NATURAL DEEP EUTECTIC SOLVENTS AS MAIN SOLVENT FOR THE EXTRACTION OF TOTAL POLYPHENOLS OF ORANGE PEEL

Clara Gómez-Urrios¹, Adriana Viñas-Ospino¹, Anna Penadés-Soler¹, Daniel Lopez-Malo², Ana Frígola¹, María José Esteve¹, Jesús Blesa*
Food Industry produces a large amount of food waste

High biological value

Food industry
Cosmetic industry

TOTAL POLYPHENOLS CONTENT

Green Chemistry
Find alternative to organic solvents

Ils (Ionic Liquids)

DES (Deep Eutectic Solvents)

NADES (Natural Deep Eutectic Solvents)

The aim of this study is to optimize the extraction procedure for the total polyphenol content (TPC) with NADES.
Materials and Methods

Table 1: Materials and molar ratios of the NADES

<table>
<thead>
<tr>
<th>Acronym</th>
<th>HBA</th>
<th>HBD</th>
<th>Molar Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>ChChl:Fruc</td>
<td>Choline Chloride</td>
<td>Fructose</td>
<td>1.9:1</td>
</tr>
<tr>
<td>ChChl:Gly</td>
<td>Choline Chloride</td>
<td>Glycerol</td>
<td>1:2</td>
</tr>
<tr>
<td>Bet:CA</td>
<td>Betaine</td>
<td>Citric Acid</td>
<td>1:1</td>
</tr>
<tr>
<td>LP:MA</td>
<td>L-Proline</td>
<td>Malic Acid</td>
<td>1:1</td>
</tr>
</tbody>
</table>

Table 2: Coded levels of independent variables

<table>
<thead>
<tr>
<th>Independent variable</th>
<th>Level</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquid/solid ratio</td>
<td>$X_1$</td>
</tr>
<tr>
<td>NADES (%, v/v)</td>
<td>$X_2$</td>
</tr>
<tr>
<td>Extraction time</td>
<td>$X_3$</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>-1</th>
<th>0</th>
<th>+1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquid/solid ratio</td>
<td>5</td>
<td>15</td>
<td>25</td>
</tr>
<tr>
<td>NADES (%, v/v)</td>
<td>10</td>
<td>50</td>
<td>85</td>
</tr>
<tr>
<td>Extraction time</td>
<td>5</td>
<td>15</td>
<td>30</td>
</tr>
</tbody>
</table>
Materials and Methods

**Determination of total polyphenol content by UV-vis spectroscopy**

1. Add 100 µl sample to 3 ml Na₂CO₃.
2. Add 100 µl Folin-Ciocalteau reagent and incubate for 1 hour.

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- **Optimization**
  - Design-Expert 8.0
  - SPSS® 26.0

- **Gallic acid calibration curve**

- **765 nm**

- **Mg GAE/100 g DW**

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100 µl sample

765 nm

Mg GAE/100 g DW
Results and Discussion

Total phenolic content in NADES extracts with different amount of water.
ChCl:Fruc Choline Chloride fructose. ChCl:Gly Choline Chloride Glycerol. Bet:CA Betaine Citric Acid. LP:MA L-Proline Malic Acid. a-e: different letters indicate that there are statistically significant differences (p < 0.05)

**Table 3: Optimum conditions**

<table>
<thead>
<tr>
<th>NADES</th>
<th>RATIO</th>
<th>TIME</th>
<th>MAX</th>
<th>DESIRABILITY</th>
</tr>
</thead>
<tbody>
<tr>
<td>ChCl:Fruc</td>
<td>5.000</td>
<td>30.000</td>
<td>6530.839</td>
<td>0.830</td>
</tr>
<tr>
<td>ChCl:Gly</td>
<td>5.234</td>
<td>23.339</td>
<td>1833.512</td>
<td>1.000</td>
</tr>
<tr>
<td>Bet:CA</td>
<td>6.000</td>
<td>28.458</td>
<td>3218.766</td>
<td>1.000</td>
</tr>
<tr>
<td>L-P:MA</td>
<td>16.689</td>
<td>29.041</td>
<td>5389.107</td>
<td>1.000</td>
</tr>
</tbody>
</table>
Conclusions

The extraction of total polyphenols content with NADES was viable.

The best percentage of NADES in water was different for two of the NADES, ChChl:Gly and LP:MA obtained the higher extraction with a high content in water, considering these solvents aqueous solutions, moreover ChChl:Fruc and Bet:CA, both showed the best extraction at 50% (eutectic mixture).
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