

Possibilities for application of ultrafiltration for utilization of rose wastewater

Marina Mitova^{*1}, Mariya Dushkova¹, Ivan Bakardzhiyski², Tanya Titova-Kosturkova³, Mariyana Sestrimska³, and Nikolay Menkov¹

¹Department of Process Engineering, Technical Faculty, University of Food Technologies, 4002, Plovdiv, Bulgaria

²Department of Technology of Wine and Beer, Technological Faculty, University of Food Technologies, 4002, Plovdiv, Bulgaria

³Department of Electrotechnical, Electronics and Automation, Technical Faculty, University of Food Technologies, 4002, Plovdiv, Bulgaria

Abstract. This study investigates the possibilities for application of ultrafiltration for utilization of rose waste water obtained from water-steam distillation of rose petals from *Rosa damascena* Mill. Ultrafiltration was conducted using an UF1-PAN membrane up to volume reduction ratio of 5. The effects of frontal filtration prior to ultrafiltration and temperature (20°C and 50°C) during membrane process were examined. The obtained retentates and permeates were analyzed according to the following parameters: total phenolic content, phenolic acids, flavonoid phenolic content and spectral characteristics. The flux and energy demand were determined during ultrafiltration. The results showed that there was no statistically significant difference between the flux during ultrafiltration with and without frontal filtration, as well as for energy demand at the same working conditions. The temperature rise led to an increase in the flux and a decrease in the energy demand. The total phenolic content, phenolic acids, and total flavonoid content were found to increase in the retentate, while they decreased in the permeate. The principal component analysis was made for all parameters measured and revealed that the samples can be separated into three groups. In further investigations, the retentate will be added to rice grits in order to obtain enriched extruded products.

1 Introduction

One of the symbol of Bulgaria, together with yoghurt, is rose oil [1]. Traditional water-steam distillation is the most used method in Bulgaria to produce rose oil. The rose wastewater obtained by hydrodistillation is a rich source of valuable water-soluble compounds including polyphenols, glycosides and tannins [2]. Conventional techniques for concentration of bioactive compounds from natural sources, such as steam and vacuum distillation, are energy-intensive processes that employ high temperatures [3]. These techniques generally demand elevated temperatures and are associated with high energy consumption [4]. Membrane processes offer an alternative to traditional concentration methods by operating at room temperatures without phase changes, thereby preserving the heat-sensitive components [5]. Ultrafiltration (UF) has been extensively utilized in the food processing industry over the past two decades due to its notable advantages in comparison with traditional separation techniques: mild treatment conditions for sensitive products, high selectivity, reduced energy demand, and obtaining of a product with high quality. Therefore, UF has become a main technique for separation and concentration in food technology [6].

The low yield of essential oil results in substantial waste generation, including solid rose petals and liquid wastewater [7]. Early in the 21st century, the concept of recycling industrial waste gained prominence with a particular focus on valorizing plant-based materials and their bioactive components. Numerous studies have explored innovative approaches, such as polysaccharide extraction. However, due to limited composition and biological activity of rose waste byproducts, most of these methods remain at the laboratory stage [8, 9, 10]. Wastewaters generated from rose oil distillation pose a significant environmental pollution, due to their disposal into drainage systems and rivers [7]. Developing strategies for wastewater management coupled with the valorization of wastewater for pharmaceutical applications presents a promising approach to address this issue [11]. This study investigates the potential of UF to recover and concentrate valuable polyphenolic compounds from rose wastewater.

2 Materials and methods

2.1 Materials

2.1.1 Obtaining of wastewater

* Corresponding author: mitovadm@gmail.com

Rose wastewater was obtained from an industrial plant at the BulPhytoOils JSC company, village of Zelenikovo, municipality of Brezovo, Bulgaria. Approximately 500 kg of rose petals from *Rosa damascena* Mill were processed per batch using a ratio of 1:5 (water: rose petals) and a flow rate of rose distillate of 500 dm³/h (8,33 dm³/min) for 120 minutes. The generated wastewater was collected for subsequent treatments.

The following parameters of rose wastewater, retentates and permeates were determined: total phenolic content (TPC), phenolic acids (PA), flavonoid phenolic content (FPC), soluble solids content (%Brix), dry matter (%), and transmittance spectrum.

2.1.2 Chemicals

The following reagents and were used for the analyses: concentrated hydrochloric acid (Merck, Darmstadt, Germany), methyl alcohol 99,9% (Marvin, Dimitrovgrad, Bulgaria), ethyl alcohol 96% (Fillab Sole LLC, Plovdiv, Bulgaria).

2.2 Methods

2.2.1 Coarse filtration

Before frontal filtration (FF) the rose wastewater was subjected to coarse filtration with pore diameter of 2 mm.

2.2.2 Frontal filtration

FF was performed using a press pad filter (Rover pompe, Polverara, Italy). The pore diameter of the filter pads was 3 μm.

2.2.3 Membrane filtration

A laboratory equipment with a replaceable plate and a frame module was used for membrane filtration. The membrane used (UF1-PAN) was polyacrylonitrile with molecular weight cut-off 1 kDa, the membrane area was 1250 cm² [12, 13]. The volume and pH of the feed rose wastewater (V_F) were 6 L and 4.1, respectively. Membrane filtration (UF) was carried out at: operating pressure of 0.2, 0.3, 0.4 and 0.5 MPa; temperature of 20°C and 50°C, feed flow rate of 330 dm³/h; volume reduction ratio of 2.5 and 5. The membrane's cleaning was performed with NaOH 0.5% at a temperature of 50°C, pressure of 0.2 MPa, with circulation time of 30 min, followed by end rinsing with distilled water.

2.2.4 Determination of main characteristics of ultrafiltration process

The permeate flux (J , dm³/(m²h)) was calculated using the following equation:

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

where V is the volume of the permeate, dm³;
 A is the membrane area, m²;
 t is the time of filtration, h.

The volume reduction ratio (VRR) was determined as:

$$VRR = \frac{V_F}{V_R} \quad (2)$$

where V_F is the feed volume, dm³;
 V_R is the retentate's volume after UF, dm³.

Energy demand was calculated as follows:

$$E = \frac{W_{pump}}{J \cdot A} \quad (3)$$

where W_{pump} is the power required by the pump, kWh;
 J is the permeate flux, dm³/(m²h)
 A is the membrane area, m²

The power required by the pump was calculated as follows:

$$W_{pump} = \frac{p \cdot Q_{feed}}{\eta} \quad (4)$$

where: p is the pressure at the entry of the membrane module, Pa

Q_{feed} is volume flow rate, m³/s

$\eta=0.7$ is the pump's efficiency according to Verberk and Van Dijk [14]

2.2.5 Determination of chemical composition

TPC, FPC and PA content were determined in initial rose wastewater, retentates and permeate by modified Glories Method [15].

Soluble solids content was measured using a refractometer Hanna instruments HI 96801 (Bedfordshire, England).

Dry matter was determined according to AOAC, 1999 [16].

2.2.6 Spectral characteristics

Spectral characteristics for the dependence of transmittance on wavelength were measured with M509T CamSpec Spectrophotometer (West Yorkshire, UK).

Transmittance and absorbance are quantitative characteristics for the intensity of spectral curves in optical spectroscopy:

$$T = \left(\frac{I}{I_0} \right) \cdot 100 \quad (5)$$

$$A = \lg \left(\frac{I_0}{I} \right) \quad (6)$$

where: T – transmittance, %

A – absorbance

I_0 and I are the output and unabsorbed electromagnetic radiation, respectively [17].

2.2.7 Principal component analysis

The main purpose of the principal component analysis (PCA) is to transform a high-dimensional experimental database by calculating new features (factors) between which there is no correlational connectivity. The reduction of the original data matrix $X_{(m \times n)}$ was performed by calculating the matrix of factor weights $B_{(m \times l)}$ and the matrix of factor coefficients $F_{(l \times n)}$, the procedure being accompanied by minimal loss of information expressed by error matrix $E_{(m \times n)}$ [17]:

$$X_{(m \times n)} = B_{(m \times l)} \times F_{(l \times n)} + E_{(m \times n)} \quad (7)$$

where: m is the number of defining features

n is the number of analyzed samples.

The new factors were uncorrelated with each other and, through the values of their eigenvectors, reflect the total variation of the original experimental database. The PCA method arranged principal component values in descending order, i.e. the first eigenvector reflects to the highest degree of the variance of the experimental array. The second principal component contained the variance of the data that is not reflected by the first eigenvector, etc.

The selection of the reduced number of features was carried out according to the general rule that the newly calculated principal components reflect 90-95% of the variance of the original database and also that only factors that increased the predictive qualities of the model were included in the analyzes.

2.2.8 Statistical analysis

Data were presented as mean values calculated from three determinations. To compare experimental groups, Fisher's Least Significant Difference (LSD) test was employed at 0.05 significance level using one-way analysis of variance (ANOVA) and Excel 2016 software.

3 Results and discussion

Figure 1A shows the permeate flux at the beginning of the process (VRR 0) and at two different concentration levels (VRR 2.5 and 5) at different transmembrane pressures (TMP 0.2, 0.3, 0.4 and 0.5 MPa) with or without FF. It can be seen that the permeate flux increased with the TMP rise. This can be attributed to improved hydrodynamic conditions resulting in increased recirculation velocity and subsequent lower concentration polarization [18]. The highest values of permeate flux were obtained at the beginning of the

process, the lowest – at VRR 5. A negative correlation was observed between permeate flux and VRR, as illustrated in the figure. This can be explained with the rising solute concentration leading to a higher dynamic viscosity and thus the flux decreases [19]. Comparing the permeate flux of rose wastewater treated with or without FF, it can be seen that there was no statistically significant difference between these two permeate fluxes for all VRR and TMP, except three points. This proves that the FF before UF of rose wastewater is not necessary to perform.

Figure 1B presents the permeate flux at the beginning of the process (VRR 0) and at two different concentration levels (VRR 2.5 and 5) at different working temperatures (20°C and 50°C), and without FF. The TMP had a positive effect on the permeate flux for all VRR and temperatures. The highest values of permeate flux were obtained at the beginning of the process (VRR 0) and 50°C, the lowest - at VRR 5 and 20°C. Comparing the permeate fluxes of rose wastewater ultrafiltered at 20°C and 50°C, it can be seen that these two permeate fluxes were statistically different for all VRR and TMP, as the effect of the temperature on the flux was positive. At elevated TMP, the permeate flux reached a plateau, indicating a lack of correlation with further pressure increases. This phenomenon is commonly observed in UF membranes and is attributed to fouling mechanisms, such as cake formation and concentration polarization, which become more pronounced at higher pressures. Permeate flux is influenced not only by TMP but also by membrane material, structure, and solute-membrane interactions [20]. A hypothesis suggests that an increase in temperature leads to a higher permeate flux due to reduced solvent viscosity, increased solvent diffusion or enhanced polymer chain flexibility [21]. The working temperature of 50°C is more suitable from technological point of view, not only for the flux increase, but because

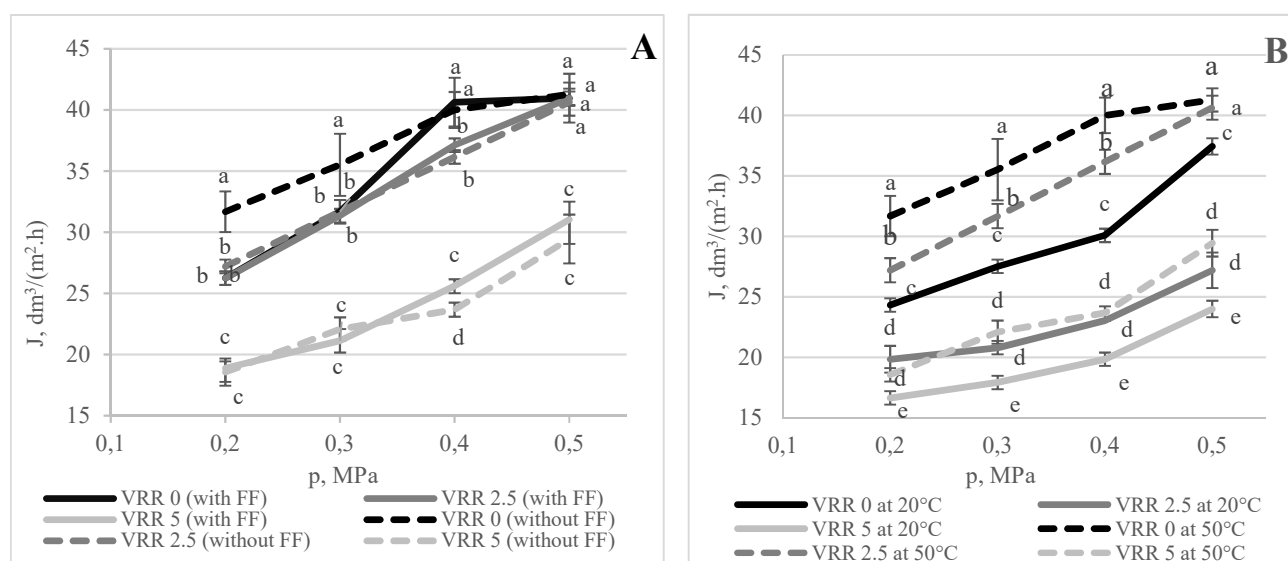


Fig. 1. Flux during UF of rose waste water at different TMPs (A - with or without FF at 50°C and different VRR; B – without FF at 20°C and 50°C and different VRR)

Different lowercase letters (a-e) show significant differences between the flux with and without FF at different VRR (A), and at 20°C and 50°C at different VRR (B), ($p < 0.05$)

is not necessary to cool to low temperature (for example 20°C) the rose wastewater after distillation performed at high temperature.

Figure 2A presents the energy demand at working conditions as in Figure 1A. The energy demand increased with the TMP and VRR rise. There was no statistically significant difference between the energy demands during UF of rose wastewater treated with or without FF, except for three points. These results are comparable with the results for flux and prove that the FF before UF of rose wastewater is not necessary to perform. Figure 2B presents the energy demand at working conditions as in Figure 1B. The energy demand increased with the TMP and VRR rise, and the temperature decrease. Higher energy requirement at

higher concentrations is due to the increased processing time. The reduction in energy demand at higher temperatures can be attributed to decreased solution viscosity and increased mass transfer rates [6]. On the basis of the results obtained, we recommend to work at higher temperature (50°C in our case) which can reduce energy demand during UF. We also recommend to use medium TMP between 0.3 and 0.4 MPa because this pressures will give the best balance between the flux and energy demand.

Table 1 shows an increase in the TPC in the retentate at VRR 5 compared to the initial solution, accompanied by a corresponding decrease in the permeate in our case with 1kDa membrane.

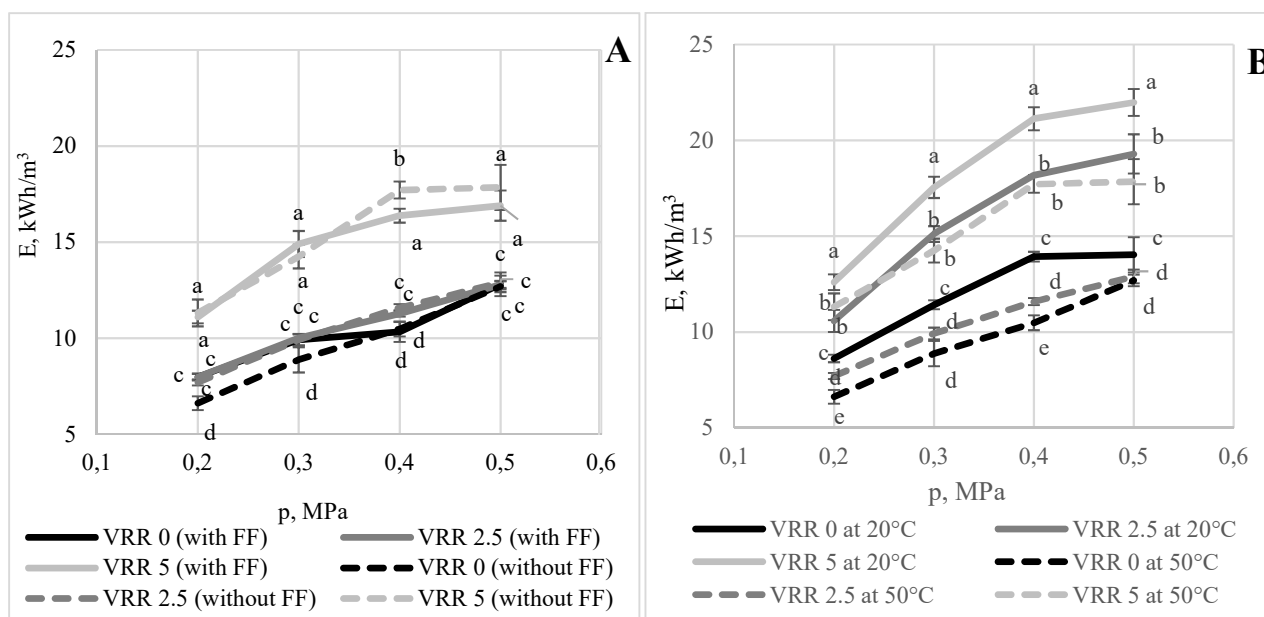


Fig. 2. Energy demand during UF of rose waste water at different TMP (A - with or without FF at 50°C and different VRR; B – without FF at 20°C and 50°C and different VRR). Different lowercase letters (a-e) show significant differences between the energy demand without FF at 20°C and 50°C, and at different VRR (50°C), ($p < 0.05$)

Table 1. Chemical composition of retentates and permeates (TPC - total phenolic content, PA - phenolic acids, FPC - flavonoid phenolic content)

No	Type	TPC, mg/dm ³ gallic acid equivalent	PA, mg/dm ³ caffeic acid equivalent	FPC, mg/dm ³ quercetin equivalent	Soluble solids content, %Brix	Dry matter, %
1	initial solution without FF	2363.04 ± 53.87a	565.02 ± 15.92a,b,c	766.22 ± 31.05a	1.3 ± 0.1a,b	8.24 ± 0.54a
2	initial solution with FF	2398.31 ± 115.79a	577.67 ± b31.83a	734.02 ± 34.82a,b	1.1 ± 0.1a,c	8.50 ± 0.68a
3	retentate without FF 50°C at VRR 5	3272.20 ± 98.60b	815.91 ± 16.73d	1139.67 ± 85.84c	1.8 ± 0d	12.92 ± 0.73b
4	retentate without FF 20°C at VRR 5	3127.20 ± 236.01b,c	756.88 ± 53.04e	1078.50 ± 89.74c	2.1 ± 0.1e	10.51 ± 1.11c
5	retentate with FF 50°C at VRR 5	2997.88 ± 35.27c	718.93 ± 15.92e	911.09 ± 22.30d	1.4 ± 0.2b	10.41 ± 1.09c
6	permeate without FF 50°C	2171.02 ± 29.59d	531.29 ± 6.32bf	653.54 ± 14.75b,e	0.9 ± 0.1f	8.05 ± 0.46a
7	permeate without FF 20°C	2272.90 ± 44.51a,d	554.48 ± 14.61b	705.05 ± 16.73a,b	1.0 ± 0.1c,f	7.71 ± 0.24a
8	permeate with FF 50°C	1978.99 ± 41.29e	505.99 ± 10.96f	614.91 ± 14.75e	1.0 ± 0c,f	8.78 ± 0.18a

a,b,c,d,e,f Different letters in a column mean statistically different values ($p < 0.05$)

A similar trend was observed for PA and FPC. The concentration of TPC increased with the temperature with a 38.5% at 50°C without FF and a 32.3% at 20°C without FF compared to TPC in initial solution. The concentration of PA increased with the temperature with 44.4% at 50°C without FF compared to a 33.96% at 20°C without FF compared to PA in initial solution, the FPC also increased with the temperature – with 48.74% at 50°C without FF and 40.76% at 20°C without FF compared to FPC in initial solution. The temperature had the same effect on the dry matter content. UF led to an increase of soluble solids content in retentates at both temperatures studied. Based on these results, it is more suitable to carry out UF at higher temperature (50°C). Furthermore, the data from Table 1 indicate that FF doesn't influence on the studied substances and is unnecessary pretreatment prior to UF. Phenolic compounds demonstrate a considerably higher permeation compared to polysaccharides, with distinct quantitative and qualitative variations observed across the evaluated materials of various membranes. Their retention is likely influenced by both molecular size and specific structural features of each solute which may explain the wider range of retention percentages noted during the process [20].

Figure 3 (A and B) presents the spectral transmittance curves of the analyzed samples measured in the range from 600 to 900 nm. The figure demonstrates a significant difference in color between the retentates and permeates when compared separately to the initial solution, as evidenced by the distinct peaks observed in the wavelength range of 660 nm to 760 nm.

All peaks correspond to the yellow-orange spectrum, indicating notable modifications of the optical properties of the samples [22, 23, 24]. The observed variations in wavelength intensity indicate changes in the molecular composition, likely influenced by the processes of concentration and permeation, resulting in modifications of the color properties of the solutions. Permeability values in the range of 15-25% were observed for the initial solution (samples S1 and S2). For the permeates without FF (S6 and S7), the spectral transmittance significantly increased, reaching values of 55-60%. As can be seen from Figure 3A, the application of FF does not significantly improve the permeability of the sample (S8). For the retentates (Figure 3B), the transmittance values were the lowest - about 1.5-3%, which could be explained by the higher density of the samples.

Figure 4 shows the output factor space of the type $X_{(8 \times 506)}$, for which the first 5 columns contain the values of the above-mentioned five indicators (total phenolic content, phenolic acids, flavonoid phenolic content, soluble solids content, dry matter), and the next 501 columns contain points of the optical transmittance taken at characteristic lengths of the electromagnetic spectrum. The high-dimensional experimental data set was subjected to analysis by the PCA, with the aim of reduction and visual interpretation of the characteristics of the studied samples [25]. Figure 4A presents a projection of the studied rose wastewater samples in the space formed by the first three eigenvectors, the result of the PCA analysis. The Figure shows that three main groups can be identified – initial solutions, retentates and permeates.

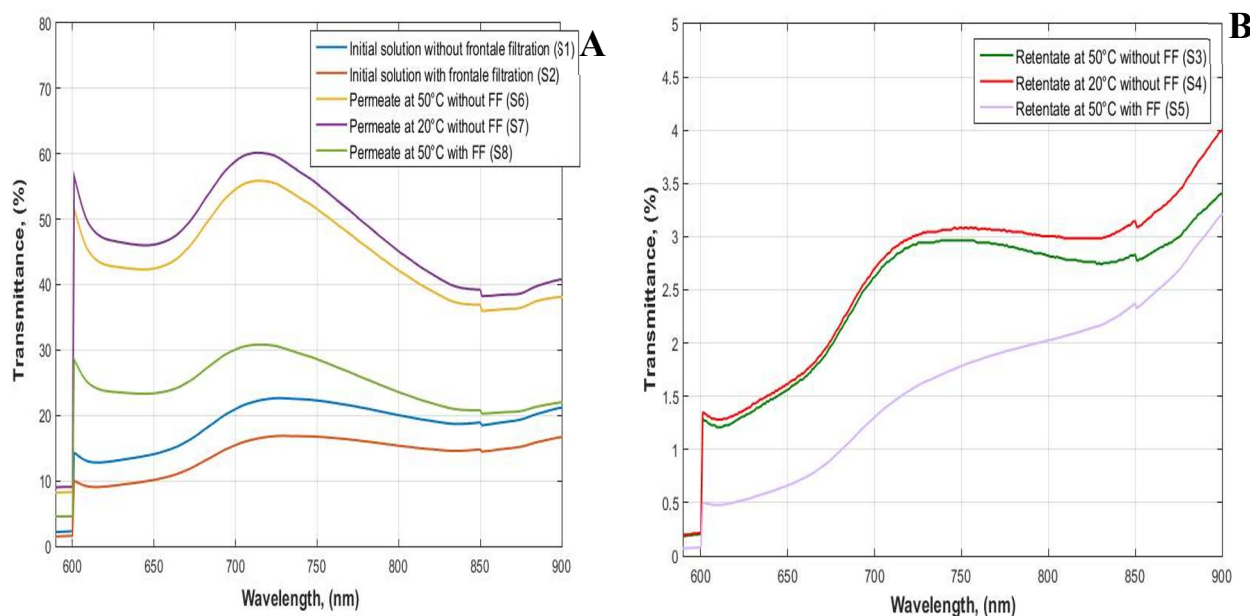


Fig. 3. Optical transmittance spectrum of initial rose waste water and permeate (A), and retentate (B)

In Figure 4B the relationship between the main components and the informative value of each of them is presented. The first eigenvector accounted for 90.01% of the variance of the source database, the second principal component for 8.24%, etc. In sum, the first three principal components accounted for 99.72% of the variation in the experimental data. The three new factors would be a good basis for training an identifying algorithm or system.

The different temperature at which UF was performed, as well as the application of FF, also led to a difference in the measured values for the respective characteristics. If more samples are available, a classifier could be synthesized to identify the samples from the individual groups and also under what conditions the study was made. At the moment, the research in this direction has a rather exploratory character.

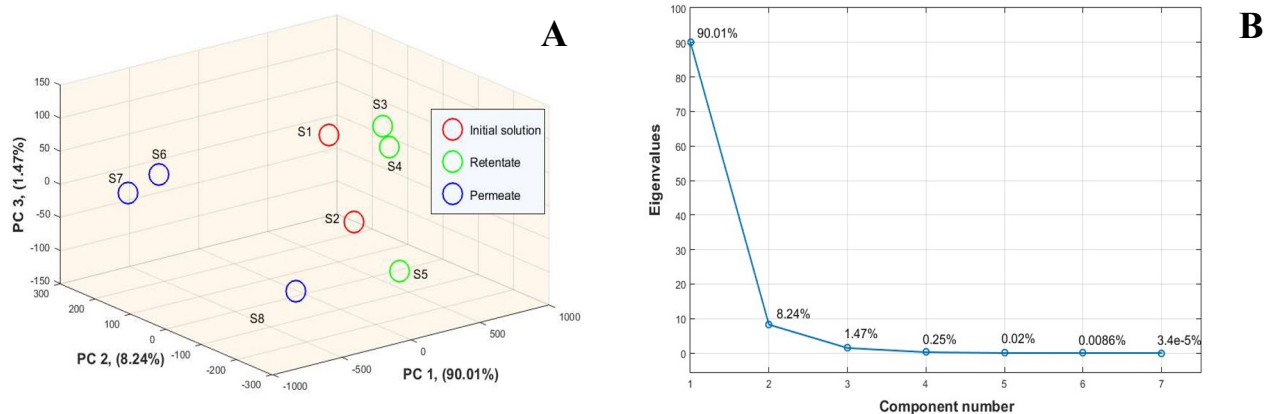


Fig. 4. Principal component analysis (A) and relationship between components (B) for rose wastewater samples (S1 – initial solution without FF; S2 – initial solution with FF; S3 – retentate without FF at 50°C; S4 – retentate without FF at 20°C; S5 – retentate with FF at 50°C; S6 – permeate without FF; S7 – permeate without FF; S8 – permeate with FF)

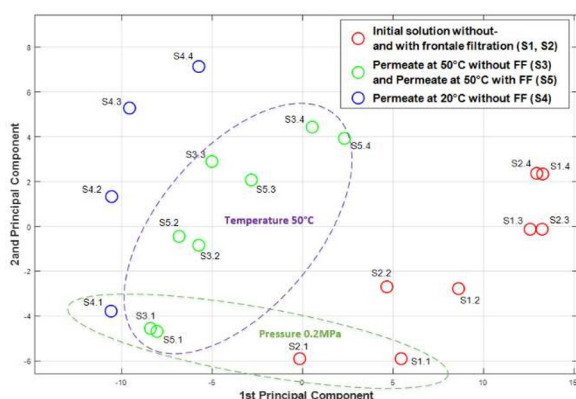


Fig. 5. Result of PCA analysis of flux and energy demand characteristics measured for different pressure values (S1 – initial solution without FF; S2 – initial solution with FF; S3 – retentate without FF at 50°C; S4 – retentate without FF at 20°C; S5 – retentate with FF at 50°C; S6 – permeate without FF; S7 – permeate without FF; S8 – permeate with FF. The numbers 1,2,3,4 after the point of the sample names (S1, S2 etc.) corresponds to TMP 0.2, 0.3, 0.4, 0.5 MPa, respectively)

Figure 5 presents the result of a PCA analysis, with the projection obtained from the permeate flux and energy demand determined for different values of pressure and temperature. The separation of three groups was observed, the first of which corresponded to the initial solution with and without FF. The second group presented permeates with and without FF at 50°C for four different TMP. The third group involved permeates at 20°C without FF at four different TMP.

The figure indicates that the temperature effect was pronounced. There was a grouping tendency of the analyzed samples according to the same pressures.

4 Conclusions

Our research demonstrated that preliminary FF is not necessary to perform prior to UF of rose wastewater. The TMP and temperature rise, and the decrease of VRR led to an increase in the permeate flux. The energy demand increased with the TMP and VRR rise, and with the temperature decrease.

The higher temperature (50°C) during UF process led to an augmentation of the total dry matter, TPC, FPC and PA in the retentates.

The principal component analysis was made for all parameters measured (total phenolic content, phenolic acids, flavonoid phenolic content, soluble solids content, dry matter) and revealed that the samples can be separated into three groups (initial solutions, retentates and permeates). Concerning the permeate flux and energy demand, three groups can be identified based on different solutions (initial solutions, permeate at 20°C and permeates at 50°C) and four groups based on different transmembrane pressure (0.2, 0.3, 0.4, 0.5 MPa).

On the basis of the results obtained, we recommend to perform UF of rose wastewater at higher temperature (50°C) which increase the permeate flux, the TPC, FPC, PA, dry matter and reduce the energy demand. We also

recommend to use medium TMP between 0.3 and 0.4 MPa because these pressures will give the best balance between the flux and energy demand.

The results demonstrate that the rose wastewater is rich in bioactive components that can be concentrated by UF and in further investigations incorporated in rice grits in order to obtain enriched extruded products.

References

1. K. Rusanov, N. Kovacheva, M. Rusanova, I. Atanassov, Low variability of flower volatiles of *Rosa damascena* Mill. plants from rose plantations along the Rose Valley, Bulgaria. *Ind. Crop. Prod.*, **37**, 6–10 (2012). <https://doi.org/10.1016/j.indcrop.2011.11.010>
2. T. Gerasimova, M. Topashka-Ancheva, A. Dobreva, A. Georgieva, M. Mileva, Evaluation of the genotoxic activity of wastewater obtained after steam distillation of essential oil of Bulgarian *Rosa alba* L. – in vivo study. *Rom. Biotechnol. Lett.* **27**(1):3292-3301 (2022). <https://doi.org/10.25083/rbl/27.1/3292-3301>
3. B. Tylkowski, I. Tsibranska, R. Kochanov, G. Peev, M. Giamberini, Concentration of biologically active compounds extracted from *Sideritis* ssp. L. by nanofiltration. *Food Bioprod. Process.* **89**, 309–314 (2011). <https://doi.org/10.1016/j.fbp.2010.11.003>
4. M. Mondal, P.P. Biswas, S. De, Clarification and storage study of bottle gourd (*Lagenaria siceraria*) juice by hollow fiber ultrafiltration. *Food Bioprod. Process.* **100**, 1–15 (2016). <https://doi.org/10.1016/j.fbp.2016.06.010>
5. M. Montenegro-Landívar, P. Tapia-Quirós, X. Vecino, M. Reig, M. Granados, A. Farran, J. Cortina, J. Saurina, C. Valderrama, Recovery of natural polyphenols from spinach and orange by-products by pressure-driven membrane processes *Membranes* **12**(7), 669 (2022). <https://doi.org/10.3390/membranes12070669>
6. A. Mohammad, C. Ng, Y. Lim, G. Ng, Ultrafiltration in food processing industry: review on application, membrane fouling, and fouling control, *Food and Bioprocess Technol.* **5**(4):1143-1156 (2012). <https://doi.org/10.1007/s11947-012-0806-9>
7. A. Georgieva, Y. Ilieva, Z. Kokanova-Nedialkova, M. Zaharieva, P. Nedialkov, A. Dobreva, A. Kroumov, H. Najdenski, M. Mileva, Redox-modulating capacity and antineoplastic activity of waste water obtained from the distillation of the essential oils of four bulgarian oil-wearing roses, *Antioxidants* **10**(10):1615 (2021). <https://doi.org/10.3390/antiox10101615>
8. A. Slavov, I. Vasileva, L. Stefanov, A. Stoyanova, Valorization of wastes from the rose oil industry, *Rev. Environ. Sci. Bio/Technol.* **16**, 309–325 (2017). <https://doi.org/10.1007/s11157-017-9430-5>
9. V. Shikov, D. Kammerer, K. Mihalev, P. Mollov, R. Carle, Antioxidant capacity and colour stability of texture-improved canned strawberries as affected by the addition of rose (*Rosa damascena* Mill.) petal extracts, *Food Res. Int.*, **46**, 552–556 (2012). <https://doi.org/10.1016/j.foodres.2011.04.004>
10. G. Akgül, T. Madenoglu, N. Cengiz, M. Saglam, M. Yüksel, Hydrothermal gasification of *Rosa Damascena* residues: gaseous and aqueous yields. *J. Supercrit. Fluids.*, **85**, 135–142 (2014). <https://doi.org/10.1016/j.supflu.2013.11.007>
11. J. Wedler, A. Weston, J. Rausenberger, V. Butterweck, In vitro modulation of inflammatory target gene expression by a polyphenol-enriched fraction of rose oil distillation waste water. *Fitoterapia*, **114**, 56–62 (2016). <https://doi.org/10.1016/j.fitote.2016.08.019>
12. M. Dushkova, K. Mihalev, A. Dinchev, K. Vasilev, D. Georgiev, M. Terziyska, Concentration of polyphenolic antioxidants in apple juice and extract using ultrafiltration, *Membranes* **12**(11), 1032 (2022). <https://doi.org/10.3390/membranes12111032>
13. M. Dushkova, A. Vardakas, V. Shikov, K. Mihalev, M. Terziyska, Application of Ultrafiltration for Recovery of Polyphenols from rose petal byproduct, *Membranes* **13**(10), 818 (2023). <https://doi.org/10.3390/membranes13100818>
14. J.Q.J.C. Verberk, J.C. Van Dijk, Air sparging in capillary nanofiltration, *Journal of Membrane Science*, **284**,1-2 (2006). <https://doi.org/10.1016/j.memsci.2006.07.050>
15. P. Nedyalkov, I. Bakardzhiyski, V. Shikov, M. Kaneva, V. Shopska, Possibilities for utilization of cherry products (juice and pomace) in beer production. *Beverages* **9**, 95 (2023) <https://doi.org/10.3390/beverages9040095>
16. AOAC (Association of Official Analytical Chemists) Official Methods of Analysis, 16th Edition, 5th Revision, Association of Official Analytical Chemists, Washington DC (1999)
17. R. Borisova, Basics of chemical analysis (Vodolei, Sofia, 2009)
18. S. Qaid, M. Zait, K. El Kacemi, A. El Midaoui, H. El Hajji, M. Taky, Ultrafiltration for clarification of Valencia orange juice: comparison of two flat sheet membranes on quality of juice production. *J. Mater. Environ. Sci.* **8**, 1186–1194 (2017).
19. A. Cassano, C. Conidi, E. Drioli, Clarification and concentration of pomegranate juice (*Punica granatum* L.) using membrane processes. *J. Food Eng.* **107**, 366–373 (2011). <https://doi.org/10.1016/j.jfoodeng.2011.07.002>
20. S. Yammine, R. Rabagliato, X. Vitrac, M. Peuchot, R. Ghidossi, Selecting ultrafiltration membranes for fractionation of high added value compounds from grape pomace extracts OENO

- One **53**, 3 (2019). <https://doi.org/10.20870/oeno-one.2019.53.3.2343>
21. D. Machado, D. Hasson, R Semiat, Effect of solvent properties on permeate flow through nanofiltration membranes. Part I: investigation of parameters affecting solvent flux, *J. of Membr. Sci.* **163**, 1 (1999). [https://doi.org/10.1016/S0376-7388\(99\)00158-1](https://doi.org/10.1016/S0376-7388(99)00158-1)
 22. A. Ghosh, P. Selvaraj, S. Sundaram, T. Mallick, The colour rendering index and correlated colour temperature of dye-sensitized solar cell for adaptive glazing application, *Solar Energy*, **163**, (2018). <https://doi.org/10.1016/j.solener.2018.02.021>
 23. P. Gilberta, W. Haerberlib, Experiments on subtractive color mixing with a spectrophotometer, *Am. J. Phys.*, **75**, 313-319 (2007). <https://doi.org/10.1119/1.2431654>
 24. C. Elvidge, D. Keith, B. Tuttle, K. Baugh, Spectral Identification of Lighting Type and Character, *Sensors*, **10** (4), 3961-3988 (2010). <https://doi.org/10.3390/s100403961>
 25. P. Nowicka, A. Wojdyło, P. Laskowski, Principal component analysis (PCA) of physicochemical compounds' content in different cultivars of peach fruits, including qualification and quantification of sugars and organic acids by HPLC, *Eur Food Res Technol*, 245, 929–938 (2019). <https://doi.org/10.1007/s00217-019-03233-z>