Pomegranate peels phenolics encapsulated in edible fruit fiber: storage stability and ingredient for functional foods

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Pomegranate

Pomegranate is an ancient fruit originating from the Middle East and nowadays the global pomegranate production is around 2 million tons.

The most important growing regions are:

- China
- Iran
- Egypt
- Turkey
- Spain
- U.S.A.

24% Peel
14% Seeds
62% Juice
## Composition-Polyphenol Content of Pomegranate Peel

<table>
<thead>
<tr>
<th>Component</th>
<th>Content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total solids</td>
<td>96.00</td>
</tr>
<tr>
<td>Moisture</td>
<td>4.00</td>
</tr>
<tr>
<td>Total sugars</td>
<td>31.38</td>
</tr>
<tr>
<td>Proteins</td>
<td>8.72</td>
</tr>
<tr>
<td>Crude Fiber</td>
<td>21.06</td>
</tr>
<tr>
<td>Fat</td>
<td>9.40</td>
</tr>
<tr>
<td>Ash</td>
<td>5.00</td>
</tr>
<tr>
<td>Total phenolics</td>
<td>8.10</td>
</tr>
</tbody>
</table>

### Phenolic Compound Content (mg/100 g dry matter)

<table>
<thead>
<tr>
<th>Phenolic compound</th>
<th>Content</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ellagic acid</td>
<td>44.19</td>
</tr>
<tr>
<td>Catechin</td>
<td>868.40</td>
</tr>
<tr>
<td>Gallic acid</td>
<td>125.80</td>
</tr>
<tr>
<td>Protocatechol</td>
<td>4.17</td>
</tr>
<tr>
<td>Vanilline</td>
<td>3.91</td>
</tr>
<tr>
<td>Caffeic acid</td>
<td>60.46</td>
</tr>
<tr>
<td>Ferulic acid</td>
<td>5.89</td>
</tr>
<tr>
<td>p-coumaric acid</td>
<td>17.64</td>
</tr>
<tr>
<td>Others</td>
<td>8.20</td>
</tr>
</tbody>
</table>

- Antioxidant activity
- Anti-mutagenic activity
- Anti-hypertension activity
- Anti-inflammatory activity
- Anti-atherosclerotic activity

References:
- Aquilera et al., 2008
- Ullah et al., 2012
- Rowayshed et al., 2013
Exploitation of Pomegranate Peels

- Pomegranate: 100 kg
- Peels: 24 kg
- Seeds: 14 kg

- Fodder
- Food Industry
- Cosmetics
  - Ice cream
  - Tea

Pomegranate Peel
Proposed Process for Pomegranate Peels Application in Food Industry

Pomegranate Peels

- Drying
- Grinding
- Ultrasound-assisted extraction
  - Solvent
  - Fodder
- Filtration
- Evaporation
- Drying
- Emulsification
  - Wall material
  - Phenolics
- Encapsulation by spray drying
  - Recycled solvent

Food additives

Microcapsules of phenolics

Kaderides et al., 2015
**Why Encapsulation of Phenolic Compounds**

- Increase of their stability during storage and passage through the gastrointestinal tract
- Improvement of color
- Masking of astringency
- Suitability for use as an additive in functional foods

*Fang & Bhandari, 2010*
## Encapsulation methods

<table>
<thead>
<tr>
<th>Method of encapsulation</th>
<th>Encapsulation efficiency</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Extrusion</td>
<td>89.39%</td>
<td>Belščak-Cvitanovic et al., 2011</td>
</tr>
<tr>
<td>Rapid Extraction of Supercritical Solution</td>
<td>79.78%</td>
<td>Santos et al., 2013</td>
</tr>
<tr>
<td>Formation of multiple emulsion using a rotating disk reactor</td>
<td>80.00%</td>
<td>Akhtar et al., 2014</td>
</tr>
<tr>
<td>Freeze drying</td>
<td>75.50%; 97.22%</td>
<td>da Rosa et al., 2014; Saikia et al., 2015</td>
</tr>
<tr>
<td>Spray drying</td>
<td>99.80%</td>
<td>Kaderides et al., 2015</td>
</tr>
</tbody>
</table>
Wall material characteristics

1. Good rheological properties at high concentration
2. Disperse or emulsify the active material and stabilize the emulsion produced
3. Chemical non reactivity with the active core materials
4. Seal and hold the active material within its structure during processing/storage
5. Provide maximum protection to the active material against environmental conditions
6. Acceptable solubility of the solvent to the food industry

Desai & Park, 2005
## Wall Materials Used for Encapsulation of Phenolic Compounds

<table>
<thead>
<tr>
<th>Phenolic extract</th>
<th>Wall material</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Spray drying</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Pomegranate peel extract</td>
<td>Maltodextrin; Whey protein concentrate; Skim milk powder</td>
<td>Kaderides et al., 2015</td>
</tr>
<tr>
<td>Carrot extract</td>
<td>Maltodextrin</td>
<td>Ersus &amp; Yurdagel, 2008</td>
</tr>
<tr>
<td>Olive leaf extract</td>
<td>Chitosan</td>
<td>Kosaraju et al., 2006</td>
</tr>
<tr>
<td>Soybean extract</td>
<td>Maltodextrin; Starch</td>
<td>Georgetti et al., 2008</td>
</tr>
<tr>
<td>Bilberry extract</td>
<td>Whey protein concentrate</td>
<td>Betz et al., 2012</td>
</tr>
<tr>
<td>Apple extract; Olive leaf extract</td>
<td>Sodium caseinate; Lecithin</td>
<td>Kosaraju et al., 2008</td>
</tr>
<tr>
<td><strong>Co-crystallization</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Yerba Mate extract</td>
<td>Calcium alginate</td>
<td>Deladino et al., 2008</td>
</tr>
<tr>
<td>Green tea EGCG extract</td>
<td>Gelatin</td>
<td>Shutava et al., 2009</td>
</tr>
<tr>
<td>Blackcurrant extract</td>
<td>Glucan</td>
<td>Xiong et al., 2006</td>
</tr>
<tr>
<td><strong>Freeze drying</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cloudberry extract</td>
<td>Maltodextrin DE 5-8 &amp; DE18,5</td>
<td>Laine et al., 2008</td>
</tr>
<tr>
<td>Hibiscus tea extract</td>
<td>Pullulan</td>
<td>Gradinaru et al., 2003</td>
</tr>
</tbody>
</table>
Wall Materials Suitable for Encapsulation of Phenolic Compounds

Wall material with appropriate encapsulation properties

Carbohydrates
- Undesired taste
- Unnatural additives

Proteins
- Replacement with alternative products

Insoluble dietary fibres
- Complex carbohydrates

Strong absorption properties

Soluble dietary fibres
- Assist regular bowel movements and flushing the intestinal system of undesirable materials

Insoluble dietary fibres
- Slow down the metabolism rates of sugars and form a lining gel within the intestines

Natural dietary fibres
### Production and Exploitation of Orange Fruit

#### Production of oranges in E.U.

<table>
<thead>
<tr>
<th>Country</th>
<th>Tons (2013)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spain</td>
<td>2 933 800</td>
</tr>
<tr>
<td>Italy</td>
<td>1 950 000</td>
</tr>
<tr>
<td>Greece</td>
<td>914 000</td>
</tr>
<tr>
<td>Portugal</td>
<td>206 300</td>
</tr>
</tbody>
</table>

- **Food industry** *(source of dietary fiber)*
  - Animal feed
  - Fertilizer
  - Wastes *(source of dietary fiber)*
- **50% juice**
- **50% by-product** *(peel, seed, pulp)*
- **Highly polluted wastewater**
- **No economic value**
## Incorporation of Pomegranate Peel Extract in Foods

<table>
<thead>
<tr>
<th>Product</th>
<th>Activity</th>
<th>Reference</th>
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<tbody>
<tr>
<td><strong>Encapsulated pomegranate peel extract</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hazelnut paste</td>
<td>Antioxidant</td>
<td>Kaderides et al., 2015</td>
</tr>
<tr>
<td>Ice cream</td>
<td>Antioxidant</td>
<td>Cam et al., 2014</td>
</tr>
<tr>
<td><strong>Pomegranate peel extract</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Shrimps</td>
<td>Antimicrobial</td>
<td>Basiri et al., 2015; Hayrapetyan et al., 2012</td>
</tr>
<tr>
<td>Meat pate</td>
<td>Antimicrobial</td>
<td></td>
</tr>
<tr>
<td>Bread</td>
<td>Antioxidant</td>
<td>Paari et al., 2012; Altunkaya et al., 2013</td>
</tr>
<tr>
<td>Sunflower oil</td>
<td>Antioxidant and</td>
<td>Iqbal et al., 2008; Kanatt et al., 2010; Naveena et al., 2008</td>
</tr>
<tr>
<td>Cooked chicken patties</td>
<td>Antimicrobial</td>
<td></td>
</tr>
</tbody>
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Incorporation of Pomegranate Peel Extract in Foods

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<tr>
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<td></td>
</tr>
<tr>
<td>Sunflower oil</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cooked chicken patties</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

![Graph showing Peroxide value vs Storage time for different extracts.](image-url)
The exploitation of pomegranate and orange wastes based on:

- *Ultrasound-assisted extraction of phenolic compounds from pomegranate peel*
- *Encapsulation of extract by spray drying using orange juice industry by-product as wall material*

**Study of:**

1. *Storage stability of crude and encapsulated pomegranate peel extract*
2. *Incorporation of crude and encapsulated extract in foods:*
   - a. *Peanut butter*
   - b. *Milk powder*
   - c. *Fresh juice*
Integrated Process for Orange Wastes Application as Wall Material for Encapsulation

Orange

<table>
<thead>
<tr>
<th>Step</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Juice production</td>
<td></td>
</tr>
<tr>
<td>Orange wastes</td>
<td></td>
</tr>
<tr>
<td>Boiling</td>
<td>20 min</td>
</tr>
<tr>
<td>Washing</td>
<td></td>
</tr>
<tr>
<td>Drying</td>
<td>60 °C, 48 h</td>
</tr>
<tr>
<td>Grinding</td>
<td></td>
</tr>
<tr>
<td>Sieving</td>
<td></td>
</tr>
<tr>
<td>Orange wastes powder</td>
<td></td>
</tr>
</tbody>
</table>

Orange wastes are boiled in hot water at 100 °C for 20 minutes, washed, dried at 60 °C for 48 hours, ground, and sieved to produce Orange wastes powder.
# Pomegranate Peel Extract – Optimized Conditions of Ultrasound Assisted Extraction

1. **Extraction temperature:** 35 °C
2. **Solvent type:** *Water*
3. **Solvent/Solid ratio:** 32/1
4. **Amplitude level:** 40% (50 W)
5. **Pulse duration/Pulse interval ratio:** 7/6
6. **Extraction time:** 10 min

*Kaderides et al., 2015*

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![Image of ultrasound equipment and extraction process](image_url)

130 W, 20 kHz VCX-130 Sonics and Materials (Danbury, CT, USA) with Ti–Al–V probe (13 mm)
Integrated Process for Encapsulation of Pomegranate Peel Extract in Orange Wastes Powder

1. **Pomegranate Peel**
2. **Washing**
3. **Grinding**
   - Ultrasound-assisted extraction
4. **Filtration**
5. **Solvent**
6. **Evaporation**
7. **Wall material (Orange wastes powder)**
8. **Emulsification**
9. **Encapsulation by spray drying**
10. **Drying**  
    - 40 °C, 48 h
Encapsulation of Pomegranate Peels Extract – Optimized Conditions of Spray Drying

1. **Wall material:** Orange wastes
2. **Inlet air temperature:** 162 °C
3. **Feed solids concentration:** 5 %
4. **Ratio of core to wall material:** 1/9
5. **Drying air flow rate:** 50%
6. **Flow rate of compressed air for atomization:** 0.80 m³/h

- Concurrent
- Two - fluid nozzle atomization
- Peristaltic pump for feed

Buchi, B-191, Buchi Laboratoriums-Technik, Flawil, Switzerland
**Storage Stability**

- Crude pomegranate peel extract
- Encapsulated pomegranate peel extract

**Measurement of:**
- Antioxidant activity
- Total phenolics content
- Color

Shelf-life test at 60°C for 40 days
Incorporation of Phenolic Capsules in Foods

- **PRODUCT:**
  - a. Peanut butter
  - b. Milk powder (full-fat)
  - c. Fresh juice (apple, orange, carrot)

- **ADDITIVE:** Phenolic extract (Crude and encapsulated)

- **PHENOLICS CONCENTRATION:** 5000 ppm

3 samples were tested:
- Sample with encapsulated extract
- Sample with crude extract
- Control sample

Measurement of:
- Antioxidant activity
- Total phenolics content
- Oxidation stability
- Color

Shelf-life test at 60°C for 30 days
Shelf-life test at 4°C for 30 days
Results
# Physicochemical Properties of Orange Wastes Powder

<table>
<thead>
<tr>
<th>Component</th>
<th>g/100 g DM</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Protein</strong></td>
<td>13.25 ± 0.11</td>
</tr>
<tr>
<td><strong>Lipid</strong></td>
<td>2.12 ± 0.05</td>
</tr>
<tr>
<td><strong>Ash</strong></td>
<td>4.25 ± 0.07</td>
</tr>
<tr>
<td><strong>Carbohydrate</strong></td>
<td>80.38</td>
</tr>
<tr>
<td><strong>Total dietary fiber</strong></td>
<td>65.7 ± 0.9</td>
</tr>
<tr>
<td><strong>Insoluble dietary fiber</strong></td>
<td>48.9 ± 0.5</td>
</tr>
<tr>
<td><strong>Soluble dietary fiber</strong></td>
<td>16.8 ± 0.8</td>
</tr>
<tr>
<td><strong>Moisture content</strong></td>
<td></td>
</tr>
<tr>
<td><em>(g water/100 g DM)</em></td>
<td>8.52</td>
</tr>
<tr>
<td><strong>Water - holding capacity</strong></td>
<td></td>
</tr>
<tr>
<td><em>(g water/g DM)</em></td>
<td></td>
</tr>
<tr>
<td>Sieving (Din 60-70)</td>
<td>9.65</td>
</tr>
<tr>
<td>No sieving</td>
<td>12.07</td>
</tr>
</tbody>
</table>
### Extraction and Encapsulation Yield by Ultrasound-Assisted and Spray Drying Method

**Optimum conditions for maximum extraction yield**

<table>
<thead>
<tr>
<th>Solvent</th>
<th>T (°C)</th>
<th>A (%)</th>
<th>t (min)</th>
<th>Pulse (sec/sec)</th>
<th>L/P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>35</td>
<td>40</td>
<td>10</td>
<td>7/6</td>
<td>32/1</td>
</tr>
</tbody>
</table>

Yield: 13.85 mg GAE/g DM

**Optimum conditions for maximum encapsulation yield**

<table>
<thead>
<tr>
<th>Wall material</th>
<th>T (°C)</th>
<th>Qa (%)</th>
<th>Qs (m³/h)</th>
<th>w/c</th>
</tr>
</thead>
<tbody>
<tr>
<td>Orange wastes powder</td>
<td>162</td>
<td>50</td>
<td>0.80</td>
<td>9/1</td>
</tr>
</tbody>
</table>

Yield: 12.96%
Storage Stability of Crude and Encapsulated Extract

**Antioxidant Activity**

- DPPH Method
  - p-value = 0.088

- Folin-Ciocalteu Method
  - p-value = 0.094

**Total Phenolics Content**

Shelf-life test at 60°C for 40 days
Storage Stability of Crude and Encapsulated Extract

**Color changes**

- **L:** lightness
- **a:** (+) red, (-) green
- **b:** (+) yellow, (-) blue

![Graph showing color changes over storage time](image)

- Δ = L, Crude extract
- • = a, Crude extract
- △ = b, Crude extract
- □ = L, Encapsulated extract
- ■ = a, Encapsulated extract
- ▲ = b, Encapsulated extract

Graph indicates the color changes over storage time for both crude and encapsulated extracts, with specific color changes noted for lightness (L) and chromatic components (a, b).
Antioxidant Capacity and Total Phenolics Content of Peanut Butter

**ANTIOXIDANT ACTIVITY**

- **DPPH Method**
  - p-value = 0.837

- **Folin-Ciocalteu Method**
  - p-value = 0.135

**TOTAL PHENOLICS CONTENT**

Shelf-life test at 60°C for 30 days

Crude extract  Encapsulated extract  Blank
Effect of Pomegranate Peel Extract on Oxidative Deterioration of Peanut Butter

Peroxide Value (meq/kg) vs. Storage time (days)

- Blank
- Encapsulated extract
- Crude extract

Shelf-life test at 60°C for 30 days

p-value = 0.010
Antioxidant Capacity and Total Phenolics Content of Milk Powder

**DPPH Method**
- Blank
- Encapsulated extract
- Crude extract

**Folin-Ciocalteu Method**
- Blank
- Encapsulated extract
- Crude extract

- **p-value = 0.000**
- **p-value = 0.034**

Shelf-life test at 60°C for 30 days
Effect of Pomegranate Peel Extract on Oxidative Deterioration of Milk Powder

p-value = 0.390
**Antioxidant Capacity and Total Phenolics Content of Fresh Juice**

- **DPPH Method**
  - Scavenging capacity (%)
  - Storage time (days): 0, 5, 10, 15, 20, 25, 30
  - p-value = 0.112

- **Folin-Ciocalteu Method**
  - Total phenolics (mgGAE/g DM)
  - Storage time (days): 0, 5, 10, 15, 20, 25, 30
  - p-value = 0.000

**Shelf-life test at 4°C for 30 days**

**ANTIOXIDANT ACTIVITY**

**TOTAL PHENOLICS CONTENT**

Blank, Crude extract, Encapsulated extract
Color Changes of Fresh Juice

Shelf-life test at 4°C for 30 days
Conclusions

An integrated approach for utilization of pomegranate peels is suggested based on the spray drying encapsulation of their phenolics compounds using an alternative wall material (orange wastes)

- The orange wastes were successfully used as wall material for encapsulation of pomegranate peels extract
- During storage of crude and encapsulated extract there was no significant change at total phenolics content and scavenging capacity
- The extract (crude and encapsulated) was found efficient in improving the shelf-life of peanut butter, milk powder and fresh juice
- The crude extract changed the texture and the color of peanut butter significantly
Thank you!