The ultrafiltration performance of composite membranes for the concentration of plant extracts

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Abstract
The paper reports the performance of polysulfone-polyaniline composite membranes in medicinal plants concentration, by means of the ultrafiltration process. The effect of operating pressure, raw material concentration on permeate flux, and some biological compounds concentration in permeate and concentrate were analyzed.

The aim of this article was to compare the performances of the ultrafiltration process, using the polysulfone and the polysulfone-polyaniline membranes for the concentration of Salvia officinalis extracts. The bioactive compounds (reducing sugars, proteins and polyphenols) have been characterized by UV-VIS spectrometry, using specific methods for each (compounds) ones.

The ultrafiltration process’ performance can be increased using a polysulfone-polyaniline membrane. The experimental results showed higher efficacy of polysulfone-polyaniline composite membranes versus polysulfone membrane for the concentration of plant extracts.

Keywords: ultrafiltration, polysulfone-polyaniline composite membrane, Salvia officinalis

Introduction
The medicinal plant Salvia officinalis L. (Labiaceae family) is known for a long time as a remedy in the traditional medicine [1]. It is used in the food industry (aromatization) and also included as a compound in many phytopreparations. Herbal tea, the alcoholic and aqueous extracts from its aerial parts are used for the body strengthening, and therefore used – before the discovery of antibiotics– for treatment of inflammatory processes in the organism [2, 3].

Phytochemical investigation of drug revealed a great number of bioactive compounds possessing a variety of biological activities [4]. Sage (S. officinalis) contains 1-2.5% volatile oil (containing salvene, pinene, camphor, cineole, borneol, 30% thujone, salvene esters and sesquiterpenes), saponins, diterpene bitter principle, flavonoids, phenolic acids (rosmarinic, fumaric, chlorogenic, caffeic), flavonoid glycosides, proteins [5-7].

A serial of major inconveniences has been identified for the traditional methods for the concentration and purification of medicinal plant extracts such as: high energy consumption, low separation rate, heat sensitive substances being easily decomposed, the characteristics of the compound being easily affected and serious pollution of the production environment.
Membrane separation processes have been extensively studied and developed for their application to medicinal plant extracts purification and separation [8-11]. Microfiltration (MF) for removing impurities and macromolecules and ultrafiltration (UF), nanofiltration (NF) or reverse osmosis (RO) for concentration, can reach the purpose of concentration and purification extracts. The integration of a membrane process and traditional methods has turned into an important development of modern parapharmaceutical processes [11].

The aim of this paper was to compare the performance of ultrafiltration process using the polysulfone and the polysulfone-polyaniline membranes for the concentration of *Salvia officinale* extracts.

**Materials and methods**

**Extract preparation and concentration**
The extracts were prepared by maceration, using the distillate water and ethylc alcohol (50%v/v) as solvents for *Salvia officinale*. The herbal’s mass concentration in the solvent was of 8% (w/v).

The extracts were processed by filtration, microfiltration through Millipore membrane with 0.45 µm pores and ultrafiltration (UF). The concentration ratio (expressed as a volumetric ratio between the permeate and concentrate) was 2:1. The installation on that micro- and ultrafiltration have been used was the KMS Laboratory Cell CF-1 type (Koch Membrane – Germany).

Four flat sheet polysulfone and polysulfone-polyaniline composite membranes were used in the experiment, each having an effective area of 0.0028 m². All the membranes were prepared at the Faculty of Applied Chemistry and Materials Science, Polytechnic University of Bucharest [12].

**Analysis**

By UV-VIS spectrometry using both Lowry method with dinitrosalicylic acid (DNS) and Folin-Ciocâlteu method the total protein, reducing sugars and polyphenols amount from extracts have been determinate [13,14].

The results of ultrafiltration process are usually measured by permeate flux and rejection rate. The flux is expressed in Eq. (1)

$$J = \frac{V}{A \cdot t}$$  \hspace{1cm} (1)

where V is the permeate volume, A the membrane effective area, t the time necessary for the V liters of permeate to be collected.

The percent rejection, % R, is calculated from the Eq. (2)

$$R = \left[1 - \frac{C_p}{C_f}\right] \times 100$$  \hspace{1cm} (2)

where $C_p$ and $C_f$ are the concentration of the given component in the permeate and the feed respectively.

**Results and discussions**

The properties of the ultrafiltration membranes are summarized in the Table 1.
Table 1 Properties of the ultrafiltration membranes

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Distillate water flux (L/m²h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type of membrane</td>
<td>PSF/NMP</td>
</tr>
<tr>
<td>Pressure (bar)</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>51,4</td>
</tr>
<tr>
<td>2</td>
<td>72,5</td>
</tr>
<tr>
<td>3</td>
<td>94,1</td>
</tr>
<tr>
<td>4</td>
<td>118,6</td>
</tr>
<tr>
<td>5</td>
<td>77,4</td>
</tr>
</tbody>
</table>

Polysulfone-polyaniline composite membranes hydrodynamic properties were much better than those of the polysulfone membranes. So, the water flux at 5 bar pressure is: 118,6 L/m²h for polysulfone in NMP membrane and 159,4 L/m²h for polysulfone-polyaniline membrane in NMP, meantime the water flux is 501,6 L/m²h for polysulfone in DMF solvent and the 665,0 L/m²h for polysulfone-polyaniline membrane in DMF.

Based on the water flux and BSA rejection rate, we chose the PSF/DMF and PSF+PANI/DMF membranes for medicinal plant extracts' concentration and purification. Reducing sugars, polyphenols and proteins concentrations were determined in permeate and concentrate after ultrafiltration of extracts. The results are shown in the Table 2.

The calibration graph has been drawn in order to determine the content of the reducing sugars (dinitrosalicylic acid method) [13], polyphenols (Folin-Ciocâlteu method) [14] and total proteins (Lowry method) [13] from sage and herb Robert biopreparations, by spectrometrical methods. (figs.1-3).

![Figure 1. The calibration graph for the reducing sugars quantitative determination at λ=640nm](image1)

![Figure 2. The calibration graph for the polyphenols quantitative determination at λ=760nm](image2)
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![Figure 3](image.png)

Figure 3. The calibration graph for the protein quantitative determination at \( \lambda = 660 \text{nm} \)

| Table 2. The results obtained at Salvia officinalis extracts processed by ultrafiltration, using polysulfone membrane and polysulfone-polyaniline composite membrane (pressure: 3 bar) |

<table>
<thead>
<tr>
<th>Sample</th>
<th>Membrane type</th>
<th>PSF/DMF membrane</th>
<th>PSF+PANI/DMF membrane</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Permeate flux (L/m²·h)</td>
<td>Permeate Concentrate</td>
<td>Permeate Concentrate</td>
</tr>
<tr>
<td>Salvia officinalis aqueous extract</td>
<td>initial</td>
<td>-</td>
<td>33.8</td>
</tr>
<tr>
<td></td>
<td>s.u. (%)</td>
<td>1.49</td>
<td>1.05</td>
</tr>
<tr>
<td></td>
<td>Glucose concentration (mg/mL)</td>
<td>16.26</td>
<td>9.72</td>
</tr>
<tr>
<td></td>
<td>Polyphenols concentration (µg/mL)</td>
<td>201.16</td>
<td>159.04</td>
</tr>
<tr>
<td></td>
<td>Protein concentration (mg/mL)</td>
<td>1.997</td>
<td>1.665</td>
</tr>
<tr>
<td>Salvia officinalis hydro alcoholic extract</td>
<td>Permeate flux (L/m²·h)</td>
<td>-</td>
<td>32.1</td>
</tr>
<tr>
<td></td>
<td>s.u. (%)</td>
<td>1.22</td>
<td>1.08</td>
</tr>
<tr>
<td></td>
<td>Glucose concentration (mg/mL)</td>
<td>9.88</td>
<td>10.81</td>
</tr>
<tr>
<td></td>
<td>Polyphenols concentration (µg/mL)</td>
<td>332.88</td>
<td>303.54</td>
</tr>
<tr>
<td></td>
<td>Protein concentration (mg/mL)</td>
<td>1.989</td>
<td>1.220</td>
</tr>
</tbody>
</table>

From the table 2 notes that permeate flux are significantly lower compared with those for distilled water due to of the complexity of the extracts composition. The permeate flux value were ranging between 32.1 to 51.7 L/m²·h at 20°C and 3 bar pressure. The water flux were 296.4 and 410.0 L/m²·h for the selected membranes, in the same condition.

Table 2 shows that PSF+PANI > PSF among the two types of membranes in permeate flux and bioactive compounds concentration. The rejection of sugars from sage aqueous extract as this is processed by ultrafiltration, through a composite membrane was of 69.7%.
Conclusion

The vegetal processing of the extracts by the ultrafiltration process leads to the biological active concentration of the compounds and its separation – especially as these are processed from the polysaccharide and from the protein classes – from smaller molecular weight compounds (free aminoacids, monosaccharide, phenolic acids etc.), which pass in the ultrafiltration permeate.

The experimental results showed the increased performance of polysulfone-polyaniline composite membranes versus polysulfone membrane for the concentration of plants extracts. The permeation flux was over 51 L/m²·h at 20°C and 3 bar pressure, and the rejection of polysaccharide was maximum 69.7% for sage aqueous extract processed by ultrafiltration using composite membrane.

Between the two types of the membranes used in these experiments, the polysulfone-polyaniline composite membranes are more efficient both, in terms of permeate flow and the analyzed rejection degree of the compounds.

Acknowledgments

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Reference

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