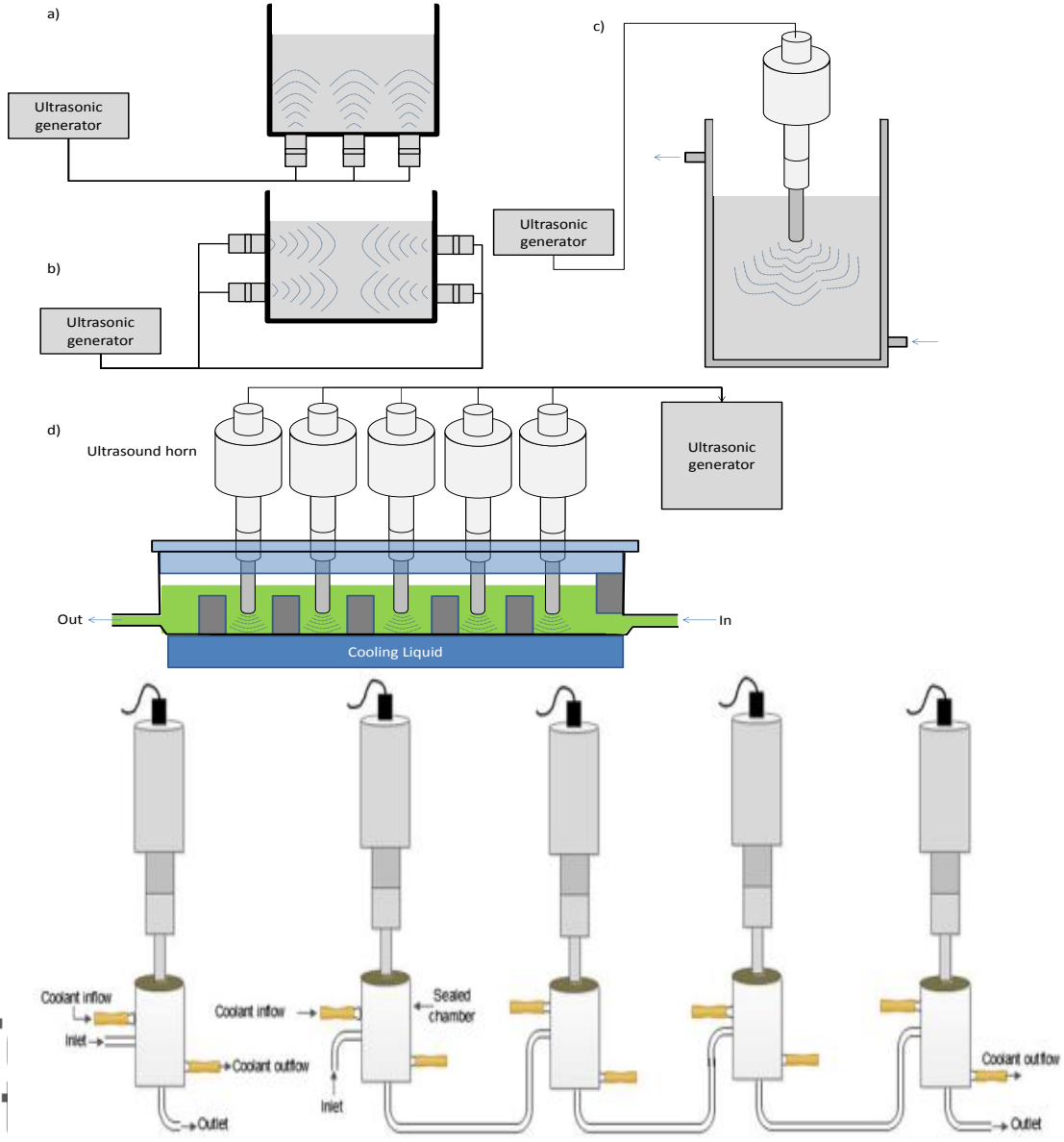
Cavitation inside the cell & rupture/washing out

Thin primary cell wall


Conventional extraction
Untreated samples
Ultrasound assisted extraction


Enhance mass transfer to substrate surface
Cavitation inside the cell and rupture/washing out
Pigment cells
Enhance diffusion across the cells
Thin cell wall
Oil bodies

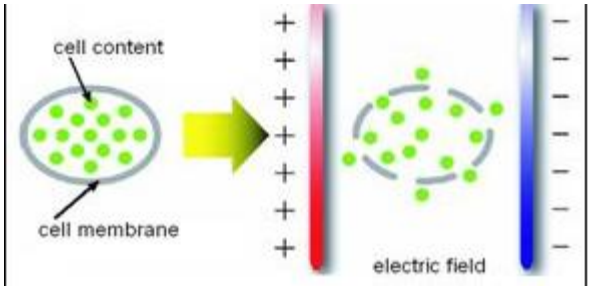
Commercially available ultrasonic systems



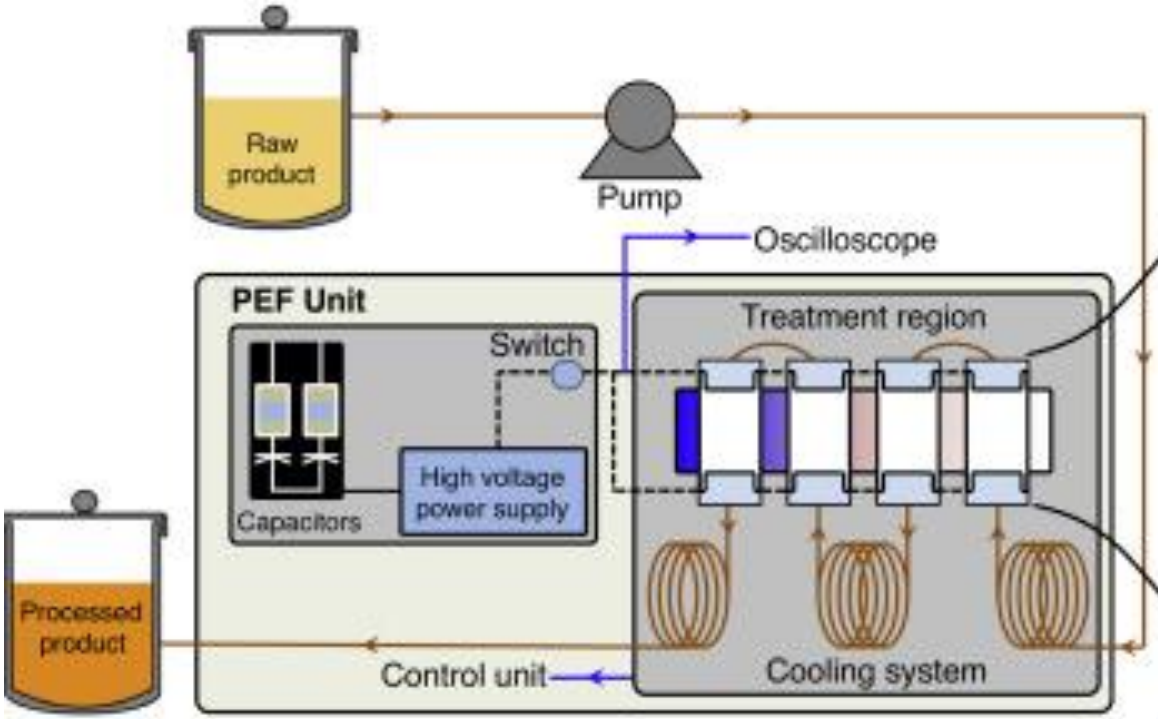
Ultrasound probe type system

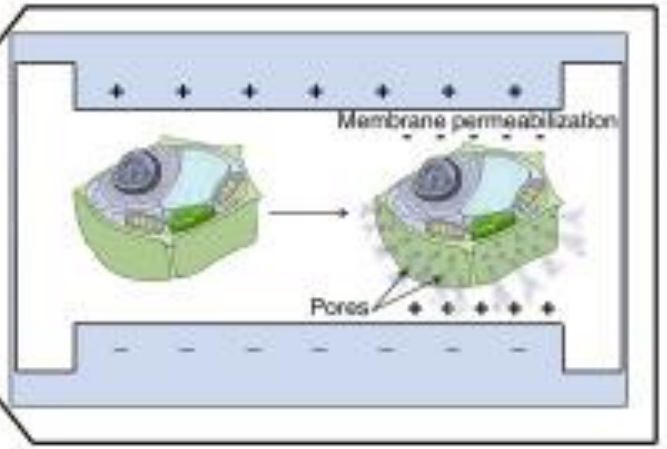
Ultrasonic bath system

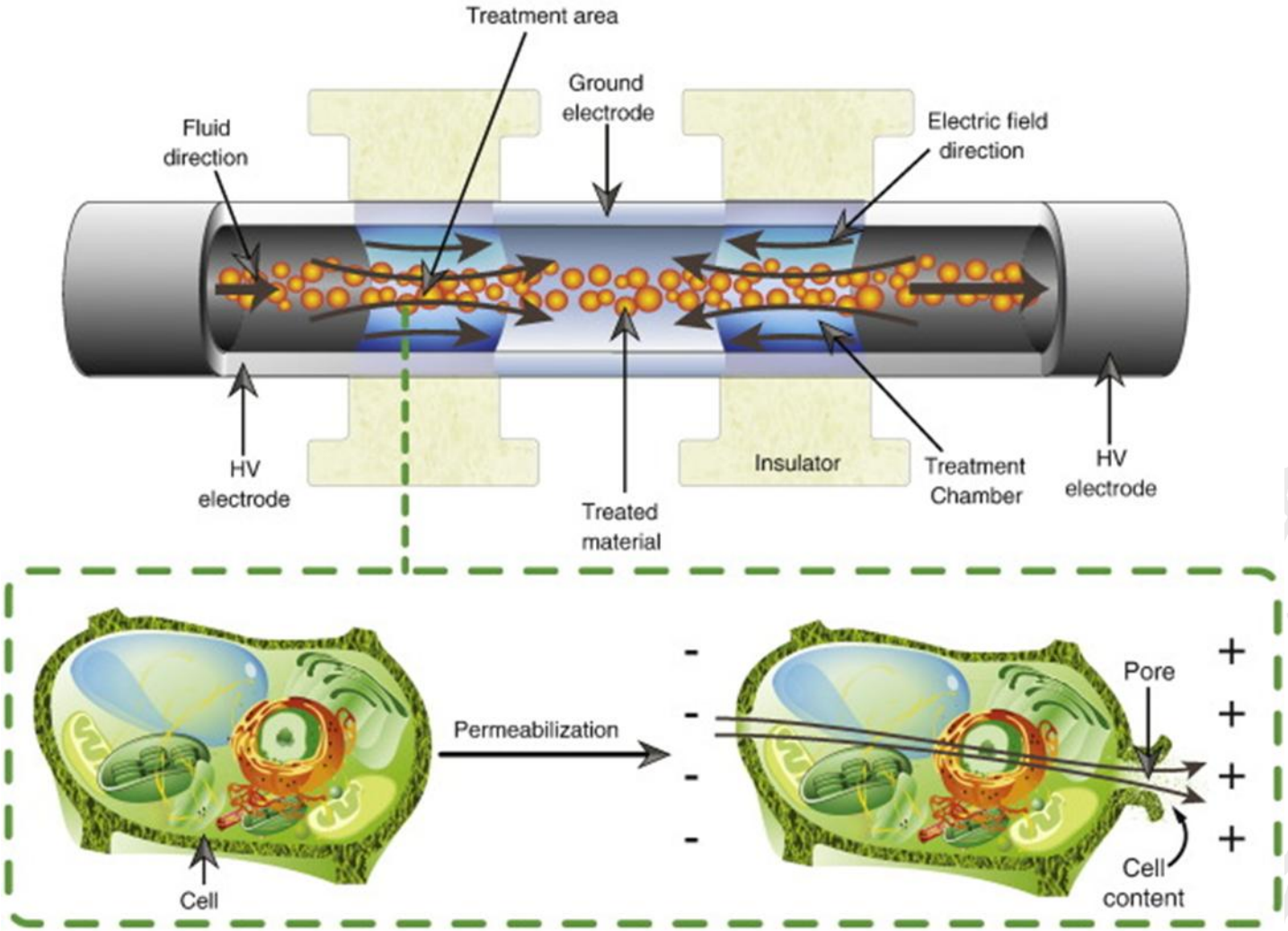
Pulsed Electric Field



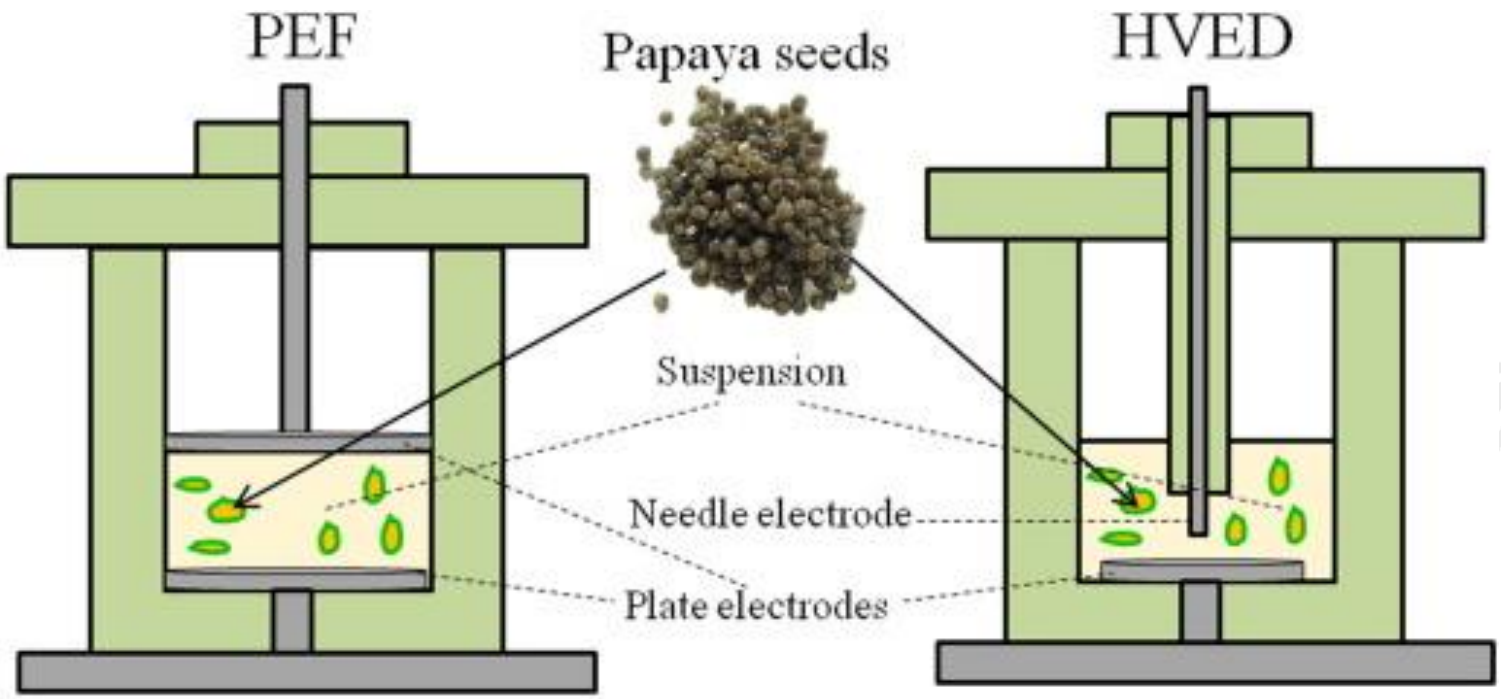
Cell permeablization



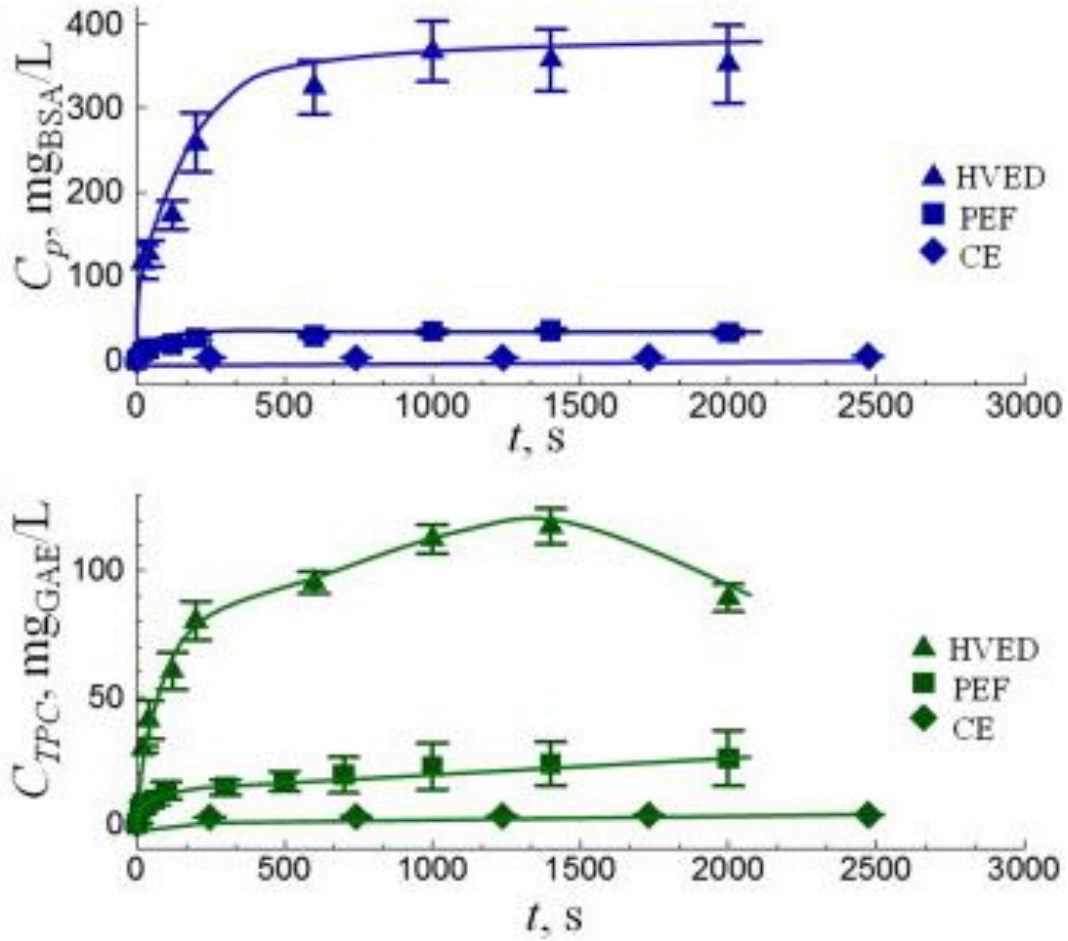



Treatment area
Ground electrode
Fluid direction
Electric field direction
HV electrode
Treated material
Insulator
Treatment Chamber
HV electrode
Cell
Permeabilization
Pore
Cell content
- - - -
+ + + +

PEF and HVED treatment chambers

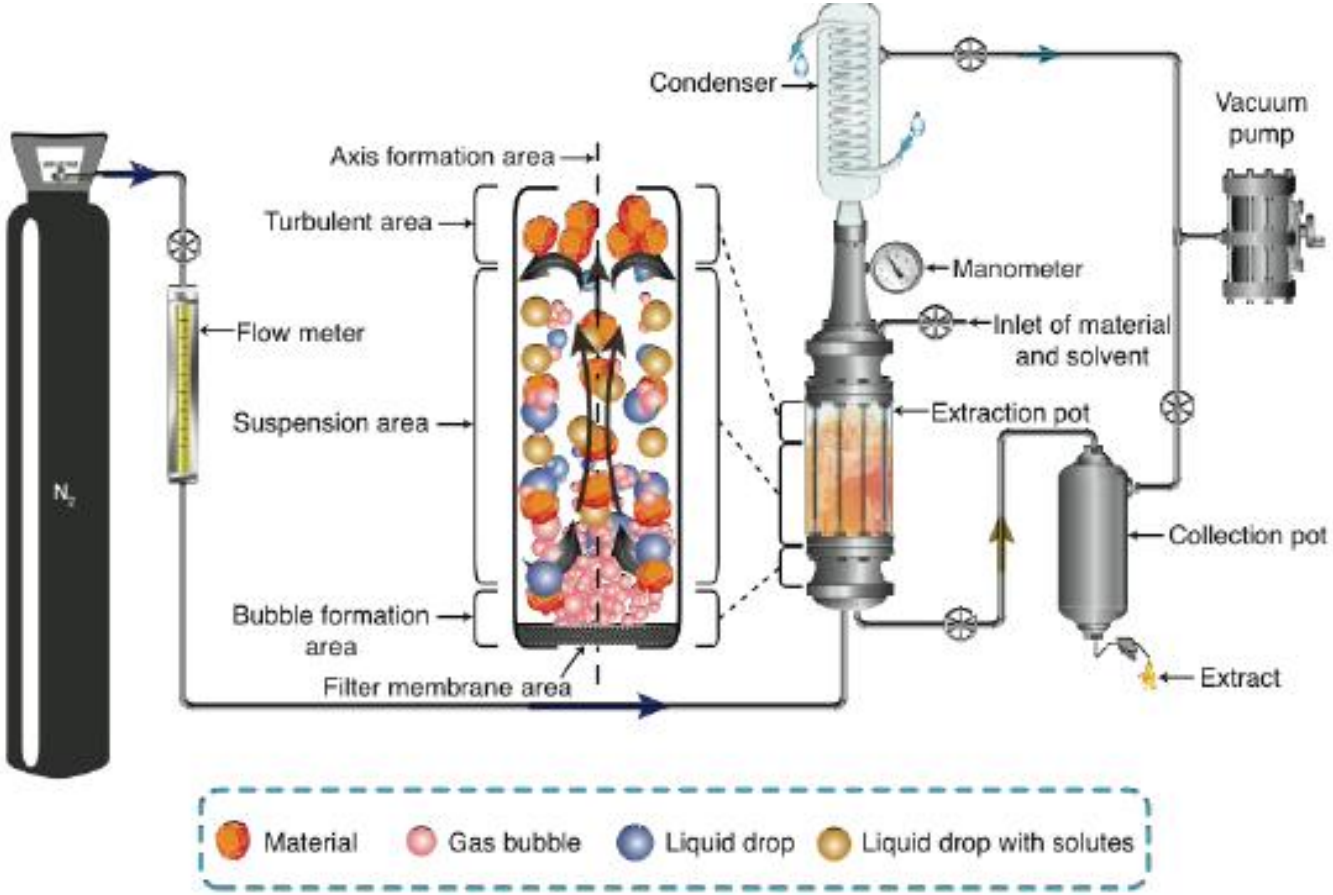


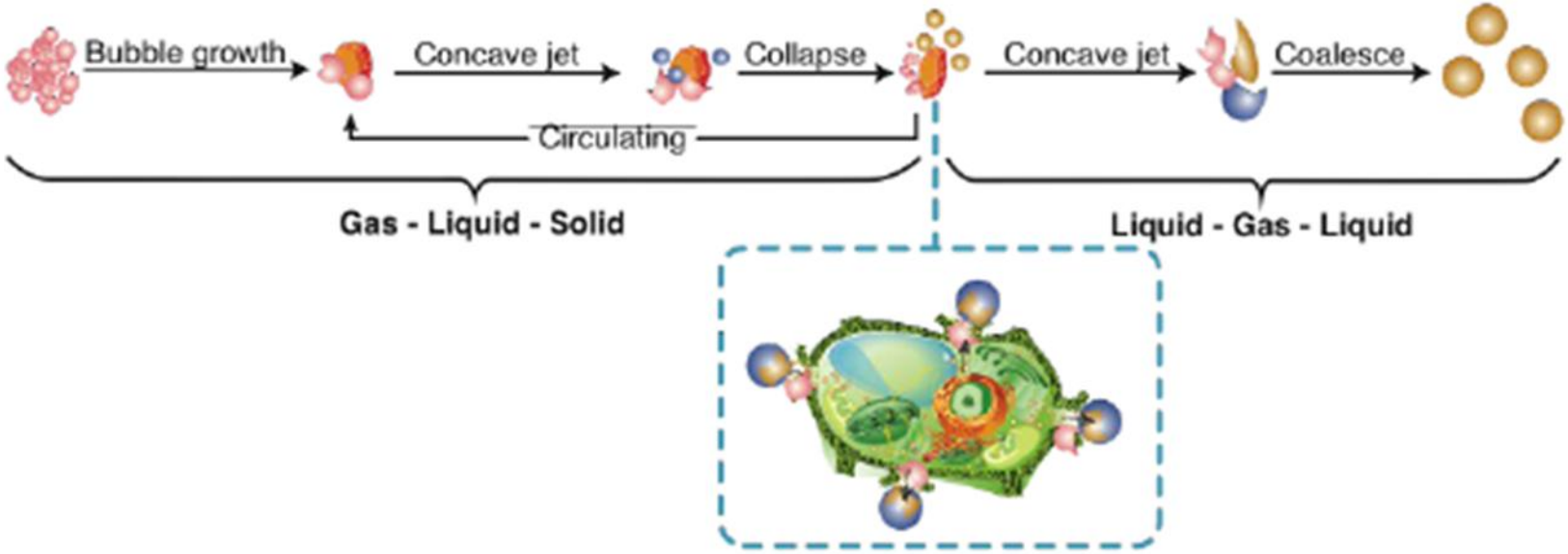
Concentration of proteins, and total phenolic compounds versus time of extraction, *t*, pulsed electric fields (PEF) and high voltage electrical discharges (HVED)-assisted extraction and conventional extraction (CE) at *T* = 20 °C and pH = 7

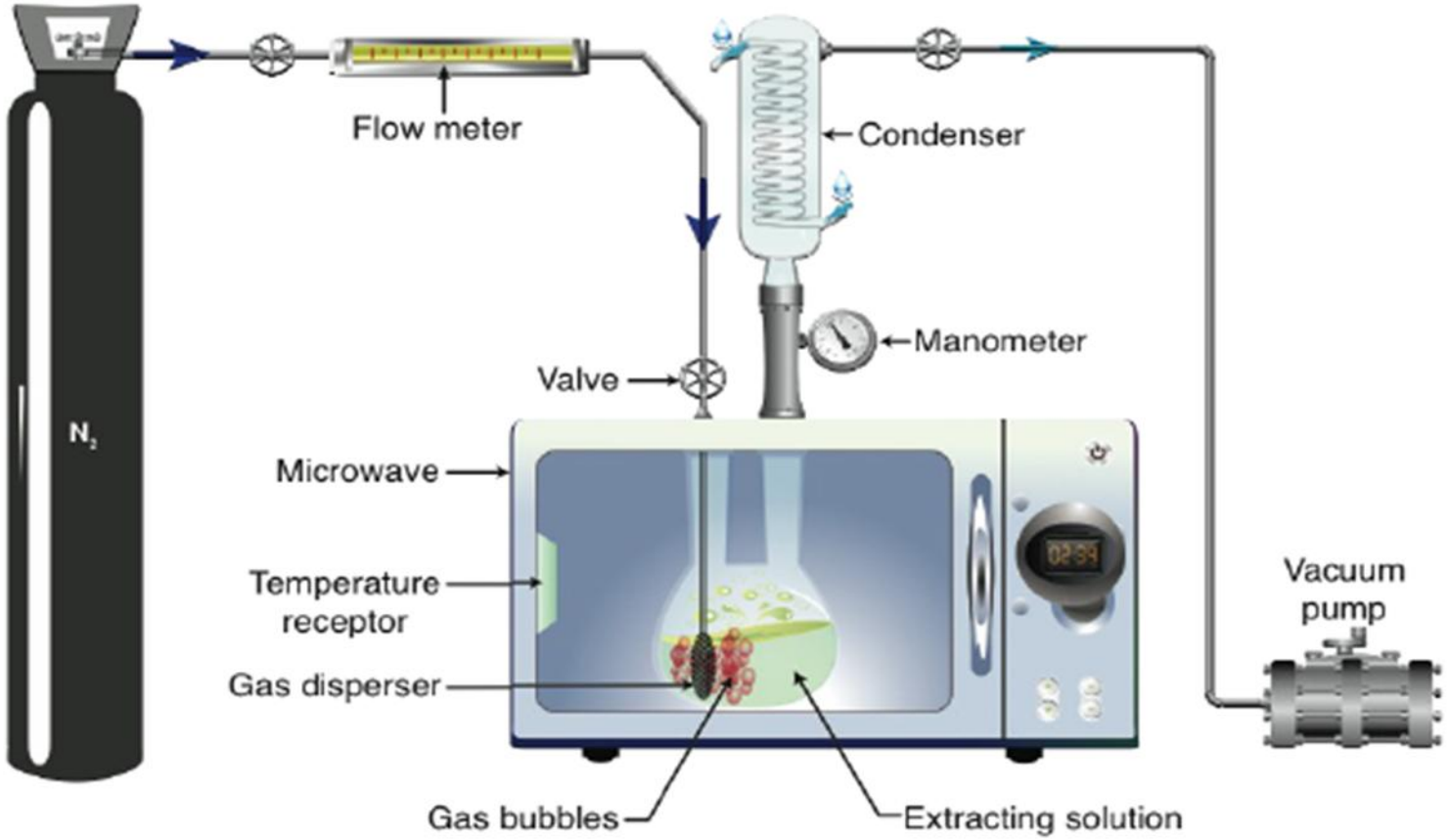


Parniakov et al., (2015)

Negative pressure cavitation



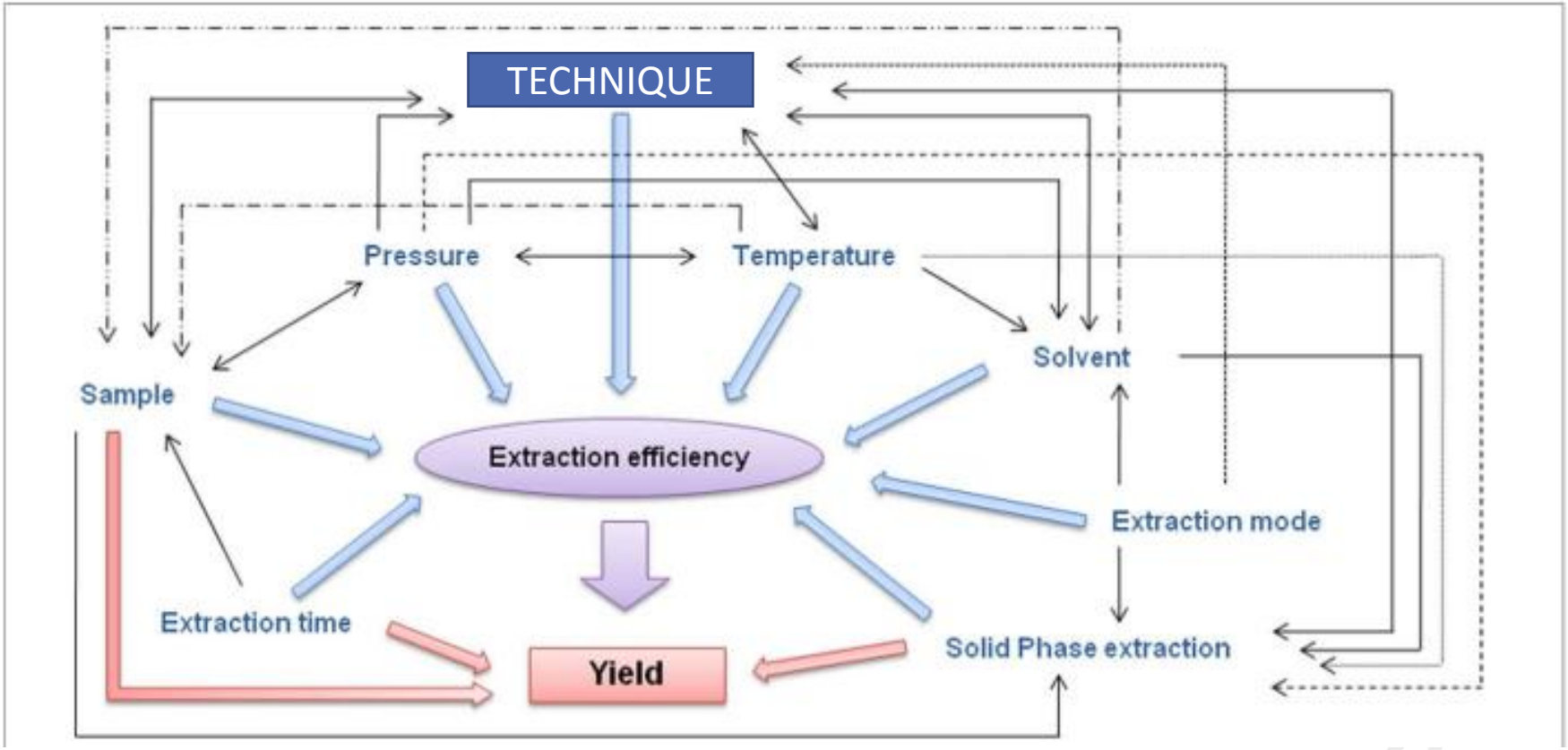

Bubble growth
Concave jet
Collapse
Concave jet
Coalesce
Circulating
Gas - Liquid - Solid
Liquid - Gas - Liquid


Flow meter
Condenser
Manometer
Valve
N_2
Microwave
Temperature receptor
Gas disperser
Vacuum pump
Gas bubbles
Extracting solution

Combination techniques

Combination	Extraction conditions	Matrix and target compound
Supercritical fluid extraction and ultrasound	Ultrasonic power: 180 – 360 W; extraction time (60 – 240 min); Ultrasonic frequency of 20 kHz SFE: 15 ± 0.5 MPa and temperature of 40 ± 3 °C, CO_2	Capsaicinoids and phenolics from malagueta pepper
Supercritical fluid extraction and ultrasound	SFE: temperature (40, 50 and 60 °C), pressure (15, 20 and 25 MPa), and ultrasound power (0, 200 and 400 W). Ethanol and water as co-solvent	Antioxidants and anthocyanins from the blackberry bagasse
Supercritical fluid extraction and ultrasound	15 min ultrasound-assisted extraction (static extraction) followed by SC–CO_2 extraction, water content in ethanol modifier (60–100%)	Oleanolic acid and Ursolic acid from Scutellaria barbata
Microwave and Ultrasound	Ultrasonic probe made of polyether ether ketone (frequency 25 kHz; power 60W); Microwave 100 W) Temperature 45^{o}C; extraction time 1 h	Oil from soybean germ and Seaweeds
Microwave and Ultrasound	microwave power 98W; Microwaves delivered to ultrasonic bath operating at 40 kHz and power of 50 W	Lycopene from tomato paste
High pressure and Ultrasound	Ultrasonic bath 47 kHz ultrasonic extraction experiments assisted and non-assisted by pulsed hydrostatic pressure	Soluble matter from mate leaves

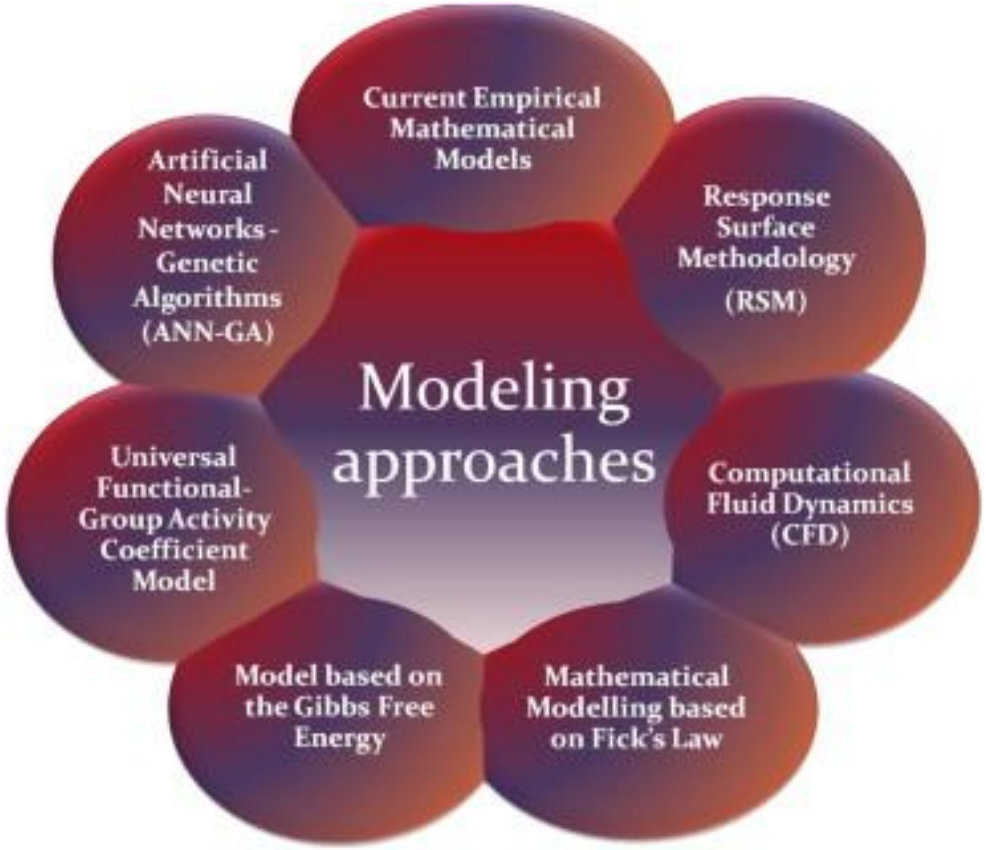
Factors affecting extraction yield



Factors affecting extraction yields

External factors	Key facts
Extraction temperature	High temperature aid in disruption of interaction of solvent and matrix High temperature enhances solvent diffusion rates Low temperature enhances cavitation Low temperature reduces thermal impact on target compound
Extraction time	Long extraction time enhances extraction yields Long extraction time may induce undesirable changes in the extracted compound.
Solvent properties	Viscous solvent reduces diffusion Volatile solvent may evaporate if extraction is carried out at higher temperature for long duration Polarity and solubility of target compound in the solvent
Matrix	Particle size Solvent matrix interaction Ratio of solvent to matrix

Modelling tools



Conclusions

- ✓Clear recent scientific advances in highlighting the potential applications of novel extraction techniques.
- ✓Novel techniques will be required to address key emerging challenges faced by the food industry.
- ✓Further research required to facilitate industry adoption of recent advances while convincing end users.

DISCLAIMER:

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This presentation reflects only
the opinion of authors and not the opinion of European Commission.

FOOD stars
Innovative Food Product Development Cycle: Frame for Stepping Up Research Excellence of FINS

European Commission

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