Recovery of Fibers from Aromatic Plant Residues via Advanced Extraction Technologies

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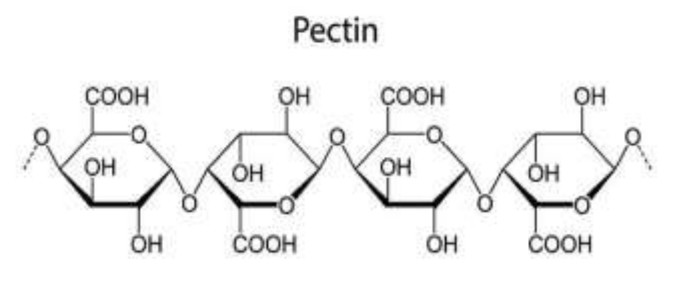
Dietary fiber (DF)

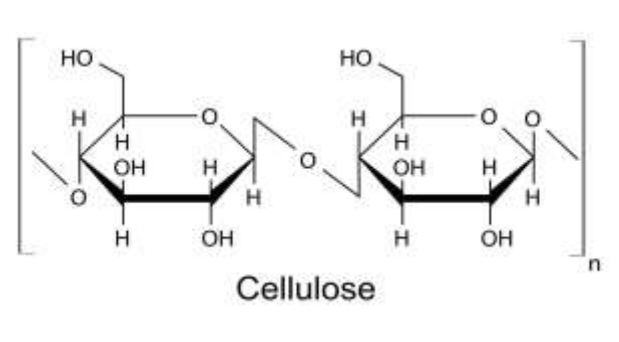
- significant physiological and functional properties
- improve hydration properties, oil holding capacity and rheological characteristics of foods
- health benefits

(preventing metabolic

Sources & Classification

- Main sources: cereal polysaccharides
- Also, fruit & vegetable residues
- Non-conventional source alternative: Aromatic plant residues
- Soluble & Insoluble DFs
- Pectin, β-glucan, gums, mucilage
- Cellulose, hemicellulose, lignin
- Resistant starch
- Each fraction: different physiological and technological





Aromatic plant residues

- Medicinal and aromatic plants (MAPs):

a rapidly growing global industry

- generation of nearly ***30 million tons of waste*** annually.
- rich in bioactives and also structural biopolymers like dietary fibers.
- considered as underutilized biosources,

Aromatic plant residues

- <u>Circular bioeconomy framework:</u>
- Valorization of these residues into high-value products across food, chemical and agricultural applications.
- Polyphenols, flavonoids, polysaccharides are among the bioactives.
- Functional & marketable products.
- Transformation of waste

Aromatic plant residues

- **Composition of MAP residues:**

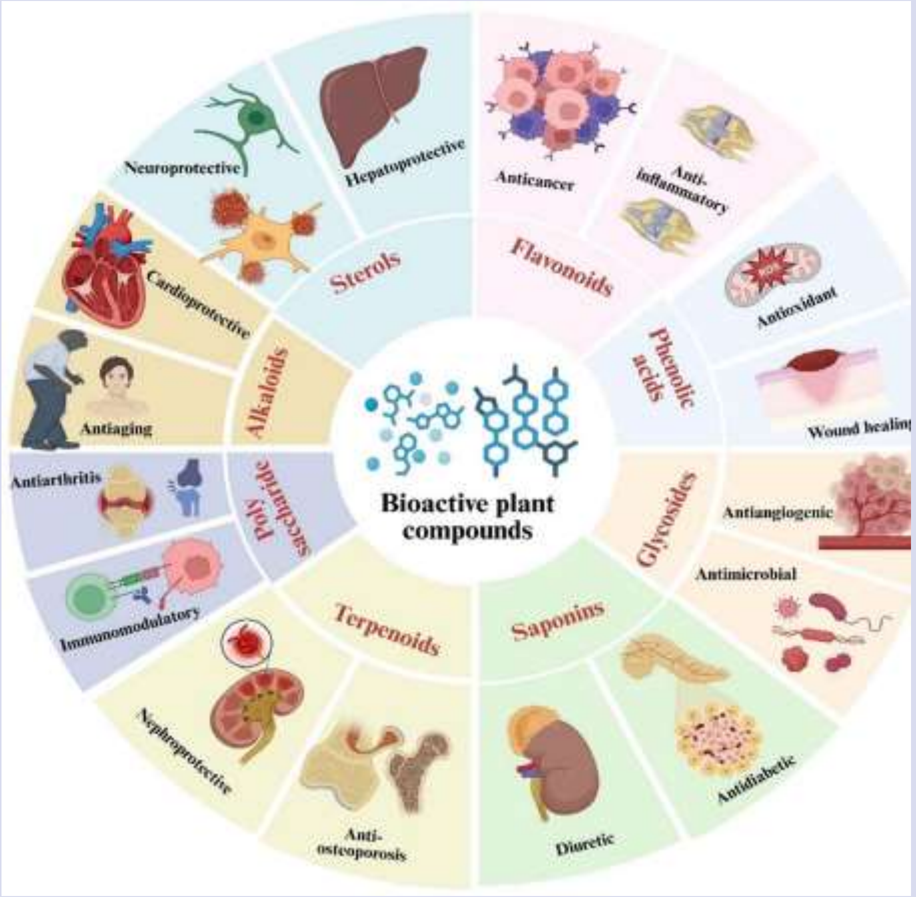
Structural polysaccharides (cellulose, hemicellulose, pectin)

Lignin

Phenolic compounds

Residual essential oil fractions

Proteins & minor components



Aromatic plant residues

- TÜBİTAK bilateral collaboration project with Bulgarian Academy of Sciences (BAS) 125N131

(Institute of Organic Chemistry with Center of Phytochemistry)

- **Focus :** LAVENDER & LEMON BALM, which are widely cultivated in Bulgaria and commonly used in essential oil production.

- Significant amount of processing residues, having valuable bioactives such as phenolics & DF.

Applications of DFs

- **Food Industry**

Stabilizers & thickeners

Texture & mouthfeel improveme

Fat replacers

- **Pharmaceutical & Nutraceutic**

Controlled release systems

Prebiotic effects

- **Cosmetic Industry**

Moisturizing & film-forming a

Anti-aging & protective effec

- **Agriculture & Biomaterials**

Biofertilizers & soil conditi

Biodegradable materials

Extraction of DFs

- Polysaccharides & other polymers: cross-linked to the cell wall with other components
- Water or more complex extraction media needed (alkali solutions or enzymatic hydrolysis) them to be removed.
- **Extraction methods :** chemical, thermal, mechanical, enzymatic..
- Some physical, mechanical and/or enzymatic pre-treaetments are also needed.

Extraction of DFs

Physical & Mechanical Pretreatments

Grinding, micronization, microfluidization

High-pressure homogenization

Structural disruption of fibers

Effects on Functionality

Increased surface area

Exposure of hydroxyl groups

Changes in hydration properties

Key Considerations

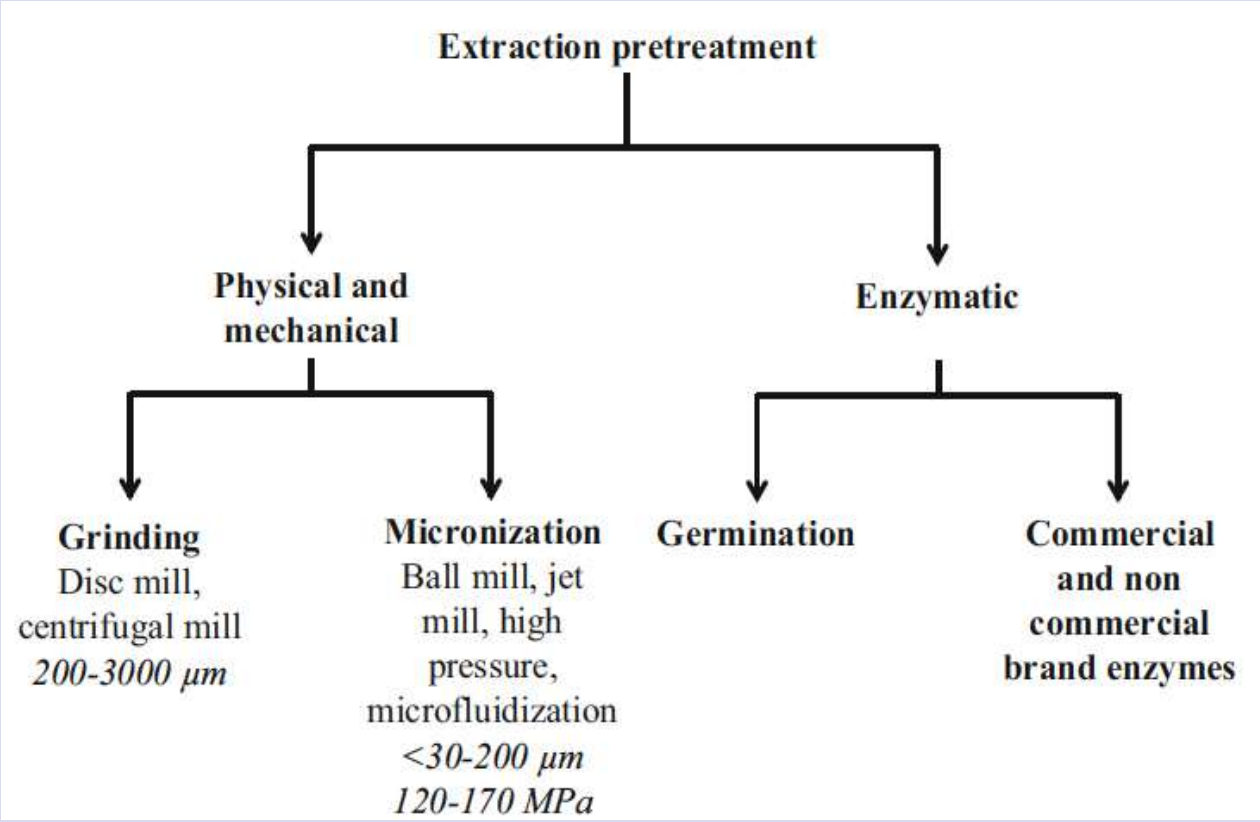
Particle size reduction may ↓ or ↑ water retention

Balance between structure damage & functionality

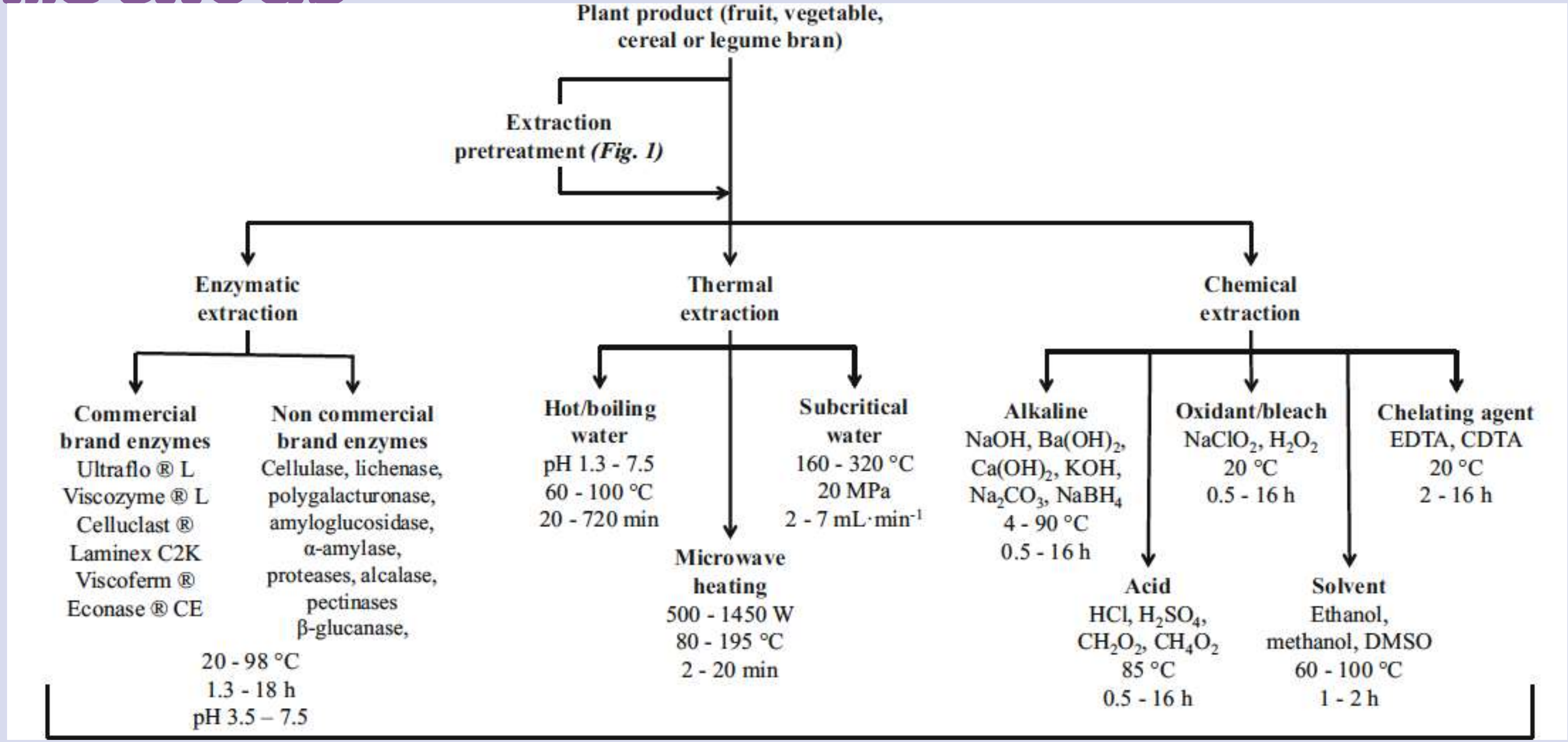
Enzymatic Pretreatments

Improve extraction efficiency

Target specific cell wall components



Extraction of DFs-conventional methods



Extraction of DFs-conventional methods

ADVANTAGES

LIMITATIONS

- Well-established and widely used
- Simple operation
- No need for sophisticated equipment
- Scalable at industrial level

- Long extraction time
- High temperature requirement
- High solvent consumption
- Possible degradation of sensitive compounds

Extraction-Emerging Technologies

•Microwave-assisted extraction

•Ultrasound-assisted extraction

•High-pressure processing

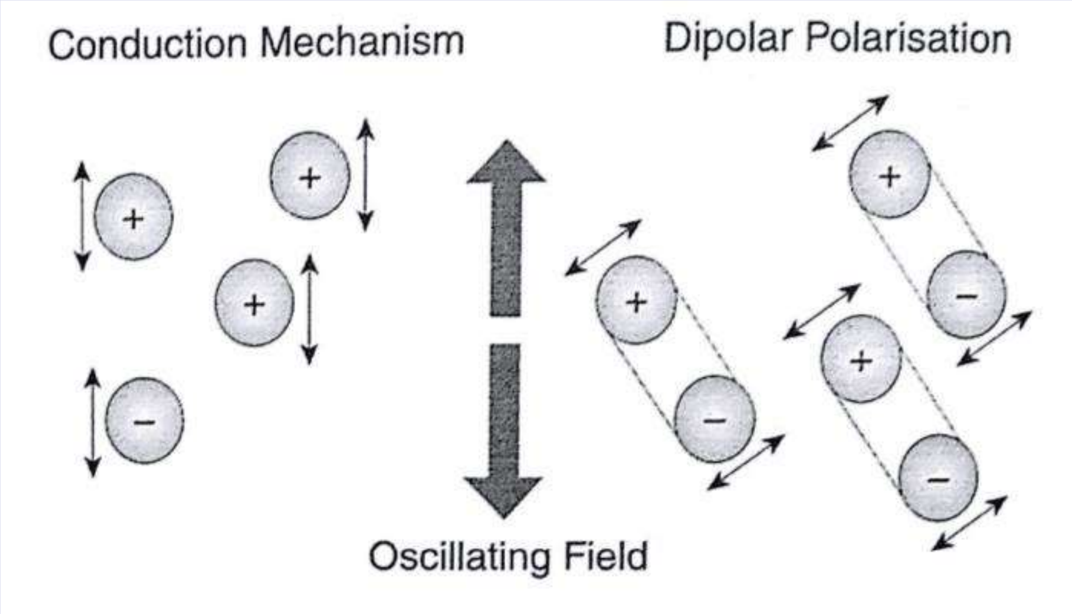
•Enzyme-assisted extraction

Key Advantages

•Reduced extraction time
•Lower temperature
•Higher efficiency
•Better preservation of bioactives

Microwave-Assisted Extraction (MAE)

- **non-conventional, green extraction technology** that addresses many of the limitations of conventional methods by enabling rapid and efficient recovery of bioactive compounds from complex plant matrices.



- Microwave heating is based on the interaction of electromagnetic waves (300 MHz–300 GHz) with matter. Two main mechanisms are responsible:

1. Ionic conduction

Microwave-Assisted Extraction (MAE)

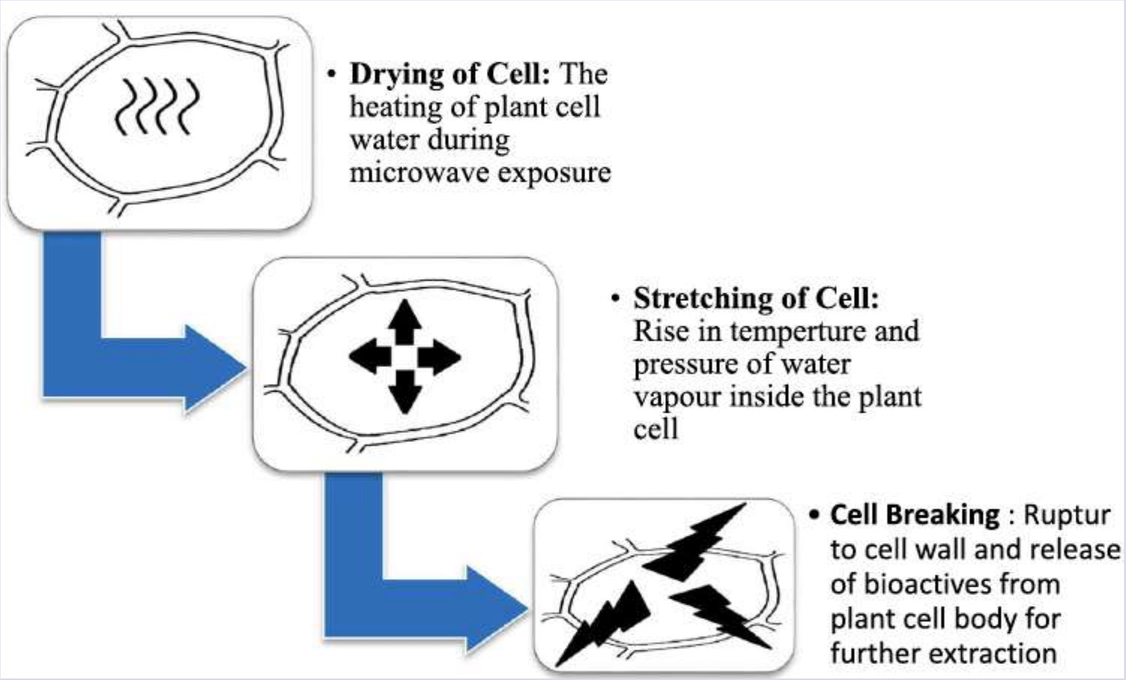
- Generally, 2 frequencies are used (**915 and 2450 MHz**) in Microwave Processing.
- The dissipation factor value (tan δ) : a measure of the capacity of extraction with which different solvent heat up under microwave.

$\tan \delta = \varepsilon'' / \varepsilon'$ (ε″: dielectric loss factor and ε′: dielectric constant)

Microwaves provide;

- Internal and volumetric heating
- Rapid pressure build-up within cells
- **Cell wall rupture and structural disruption**
- Faster release of target compounds
- Shorter extraction time

Microwave-Assisted Extraction (MAE)



- Plant cells contain **intracellular moisture**, even in dried materials
- Microwave energy interacts with **polar molecules (mainly water)**
- Rapid heating causes **instant evaporation of internal moisture**
- Evaporation leads to **high internal pressure inside the cell**
- Cell wall is **weakened and ruptured from inside**
- Disruption enables **release of intracellular bioactive compounds**
- Result: **Enhanced mass transfer and higher extraction yield**

Instrumentation

Microwave generator (magnetron):

produces microwave energy

Waveguide: transfers microwave energy

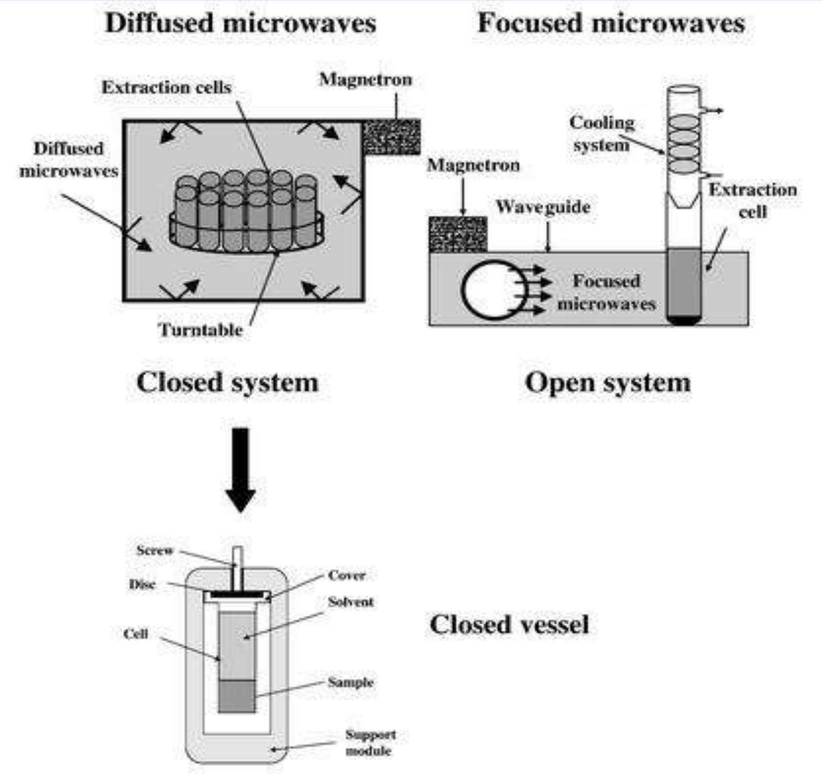
into the cavity

Circulator: directs and controls

microwave flow

Applicator (extraction vessel):

contains the sample



Instrumentation

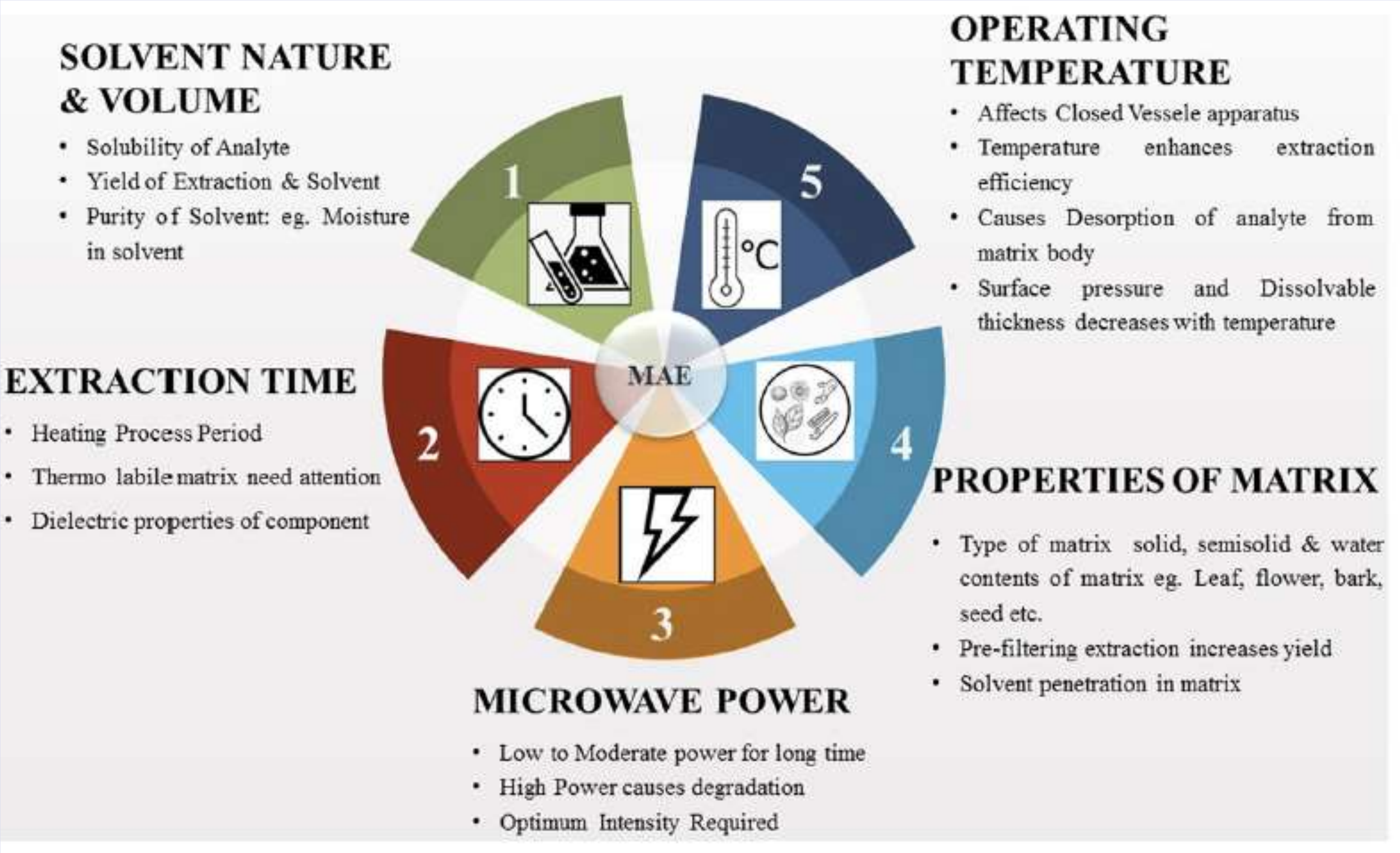
Closed Vessel Systems (Multimode)

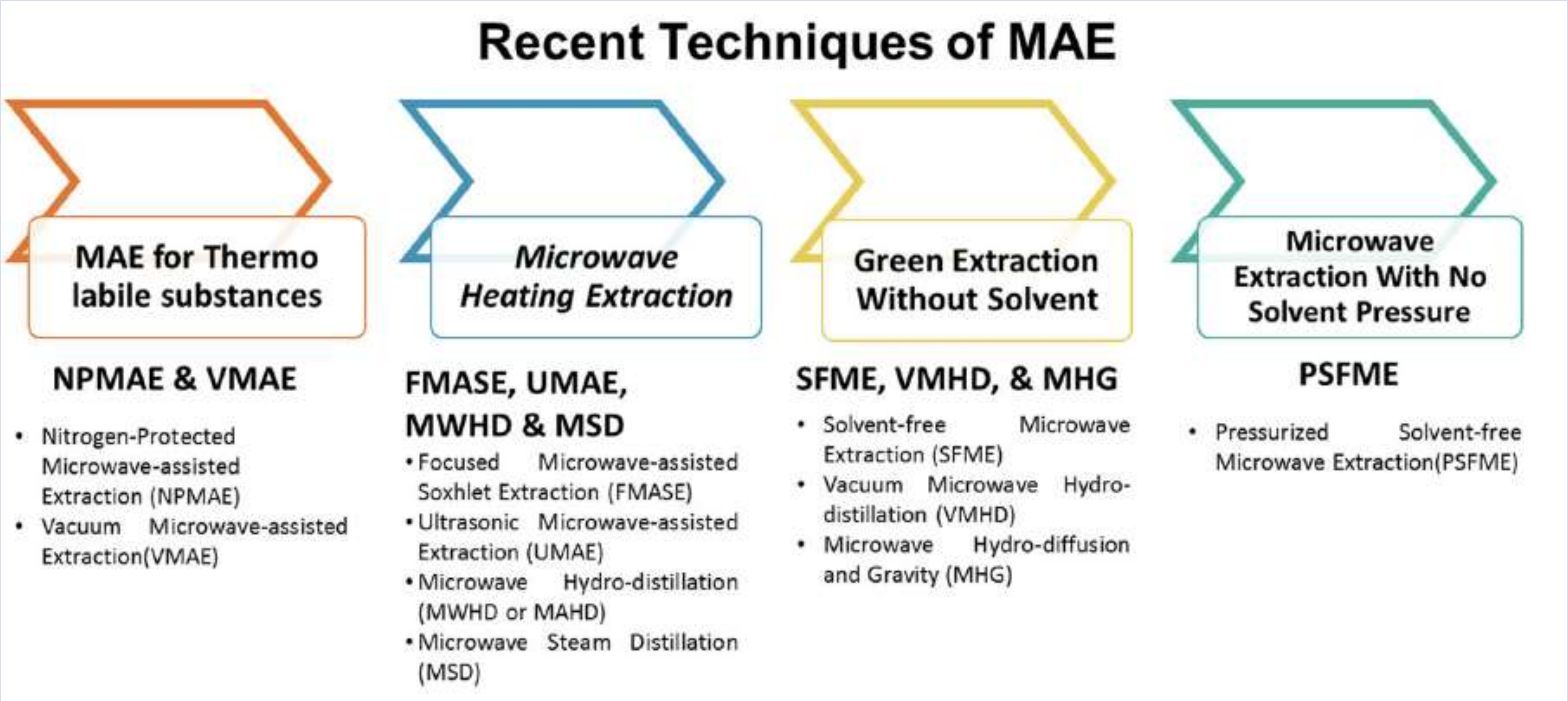
- Operate under pressure
- Uniform microwave distribution
- Higher temperature and extraction efficiency
- Risk of degradation for thermolabile compounds

Open Vessel Systems (Monomode)

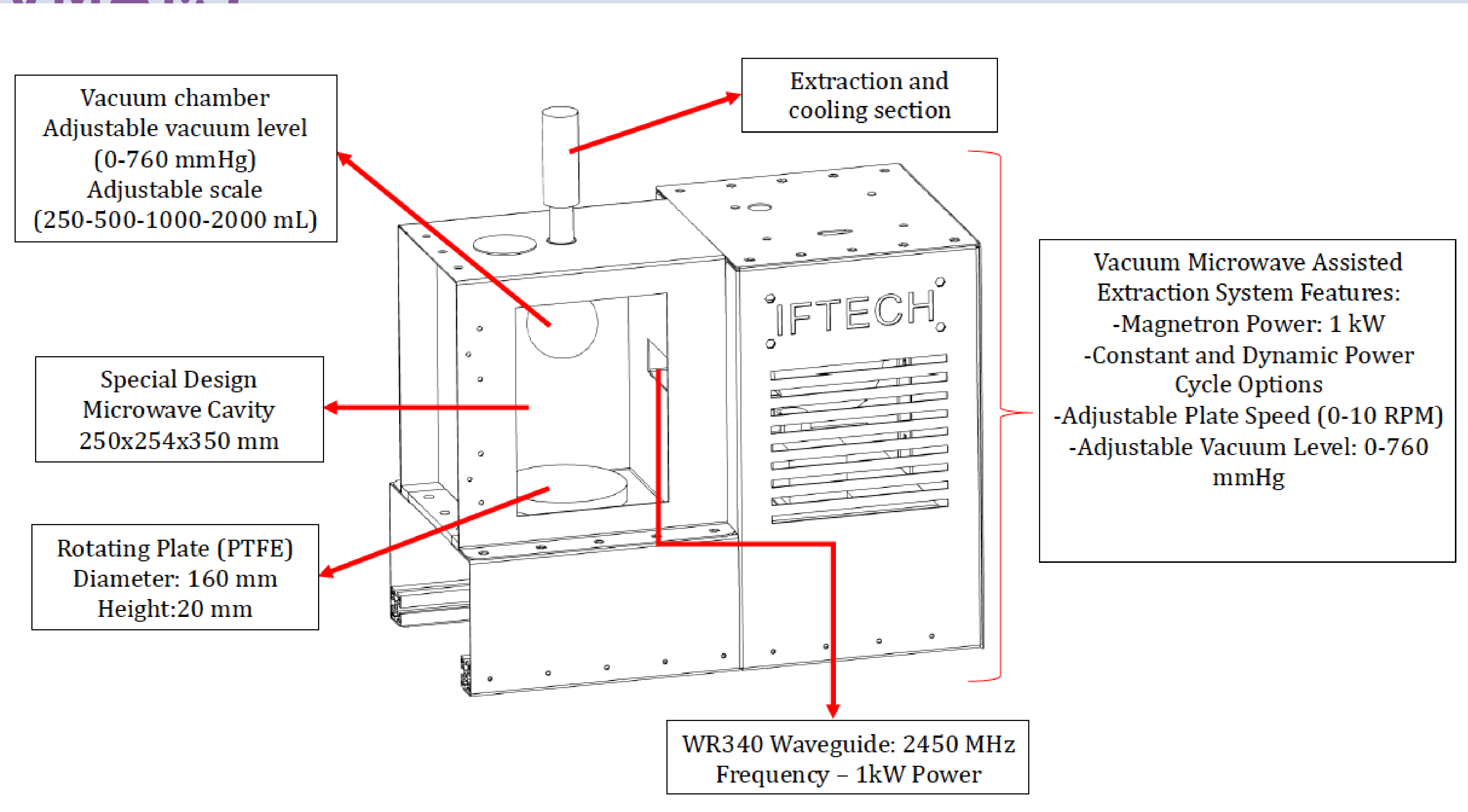
- Operate at atmospheric pressure
- Focused microwave field
- More controlled and homogeneous heating
- Suitable for thermolabile compounds

Factors affecting MAE




Recent Techniques of MAE
MAE for Thermo labile substances
NPMAE & VMAE
• Nitrogen-Protected Microwave-assisted Extraction (NPMAE)
• Vacuum Microwave-assisted Extraction(VMAE)
Microwave Heating Extraction
FMASE, UMAE, MWHD & MSD
• Focused Microwave-assisted Soxhlet Extraction (FMASE)
• Ultrasonic Microwave-assisted Extraction (UMAE)
• Microwave Hydro-distillation (MWHD or MAHD)
• Microwave Steam Distillation (MSD)
Green Extraction Without Solvent
SFME, VMHD, & MHG
• Solvent-free Microwave Extraction (SFME)
• Vacuum Microwave Hydro-distillation (VMHD)
• Microwave Hydro-diffusion and Gravity (MHG)
Microwave Extraction With No Solvent Pressure
PSFME
• Pressurized Solvent-free Microwave Extraction(PSFME)

Vacuum Microwave Assisted E. (VMAE)



Advantages of VMAE

- Lower extraction temperature
- Reduced thermal degradation of bioactives
- Enhanced mass transfer efficiency
- Faster extraction process
- Lower solvent consumption
- Improved extraction yield
- Better preservation of functional properties
- More energy-efficient process

Two-step VMAE Strategy

In our project;

- **Step 1:** Recovery of phenolic compounds from Lavender and Lemon balm essential oil residues
- **Step 2:** Recovery of pectic polysaccharides/oligosaccharides from alcohol-insoluble residues from the Step 1.
- Integration of enzymatic (cellulase) pre-treatment before

Step 1: Recovery of phenolic compounds

Optimization of the extraction conditions is crucial

Response surface methodology (RSM) (Box-Behnken design)

Independent variables:

- Vacuum pressure (200-500 mmHg)
- Extraction time (5-15 min)
- Ethanol concentration (20-70 %)
- Sample-to-solvent ratio (1:10-1:30)

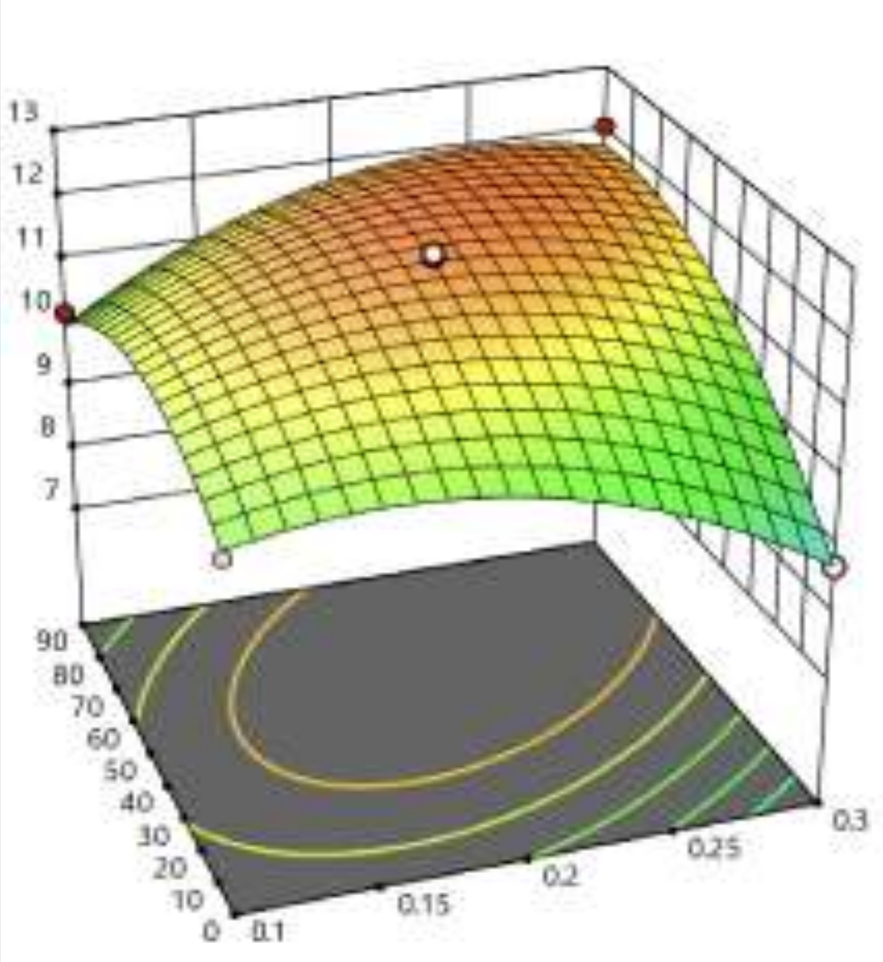
Step 1: Key findings (study continues..)

•**TPC (mg GAE/g)**
Lavender: 12.76 – 19.76
Lemon balm: 38.2 – 67.9

•**DPPH (mM TE/kg)**
Lavender: 66.63 – 243.40
Lemon balm: 325.9 – 857.8

•**CUPRAC (mM TE/kg)**
Lavender: up to 1,087.5
Lemon balm: up to 5,567.3

•**TFC (mg QE/g)**
Lavender: 0.69 – 3.14
Lemon balm: 0.4 – 3.5



Step 2: VMAE of pectic polysaccharides (ongoing)

- Raw material: Alcohol Insoluble Residue (AIR) (from step 1)
- Enzymatic pretreatment (Cellulase enzyme)
- Followed by VMAE

Independent Variables:

- **pH:** 4 – 7
- **Extraction time:** 5 – 15 min
- **Vacuum pressure:** 200 – 600 mmHg
- **Solid-to-solvent ratio:** 1:10 – 1:40
- **Enzymatic incubation time:** 60 – 120 min

Response variables:

- Pectin yield (%)
- Uronic acid content (%)

Conclusion

- Aromatic plant residues are **valuable and underutilized bioresources**
- **Two-step VMAE strategy** enables recovery of multiple bioactives
- High phenolic yield & antioxidant capacity (Step 1
- **Pectic polysaccharide recovery (Step 2 – ongoing)**

Potential Applications of Recovered Polysaccharides

Food systems: stabilizers, thickeners, texture modif

Functional foods: dietary fiber enrichment

Encapsulation systems: carriers for bioactives

Sustainable materials: biodegradable formulations

Thank you for listening

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