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AGRIMA

The Hidden Treasure in Grape Stalks: A Green Extraction Quest

AGRIMA Summer Camp | July 7 – 11, 2025 | Marseille, France

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Agri-food Waste Management for Sustainable bio-economy through Higher Education curricula and upskilling

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Goals

AGRIMA aims to foster universities' **capacity building** for the **green transition** through **innovative practices and higher education curricula updating in agri-food waste management** for the **circular bioeconomy**.

AGRIMA addresses:

1. **Advancing pedagogical methods** for industrial agri-food waste valorisation **based on business-academia synergies**.
2. **Integrating citizen science** in bio-economy-enhanced waste valorisation as a means of **civic engagement and environmental advocacy**.

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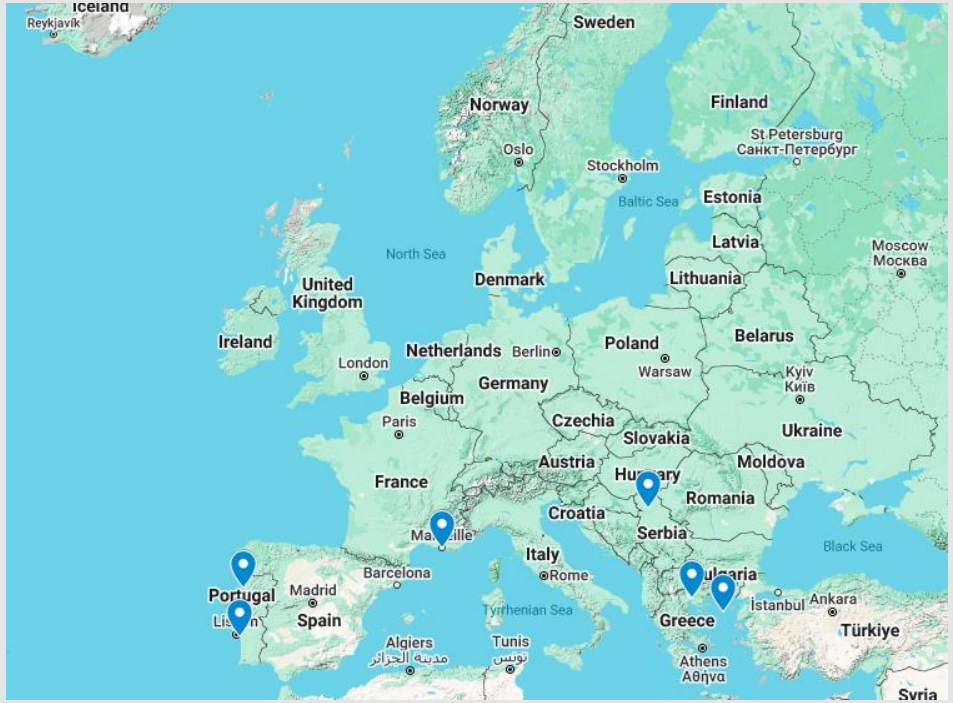

Partners

amU Aix Marseille Université

AVIPE Associação de Viticultores do Concelho de Palmela

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2 Materials and methods

3 Results

4 Conclusion

5 Future prospects

1. Introduction

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Background information

- ✓ Portugal: 3.1 % of global wine production (2024)
- ✓ Challenges in waste management
- ✓ 30 % of the original grape weight → by-products
  - Grape pomace, grape stalks and wastewater
  - Fertilizer or animal food
  - Rich in bioactive compounds

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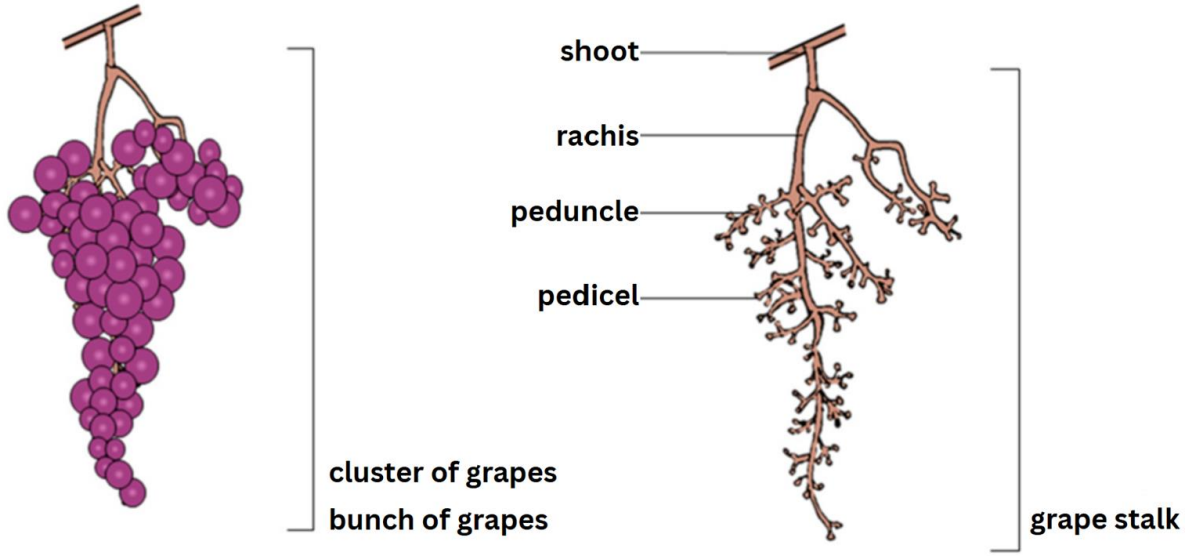
Objective and scope

- ✓ Develop and optimize a green extraction method
- ✓ Recovery of polyphenolic compounds from grape stalks
- ✓ Ecological and economic benefits



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2. Materials and methods

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Overview of the main phases

Phase 1: Identification of the most significant factors affecting solid-liquid extraction (SLE)

Phase 2: Determination of SLE optimal extraction conditions

Phase 3: Assessment of extraction methods and grape varieties influence on antioxidant properties

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Phase 1: Identification of the most significant factors affecting SLE

- ✓ Reducing the total number of experiments in next phases;

- ✓ Factors:

Solvent concentration
Extraction time
Extraction temperature
Solid-lo-Liquid Ration
Stirring spreed

- ✓ TPC results determine the influence of each factor

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Phase 2: Determination of SLE optimal extraction conditions

- Response surface methodology (RSM)
  - Model potential curvature and fine-tune the significant variables
- Significant variables
  - Solvent concentration
  - Extraction temperature
  - Solid-to-liquid ratio
- TPC determination

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Phase 3: Assessment of extraction methods and grape varieties influence on antioxidant properties

- Advanced extraction techniques

Ultrasound-assisted extraction

Amplitude (%)	Time (min)
50	10
50	20
70	10
70	20

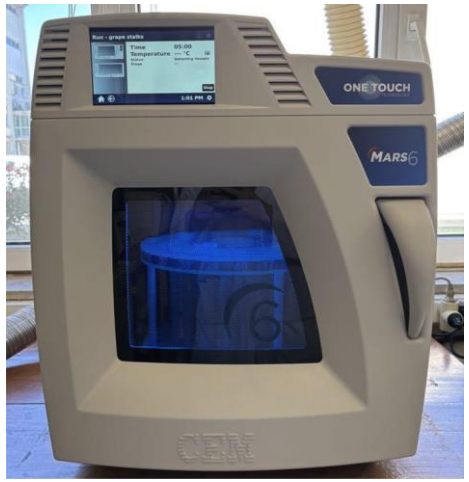
Microwave-assisted extraction (MAE)

Temperature (°C)	Time (min)
40	10
40	20
60	10
60	20

Subcritical water extraction (SWE)

Temperature (°C)	Pressure (bar)
120	10
120	20
150	10
150	20

(Time: 20 minutes)



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Phase 3: Assessment of extraction methods and grape varieties influence on antioxidant properties

- Grape varieties
  - *Tinta Miúda*
  - *Cerceal Branco*
- Evaluation assays
  - TPC (mg GAE/g dw)
  - Ferric reducing antioxidant power (FRAP) (mg AAE/g dw)
  - ABTS radical scavenging activity (mg AAE/g dw)

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3. Results

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Results

Phase 1: Identification of the most significants factors affecting SLE

- Solvent concentration (% ethanol) and solid-to-liquid ratio
  - Statistically significant effect
  - Most influential effects
- Extraction temperature
  - Moderate but statistically significant effect
- Extraction time and stirring speed
  - No statistically significant effect
- Two-way interaction between solvent concentration and solid-to-liquid ratio
  - Statistically significant effect

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Results

Phase 1: Identification of the most significant factors affecting SLE

	Solvent concentration (% EtOH)	Solid-to-liquid ratio (g/mL)	Extraction temperature (°C)	Extraction Time (min)	Stirring speed (rpm)	TPC (mg GAE/g dw)
1	80	1:100	30	120	250	23.0
2	40	1:100	60	120	250	53.0
3	80	1:20	30	30	250	11.8
4	40	1:20	60	30	250	14.8
5	40	1:20	30	30	500	14.6
6	40	1:20	30	120	250	15.2
7	40	1:100	60	30	500	45.7
8	40	1:100	30	120	500	43.3
9	40	1:20	6	120	500	14.5
10	40	1:100	30	30	250	39.4
11	80	1:20	60	120	250	14.0
12	80	1:20	60	30	500	12.8
13	80	1:100	60	30	250	28.1
14	80	1:100	30	30	500	17.9
15	80	1:100	60	120	500	33.5
16	80	1:20	30	120	500	12.5

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Phase 2: Determination of SLE optimal extraction conditions

Optimization results for TPC extraction:

Solution	Solvent (% ethanol)	Ratio	Temperature (° C)	TPC (mg GAE/g dw) Fit	Composite desirability
1	43.6364	1:150	75	53.1907	0.997053
2	43.6364	1:150	60	50.4847	0.924115

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Phase 2: Determination of SLE optimal extraction conditions

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1	43.6364	1:150	75	53.1907	0.997053
2	43.6364	1:150	60	50.4847	0.924115

- Lower extraction temperature is preferred:
  - Reduction of energy consumption
  - Decrease of the risk of polyphenol degradation
  - Reduction in TPC yield and composite desirability is acceptable compared to the advantages

- Small difference (1.21%) between predicted and observed (49.87 mg GAE/g dw) TPC

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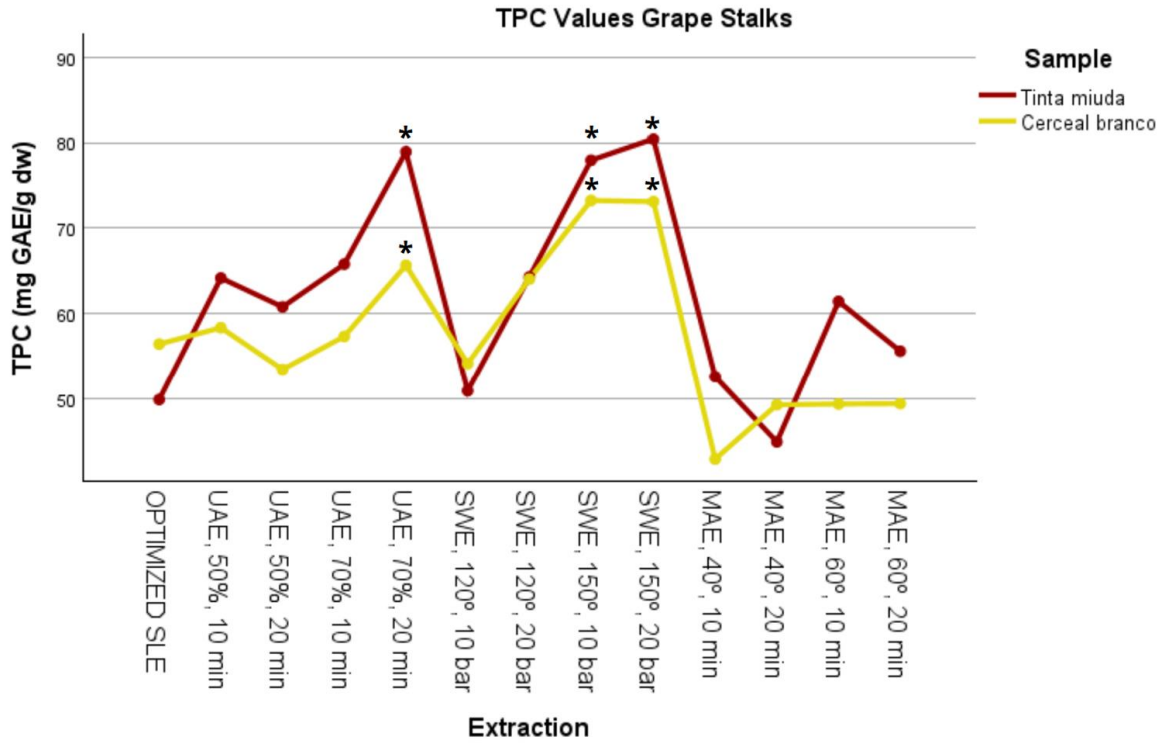
Results

Phase 3: Assessment of extraction methods and grape varieties influence on antioxidant properties



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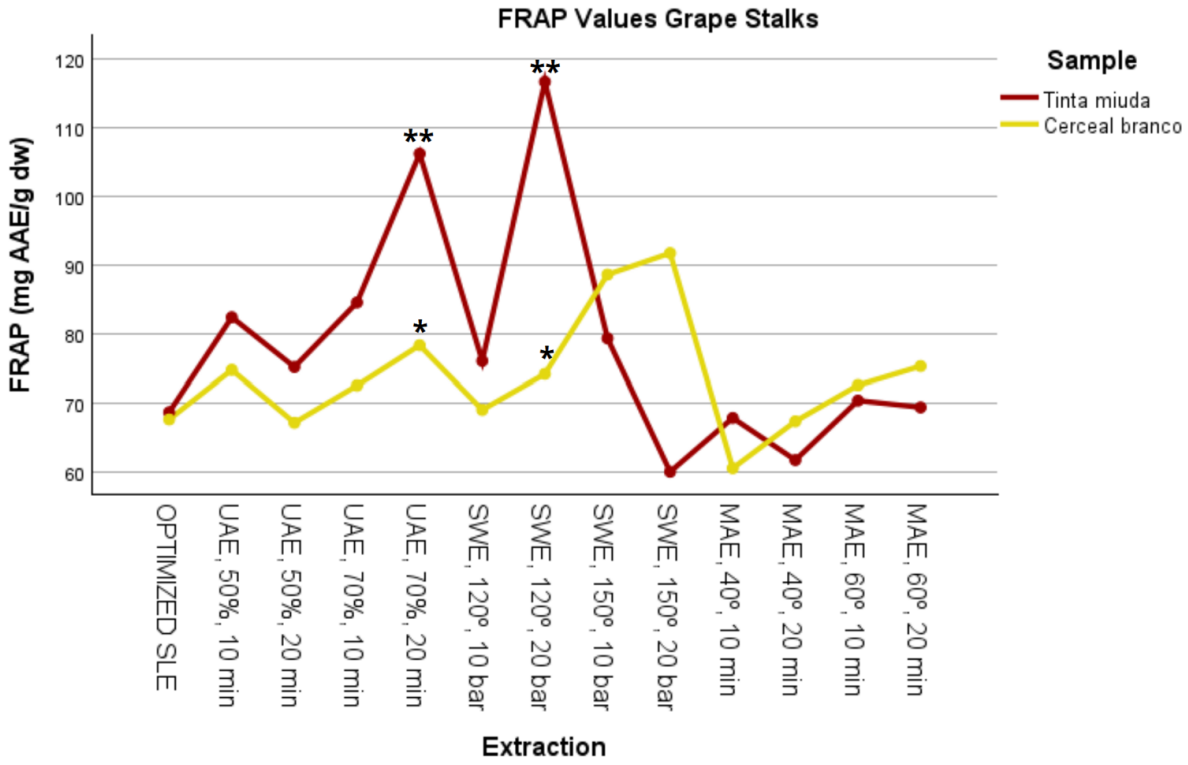
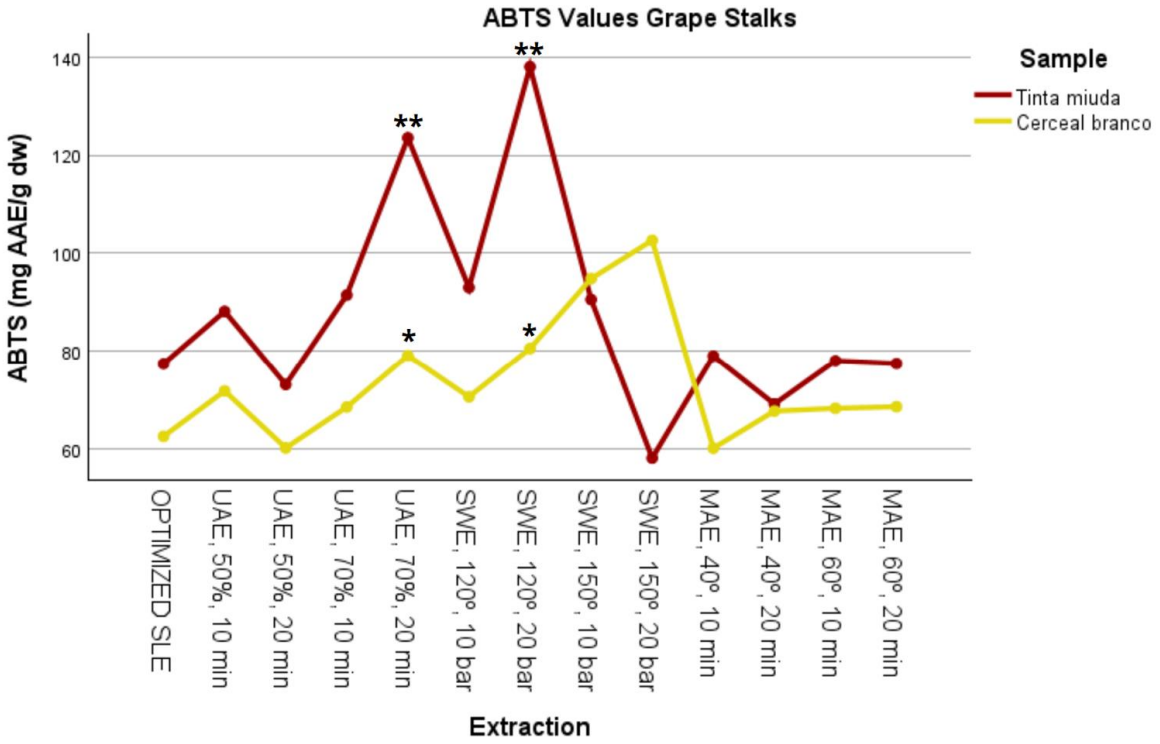
Results

Phase 3: Assessment of extraction methods and grape varieties influence on antioxidant properties





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Results

Phase 3: Assessment of extraction methods and grape varieties influence on antioxidant properties

Tinta Miúda grape stalk extracts:

	TPC	ABTS	FRAP
TPC	1	0,264*	0,391**
ABTS	0,264*	1	0,947**
FRAP	0,391**	0,947**	1

The color intensity represents the strength of the correlation, with darker shades indicating higher correlation. * The correlation is significant at the 0.05 level (2 ends). ** The correlation is significant at the 0.01 level (2 ends).

Cerceal Branco grape stalk extracts:

	TPC	ABTS	FRAP
TPC	1	0,827**	0,833**
ABTS	0,827**	1	0,839**
FRAP	0,833**	0,839**	1

The color intensity represents the strength of the correlation, with darker shades indicating higher correlation. ** The correlation is significant at the 0.01 level (2 ends).

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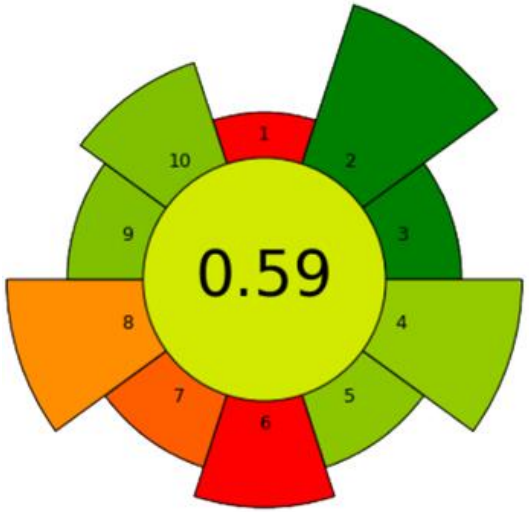
Materials and methods

Greenness evaluation



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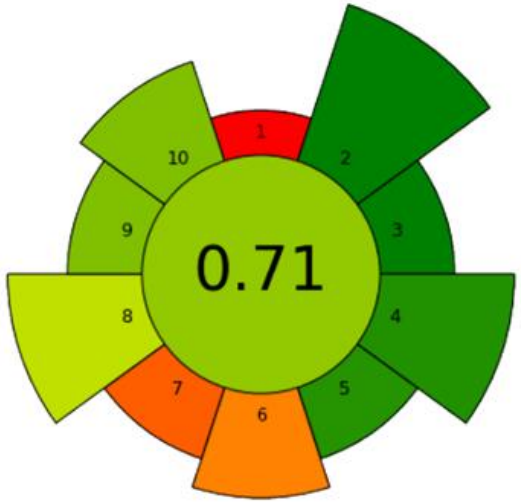

UAE

SLE

MAE

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4. Conclusion

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Conclusion

- ✓ UAE
  - Amplitude: 70 %
  - Extraction time: 20 minutes
  - Solvent concentration: 43.6 % ethanol
  - Solid-to-liquid ratio: 1:150

  - *Tinta Miúda*: TPC: 78.9 mg GAE/g dw, ABTS: 123.5 mg AAE/g dw, and FRAP: 106.2 mg AAE/g dw
  - *Cerceal Branco*: TPC: 65.6 mg GAE/g dw, ABTS: 79.1 mg AAE/g dw, and FRAP: 78.3 mg AAE/g dw

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Conclusion

- ✓ Variation in antioxidant properties among grape stalk varieties
- ✓ Potential as valuable and sustainable source of natural antioxidants
- ✓ Role of green extraction techniques in valorizing by-products

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5. Future prospects

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Future prospects

- ✓ HPLC analysis
- ✓ Incorporation of the extracts into a food product
  - Gummy bears
  - Yogurt

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