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COMPARISON OF THE EFFICIENCY OF DIFFERENT METHODS FOR HESPERIDIN AND NARIRUTIN EXTRACTION FROM ORANGE PEEL

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INTRODUCTION

Systematic comparison between microwave-assisted (MAE) and ultrasound-assisted extraction (UAE) was performed. In both MAE and UAE methods, the influence of following extraction parameters was investigated in order to perform adequate comparison; temperature (30, 50, 70 °C), extraction time (5, 10, 15 min), and solvent type (water, 80 % aqueous ethanol solution, 50 % aqueous methanol solution), at investigated microwave power (300 - 800 W) in MAE, as well with frequency (37 Hz) and power (50 W) of ultrasound in UAE method. Extracts were characterized with different levels of hesperidin and narirutin (determined and quantified by HPLC) depending on the applied process parameters and extraction method. Both methods have demonstrated predominance of hesperidin in orange peel compared to narirutin. Extraction efficiency for both compounds was enhanced in aqueous ethanol and methanol solutions.

MATERIALS AND METHODS

Microwave-assisted extraction (MAE)

Temperature: 30 – 70 °C
Time: 5 – 15 min
Solvent fraction: 10 – 30 mL
Power: 300 – 800 W

Ultrasound-assisted extraction (UAE)

Temperature: 30 – 70 °C
Time: 5 – 15 min
Solvent fraction: 10 – 30 mL
Power 50 W and frequency 37 Hz

ULTRASOUND-ASSISTED EXTRACTION (UAE)

High performance liquid chromatography (HPLC) analysis

- **Low efficiency of water extraction; additional problem: gel-forming**
- **Higher extraction efficiency with methanol: 4.09 – 12.75 µg/mL (hesperidin) and 2.46 – 4.30 µg/mL (narirutin) – parameters are not convenient for narirutin extraction**
- **The highest extraction efficiency with ethanol: 4.61 – 12.99 µg/mL (hesperidin) and 1.71 – 4.69 µg/mL (narirutin)**

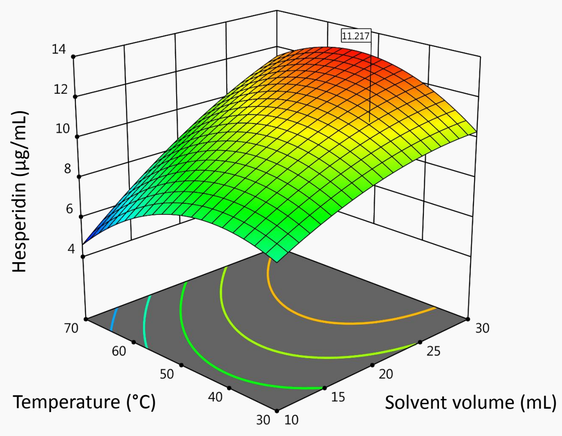
HESPERIDIN AND NARIRUTIN

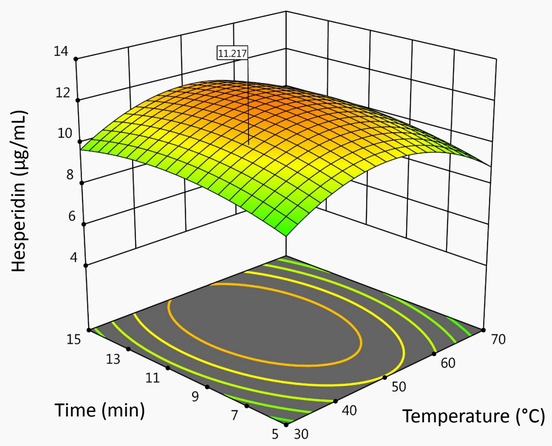
WATER **50 % METHANOL** **80 % ETHANOL**

80 % ETHANOL – RSM ANALYSIS (hesperidin)

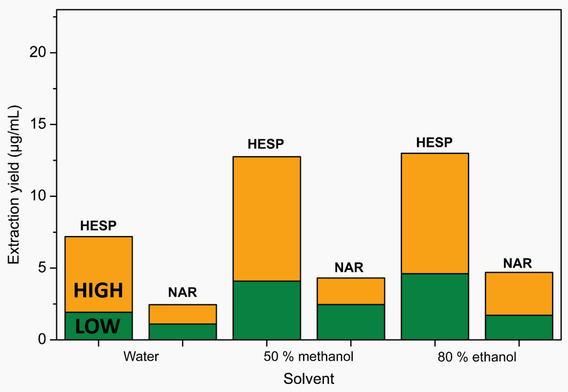
17 RUNS for each solvent (Box-Behnken design) – model for the most efficient solvent presented

$R^2 = 0.9137$





Lack of fit nonsignificant (p > .05)



Response surface methodology (RSM)

- Most efficient extraction with 80 % of ethanol – under these operating conditions no significant increase in the extraction yield of narirutin was observed.
- Temperature and extraction time had no statistically significant influence on hesperidin content.
- A dominant factor affecting extraction process is the solid/solvent ratio (statistically significant).

The highest extraction yields = 12.99 µg/mL (50 °C, 15 min, and 1:30 solid/solvent ratio, 80 % ethanol) for **hesperidin** and 4.69 µg/mL (50 °C, 10 min, and 1:20 solid/solvent ratio, 80 % ethanol) for **narirutin**

MICROWAVE-ASSISTED EXTRACTION (MAE)

High performance liquid chromatography (HPLC) analysis

- **Low efficiency of water extraction; additional problem: gel-forming**
- **Higher extraction efficiency with methanol: 4.88 – 16.16 µg/mL (hesperidin) and 0.95 – 3.81 µg/mL (narirutin) – parameters are not convenient for narirutin extraction**
- **The highest extraction efficiency with ethanol: 3.65 – 19.42 µg/mL and 0.93 – 4.20 µg/mL (narirutin) – operating parameters are not convenient for narirutin extraction**

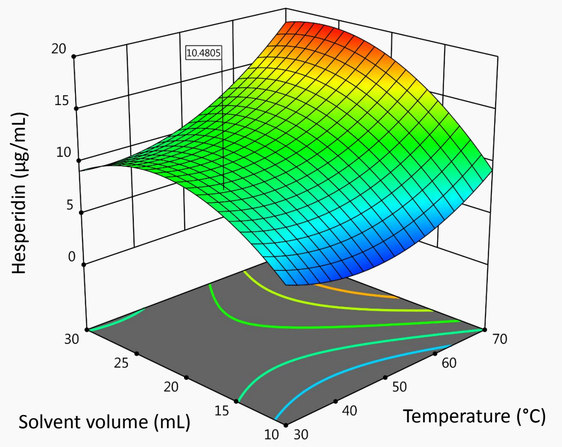
HESPERIDIN AND NARIRUTIN

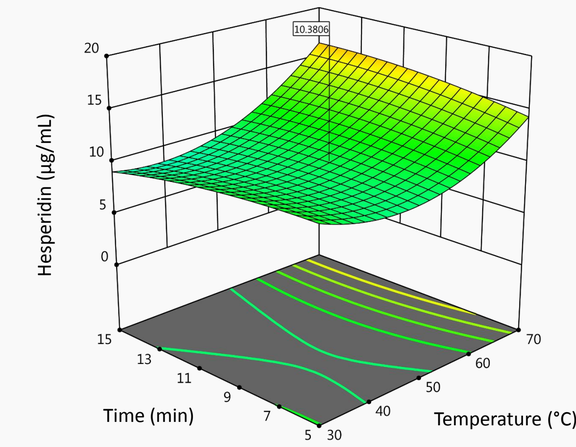
WATER **50 % METHANOL** **80 % ETHANOL**

80 % ETHANOL – RSM ANALYSIS (hesperidin)

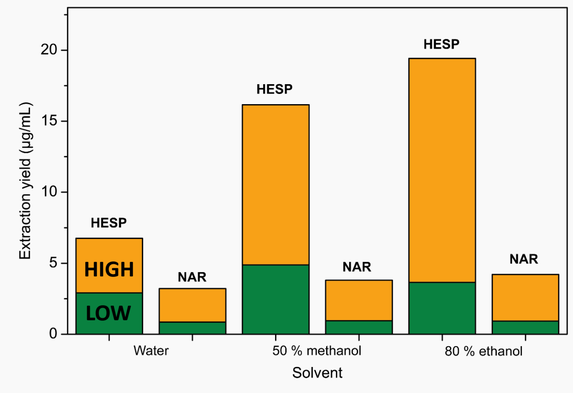
17 RUNS for each solvent (Box-Behnken design) – model for the most efficient solvent presented

$R^2 = 0.9560$





Lack of fit nonsignificant (p > .05)



Response surface methodology (RSM)

- Most efficient extraction with 80 % of ethanol – under these operating conditions no significant increase in the extraction yield of narirutin was observed.
- Temperature and solid-solvent ratio had statistically significant influence on hesperidin content.
- Extraction time had no statistically significant influence on hesperidin content.

The highest extraction yields = 19.42 µg/mL (70 °C, 15 min, and 1:20 solid/solvent ratio, 80 % ethanol) for **hesperidin** and 4.20 µg/mL (70 °C, 15 min, and 1:20 solid/solvent ratio, 80 % ethanol) for **narirutin**

CONCLUSIONS

Ultrasound-assisted extraction (UAE)

- The best extraction efficiency of selected components with 80 % ethanol; no significant increase in narirutin extraction under selected operating parameters.

1. **Water extraction** – the lowest extraction yields of hesperidin and narirutin
2. **80 % ethanol and 50 % methanol** - the medium strength of the selected process parameters should be sufficient to obtain the desired hesperidin yield.

Microwave-assisted extraction (MAE)

- The best extraction efficiency of selected components with 80 % ethanol

1. **Water extraction** – the lowest extraction yields of hesperidin and narirutin
2. **80 % ethanol and 50 % methanol** – higher temperature enhances the extraction yield of hesperidin, as well as increase in solvent volume – increase in microwave radiation and larger solvent volume can dissolve constituents more effectively.

Components are more soluble in water–alcohol solutions than in pure alcohol. A low content of phenolic substances in water extracts can be attributed to the increased activity of the enzymes (polyphenol oxidase, PPO) which degrade phenolics.

ACKNOWLEDGEMENT

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