The Determination of Physicomechanical Properties of Nettle Seed (Urtica pilulifera) and Optimization of Its Mucilage Extraction Conditions using Response Surface Methodology

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Abstract
In this study, some geometrical, gravimetrical and frictional properties of nettle seed were evaluated for the first time. The length, width and thickness of seeds were 2.49, 1.98 and 0.78 mm, and the average arithmetic diameter, average geometric diameter, sphericity and surface area of nettle seeds were, respectively, 1.75, 1.29 mm, 0.52 and 5.31 mm². The true density, bulk density and porosity of nettle seeds were 1168.12, 457.19 kg/m³ and 60.86%, respectively, and the static friction coefficient on the plywood surface, glass, rubber, fiberglass and galvanized iron were 0.28, 0.21, 0.34, 0.22 and 0.27, respectively. Then, using the response surface method, the optimal conditions for extracting nettle mucilage as a new source of hydrocolloids were determined by achieving maximum seed surface ratio, extraction yield, and viscosity. Face central composite design analysis of the effect of three independent variables, including soaking temperature (25-60 °C), soaking time (0.5-4 h) and water to seed ratio (1:20-1:60), was studied. The ANOVA results showed that the quadratic polynomial model was the best model for interpreting the behavior of the three responses. Statistical analysis of the data showed that extraction yield and viscosity were significantly affected by all independent variables (P<0.05), while the effect of temperature on seeds surface ratio was not significant (P>0.05). Based on the numerical optimization method, optimized conditions for extraction of nettle seed gum were determined in terms of soaking temperature of 59 °C, soaking time of 3.4h (204 min) and water to seed ratio of 1:40. Under the optimum conditions, the seeds surface ratio, extraction yield and viscosity values were obtained as 2.39, 9.70% and 6.25 mPa/s, respectively. The results of this study are of practical value for designing post-harvesting and processing equipments for nettle seeds as well as extracting mucilage from the seeds.

Keywords: Extraction optimization, Mechanical properties, Mucilage, Nettle seed, Viscosity

Introduction
Hydrocolloids are high molecular weight and hydrophilic biopolymers that are functional additives in nutritional formulations (Dickinson, 2003). Due to the importance of hydrocolloids in the food industry, the search for identification of new natural hydrocolloids continues, as they can be potential alternatives for some commercial hydrocolloids
Urtica pilulifera L. is an annual herb with a troublesome, creeping branched rhizome. The plant has been employed in the treatment of liver respiratory, gastritis and enteritis, rheumatism and skin disorders (Yiiksel et al., 2009). Seeds are obtained from its mature fruits; these seeds form a layer of mucilage when are soaked in water (Baytop, 1999). Literature review indicates a lack of scientific information and a thorough understanding of the physical and mechanical properties of the seed. Also, no research has been carried out on optimizing the extraction conditions of nettle seed mucilage. Therefore, this study aimed to investigate the physical and mechanical properties of nettle seeds and to find the most suitable conditions for the gum extraction.

Materials and methods
Measurement of physicochemical properties of nettle Seed
After preparing the Nettle seed, physical properties of the seeds, including size, dimension, mass, true and bulk density as well as porosity, were measured using the standard methods and, calibrated equipment. In the case of mechanical properties, static coefficient of friction on different surfaces (galvanized iron, plywood, glass, fiberglass, and rubber) as well as the filling and emptying angle of repose were determined (Razavi & Fathi, 2009).

Optimization of Gum Seed Extraction Conditions
Three independent variables of temperature, time and, water to seed ratio were applied for extraction of the nettle seed gum, respectively, in the range of 25-60 °C, 0.5-4 h and 20:1 to 60:1 water: seed ratio. Before adding the seeds, the water was heated to the desired temperature. Gum was then separated from the swollen seeds by a rotary plate extractor. It was then filtered and, it was dried at 36 °C. The extracted gum was milled after drying and kept in a cool and dry place.

Measuring seed surface ratio by image processing method
Seed surface image was captured by DINO microscope (Model AM313T, Taiwan). Seed surface were studied before and after soaking at the different conditions of extraction. After imaging, the images were processed using Image J software and the seed surface ratio was calculated.

Extraction yield
The yield of the extracted gum at various extraction conditions was determined using the following equation (Razavi et al., 2009b).
\[ Y = 100 \times \left( \frac{\text{mass of extracted gum (g)}}{\text{mass of nettle seed (g)}} \right) \]  

Measurement of viscosity
The dynamic viscosity was determined using a capillary tube viscometer. For this purpose, 1% solution of each sample of gum powder was prepared. The dynamic viscosity (\( \eta \)) of the sample was calculated by the following equation (Razavi & Akbari, 2012):
\[ \eta = K \rho t \]  

In this equation, \( K \) is the viscometer constant (equal to 0.0404), \( \rho \) is the sample density and \( t \) is the sample passage time of the two viscometer signal lines.

Experimental design and statistical analysis
In this study, the effect of temperature, time and, water: seed ratio on three response variables
including seed area ratio, extraction yield, and viscosity was optimized by the response surface methodology (RSM) and flat central composite design (FCCD). The data were analyzed using Design-Expert software version 10.0.7.0.

Results and discussion

Physicomechanical properties of nettle seed

The physical and mechanical properties of nettle seed measurements are shown in Table (1).

<table>
<thead>
<tr>
<th>Property</th>
<th>Replication</th>
<th>Average</th>
<th>Standard deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Length (mm)</td>
<td>50</td>
<td>2.4888</td>
<td>0.1537</td>
</tr>
<tr>
<td>Width (mm)</td>
<td>50</td>
<td>1.9842</td>
<td>0.1439</td>
</tr>
<tr>
<td>Thickness (mm)</td>
<td>50</td>
<td>0.7828</td>
<td>0.0631</td>
</tr>
<tr>
<td>Average diameter (mm)</td>
<td>50</td>
<td>1.7519</td>
<td>0.0824</td>
</tr>
<tr>
<td>Average geometrical diameter (mm)</td>
<td>50</td>
<td>1.2894</td>
<td>0.1689</td>
</tr>
<tr>
<td>Sphericity</td>
<td>50</td>
<td>0.5170</td>
<td>0.0494</td>
</tr>
<tr>
<td>Area (mm²)</td>
<td>50</td>
<td>5.3114</td>
<td>1.3690</td>
</tr>
<tr>
<td>Mass of 100 (gr)</td>
<td>3</td>
<td>2.1826</td>
<td>0.0377</td>
</tr>
<tr>
<td>True density(kg/m³)</td>
<td>3</td>
<td>1168.12</td>
<td>12.5122</td>
</tr>
<tr>
<td>Bulk density(kg/m³)</td>
<td>3</td>
<td>457.186</td>
<td>4.1677</td>
</tr>
<tr>
<td>Porosity (%)</td>
<td>3</td>
<td>60.8591</td>
<td>0.4696</td>
</tr>
<tr>
<td>CSF (plywood)</td>
<td>3</td>
<td>0.2773</td>
<td>0.0093</td>
</tr>
<tr>
<td>CSF (rubber)</td>
<td>3</td>
<td>0.3444</td>
<td>0.0195</td>
</tr>
<tr>
<td>CSF (glass)</td>
<td>3</td>
<td>0.2137</td>
<td>0.0021</td>
</tr>
<tr>
<td>CSF (fiberglass)</td>
<td>3</td>
<td>0.2247</td>
<td>0.0052</td>
</tr>
<tr>
<td>CSF (galvanized iron)</td>
<td>3</td>
<td>0.2742</td>
<td>0.0108</td>
</tr>
<tr>
<td>unloading repose angle (°)</td>
<td>3</td>
<td>18.4466</td>
<td>2.4511</td>
</tr>
<tr>
<td>loading repose angle (°)</td>
<td>3</td>
<td>19.5156</td>
<td>0.9195</td>
</tr>
</tbody>
</table>

Model fitting

The quadratic polynomial response model was fitted to each of the response variables. The coefficient of determination of the models for all the models were higher than 0.80, indicating acceptable fit of the models to the experimental data. Also the lack of fit was not statistically significant for all responses \((P>0.05)\), which confirms the accuracy of the models in predicting the responses.

Seed surface ratio

Among the independent variables, water: seed ratio had the highest and, soaking temperature had the least effect on seed surface ratio. As the soaking time increases, the seeds are more exposed to water, and the seed surface ratio increases. At higher temperatures, the mucilage adhesion to the seeds decreases, and the mucilage around the seeds is excised, reducing the seed surface ratio. By increasing the amount of water to seed ratio, the conditions for osmosis in the seed increase and the seeds swell more.

Extraction yield

Soaking time had the highest and water: seed had the least effect on the extraction yield. As the soaking time of the seeds increases, water penetrates the seeds, the dissolution of gum and its diffusion into the water intensifies (Ye & Jiang, 2011). At higher temperatures, the bonding of the gum to seed decreases, gum extraction occurs easier (Koocheki, Mortazavi, Shahidi, Razavi, & Taherian, 2009b). By increasing the water: seed ratio further, the dilution effect reverses the water osmosis process, and gum diffusion, thereby reducing the yield (Ghobadi, Varidi, Varidi, & Koocheki, 2018).
Viscosity
Soaking temperature, as well as time-temperature and time-water: seed interaction, respectively, have the most significant impact on the viscosity of the gum. The increase in viscosity due to the increase in soaking temperature can be synchronized with the effect of increasing temperature on the gum extraction yield (Ghobadi et al., 2018; Singthong, Ningsanond, & Cui, 2009). The increase in viscosity at high soaking times can be attributed to the increased extraction yield under these conditions. The water: seed ratio up to 1:40 increased the viscosity but reverse trend was observed for higher the water: seed ratio. This phenomenon was consistent with the effect of water: seed ratio on the extraction yield. Similar results were reported by Koocheki, Taherian, Razavi, & Bostan (2009a), Wu, Cui, Tang, & Gu (2007) and Singthong et al. (2009).

Conclusions
This study identified some physico-mechanical properties of nettle seeds and optimized the gum extraction conditions. Based on the results of the response surface methodology, the optimum conditions for the extraction of nettle seed gum were soaking temperature of 59 °C, water: seed ratio of 1:40, soaking time of 3.4 hours. The gum extracted under these conditions had a maximum surface area ratio of 2.39, a yield of 9.7% and, a viscosity of 6.25 mPa/s Seed mucilage can be used as a new and indigenous source of hydrocolloids, but further research on the physicochemical, rheological, and functional properties of this new hydrocolloid needs to be carried out.

References
Razavi, S. M. A., & Akhari, R. (2012). Biophysical properties of agricultural and food materials: Published by Ferodowski University of Mashhad, Iran (in Persian)

