

THE INFLUENCE OF TRANSMEMBRANE PRESSURE
ON FLUX AND ETHANOL REJECTION DURING
NANOFILTRATION OF MAVRUD RED WINES
FROM HARMANLI AND ASENOVGRAD

Alex Georgiev, Viktor Prodanov, Maria Lyudmilova,
Maria Dencheva-Zarkova, Julia Genova[✉]

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Abstract

Nanofiltration technology is an effective technique for reducing alcohol content in wine. This study examines nanofiltration experiments conducted with Bulgarian Mavrud red dry wines sourced from the Harmanli and Asenovgrad regions, utilizing a MaxiMem – PS Prozesstechnik GmbH membrane filtration system equipped with an Alfa Laval NF99HF polyester membrane (MWCO 300 Da). The experiments were carried out in concentration mode of the filtration system, utilizing a Lauda Alpha RA8 thermostat cooling bath for temperature management. The operational parameters consisted of transmembrane pressures of 10–50 bars, along with a cross-flow rate of 1.2 l/min. The objective of the study is to explore how pressure affects alcohol concentration and flux behaviour in both wines and to compare the results. The experimental results confirm that Alfa Laval NF99HF membrane delivers consistently high permeate flux and effective ethanol retention throughout the nanofiltration process.

Key words: nanofiltration, flux, transmembrane pressures, ethanol rejection, red wine

Introduction. Red wines with higher ethanol content have become increasingly common due to warmer growing seasons, which are a result of climate change. Higher temperatures accelerate grape sugar accumulation, resulting in increased

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ethanol production and altering the wine balance [1]. This can be a considerable concern for winemakers because, according to Bulgarian law and the European Commission regulations, the alcohol in wine should be between 14 and 24 vol.%, neither more nor less [2, 3]. Interest in low alcoholic wine has increased recently due to consumers pursuing healthier lifestyles and the necessity to provide healthy options to consumers with specific medical conditions [4]. Together, these considerations have driven growing demand of dealcoholized wine with minimal changes to its sensory and chemical properties.

Red wine is recognized for its considerable antioxidant activity, and moderate consumption has been associated with a reduced risk of cardiovascular diseases. These protective effects are largely attributed to its high concentration of polyphenolic substances. It is important to note that, according to the reports, reducing the ethanol content in red wine does not diminish its cardioprotective properties [5].

Removing ethanol from wine can be done by altering the fermentation process or by removing it after the beverage is produced. The latter can be done using thermal-based processes like evaporation and distillation, but this leads to a considerable loss in volatile aroma compounds, a factor that should be carefully considered in the process [6]. The impact of such technical choices is not only technological but also sensory: consumer-oriented studies emphasize that dealcoholization processes must balance efficiency with flavour and aroma retention, since any decline in sensory quality remains one of the main barriers to consumer acceptance of alcohol-free wines [7]. Membrane processes have proven to be a good alternative for obtaining low alcohol wine. They can be carried out at lower temperatures, thus retaining more of the original wine's composition [8]. Among these membrane-based approaches, nanofiltration (NF) and reverse osmosis (RO) have emerged as the most relevant techniques for wine dealcoholization. NF membranes operate at lower transmembrane pressures than RO and exhibit selective rejection behaviour: achieving complete retention of larger solutes such as polyphenols and sugars, while allowing partial permeation of smaller molecules like ethanol and water. RO membranes, by contrast, employ higher operating pressures and feature a denser active layer that enables more effective ethanol removal, though at the expense of reduced permeate flux; both processes can extract an ethanol-water fraction from the feed, which is subsequently blended with the retentate to achieve the desired target alcohol content [9]. Recent studies demonstrate that NF can effectively reduce ethanol content of wine while predominantly retaining bioactive compounds – such as polyphenols, anthocyanins, sugars, and acids – that are essential for its organoleptic properties [10, 11]. Although RO membranes achieve more complete ethanol separation compared to NF, they operate at significantly lower permeate fluxes and exhibit higher rejection of volatile aroma compounds, which is undesirable in the context of wine dealcoholization. This makes NF membranes more suitable for partial dealcoholization [8].

Critical parameters in these processes include transmembrane pressure, cross-flow rate, permeate flux, and the rejection coefficient. Together, these variables define not only the separation efficiency but also the quality of the dealcoholized product, so it is essential to understand them. Previous works demonstrate that increasing pressure and/or cross-flow rate enhances permeate flux and thus efficiency [12], while transmembrane pressure can also affect rejection behaviour and process kinetics. However, careful selection of the membrane and operating conditions is essential to reduce exposure to fouling and compound loss. The Alfa Laval NF99HF nanomembrane can effectively separate polyphenols and anthocyanins from wine. Full rejection (100%) of anthocyanins is observed, with the rejection coefficients of polyphenols and ORAC capacity being over 97% and 98%, respectively [13].

This article examines a previously unstudied wine from Asenovgrad region and compares the results with those obtained for a similar type of wine from another producer and region (Harmanli) in order to discover general trends for Mavrud wine, independent of the producer and region, and to highlight differences in optimal parameters in regard to flux permeate behaviour and ethanol rejection.

Materials and methods. We examine the extraction of ethanol from two types of dry red wines, Mavrud, produced in the Harmanli and Asenovgrad regions, during nanofiltration experiments in concentration mode. The membrane filtration system MaxiMem – PS Prozesstechnik GmbH was used, which was described in a previous publication [14]. The filtration unit has been used with rectangular flat sheet nanofiltration membrane Alfa Laval NF99HF (thin film composite polyester, MWCO 300 Da) with an active area of 215 cm². In the present investigation temperature regime with cooling is applied using the thermostatic cooling bath LAUDA Alpha RA 8. Details of the experimental procedure used are given in a previous publication [14]. The operational parameters of the experiment included constant-transmembrane pressures of 10 to 50 bars and a cross-flow rate of 1.2 l/min, which was determined as optimal for nanofiltration with NF99HF during previous experiments [12].

Time was measured for every 10 ml of collected permeate, which was then used to calculate the mean value and the evolution over time of the permeate flux using the formula:

$$J = V/(S \times t),$$

where V is the volume, S is the effective surface area of the membrane exposed to the feed (0.0215 m²), and t is time.

Samples taken for ethanol analysis include: a control sample, samples after the 180 and 360 ml collected permeate, and cumulative average permeate and retentate samples. Before the experiments, prewashing of the membrane was performed until the initial membrane flux was restored. The rejection coefficient

was calculated using the formula:

$$R = (1 - C_p/C_f) \times 100, \%$$

where C_p is the average permeate final concentration of 360 ml, and C_f is the ethanol concentration in the wine from the bottle.

The analysis of the samples was carried out using a gas chromatograph Agilent 8890 equipped with a flame ionization detector, a capillary injector, and an automatic injector controller. The chromatograph is equipped with a chromatographic column Agilent CP-Wax 52 CB, 0.25 mm $\times$ 50 (df = 20 μ m), for the analysis of wines and wine beverages. The data analysis for the calibration procedure was carried out using Agilent CG Software. Calibration was performed using standard ethanol solutions in the concentration range of 2–80 vol%, yielding a correlation coefficient of $R^2 = 0.998$. N-propanol was utilized as the internal standard.

The morphology of the profile of the used membrane Alfa Laval NF99HF before and after nanofiltration was inspected under scanning electron microscope (SEM Jeol T200) with parameters (accelerating voltage 5.0–25 kV; maximal magnification 100 000 times; maximal lateral resolution 10 nm; Secondary Electronic Emission SEI; Back Scattered Electrons BEI-topo; imaging of qualitative differences in the elemental composition e BEI-compo).

Results and discussion. In concentration mode nanofiltration, permeate flux increases consistently with rising transmembrane pressure up to 40 bars. Beyond this point, the membrane fouling and mechanical compression of the active layer are likely responsible for the observed flux decline at 50 bars, indicating that the upper pressure threshold for efficient operation of this membrane has been reached. Looking at the graphs for the average flux as a function of pressure, we can see that the data demonstrates a prevailing tendency: for both wine samples, the flux increases with the increasing of the pressure (Fig. 1A, B), which is typical for pressure-driven membrane processes. This trend is most pronounced at lower pressures, where the increase in flux is nearly proportional to the applied pressure.

Higher operating pressures (40–50 bars) enhance the permeate flux but may also lead to increased fouling, attributed to membrane pore blocking and cake formation [12]. An increase in pressure leads to a decreasing flow rate due to the accumulation of retained dissolved substances on the membrane surface. The Harmanli sample consistently exhibits slightly higher average flux values compared to the Asenovgrad sample at the same pressures. This suggests that the Harmanli wine has lower fouling potential, due to lower density and more favourable physicochemical composition (alcohols, organic acids, sugars, polyphenols, aromatic and mineral substances) regarding membrane permeability. At 10 bars, the average flux for Asenovgrad wine is approximately 7.02 L/(m²h), while during Harmanli wine tests it reaches about 7.53 L/(m²h). The variation, though modest, is con-

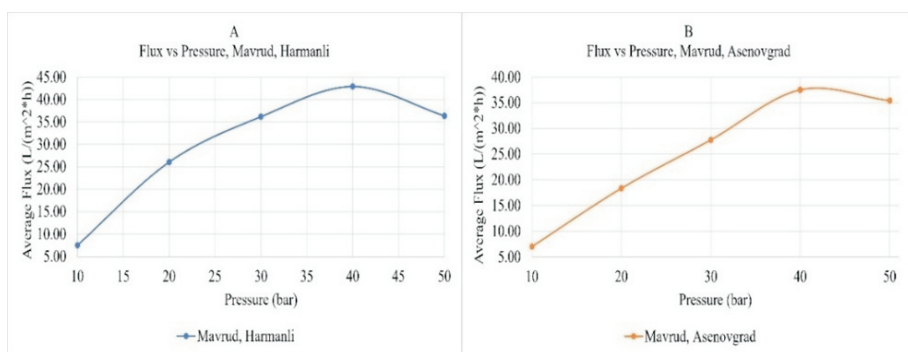


Fig. 1. Average flux as a function of transmembrane pressure, Mavrud wine from Harmanli (A) and Asenovgrad (B) regions

sistent across the pressure range, supporting the observation of regional influence on nanofiltration performance.

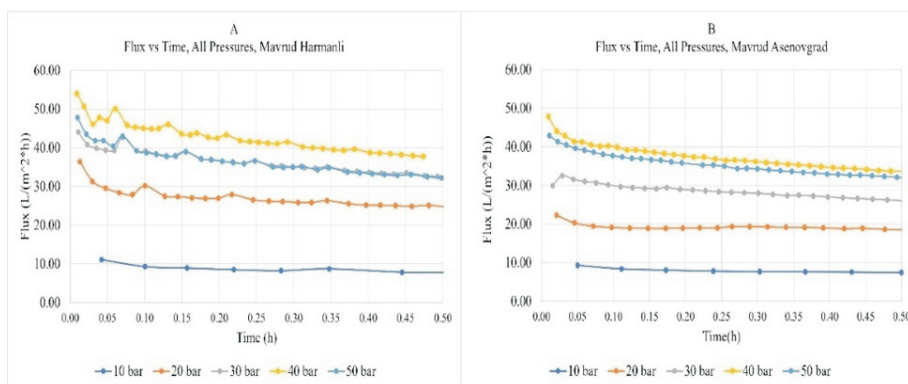


Fig. 2. Flux decline over time at varying pressures during membrane filtration of Mavrud wine from Harmanli (A) and Asenovgrad (B) regions

Looking at the graphs for the behaviour observed around the flux vs. time (Fig. 2A, B) the overall pattern reveals a characteristic decline in flux as nanofiltration progresses, particularly at higher pressures. As implied earlier in the flux vs. pressure debrief, this decline is attributed to membrane fouling concentration polarization, where wine bioactive components (polyphenols, sugars, antioxidants, etc.) accumulate on or within the membrane, increasing resistance and consequently reducing permeability over time. The initial period of filtration sets out a rapid drop in flux, followed by a slower, more gradual decline. We mark the semblance in deviations for the Harmanli wine noticed at even intervals over time. This pattern is typical in wine filtration and is primarily due to the membrane compression which becomes more pronounced as nanofiltration continues. Both Harmanli and Asenovgrad wines grant the best flux over time at 40 bars of pressure.

T a b l e 1

Relation of rejection to pressure

Pressure	Rejection (Harmanli)	Rejection (Asenovgrad)
10 bar	7%	9%
20 bar	18%	20%
30 bar	31%	15%
40 bar	16%	17%
50 bar	22%	14%

The ethanol rejection (Table 1) shows that at 10 bars, there is minimal retention of alcohol (7% for the Harmanli wine and 9% for Asenovgrad). Consequently, the ethanol concentration in the retentate remains largely unchanged, indicating that a transmembrane pressure of 10 bars is insufficient to achieve effective ethanol separation across the membrane. At 20–30 bars, rejection improves significantly, especially for Harmanli wine (up to $\sim 31\%$). At 20 bars, the rejection reaches 20% for Asenovgrad wine, which is its maximum value. Beyond 30 bars, the rejection for Harmanli wine decreases sharply to 16% at 40 bars, before recovering to 22% at 50 bars, where the elevated transmembrane pressure provides a stronger driving force for ethanol transport through the membrane. Ethanol rejection for Asenovgrad wine rises at 40 bars to 17%, before declining steadily to 14% at 50 bars. For Asenovgrad wine, the higher transmembrane pressure at 50 bars is insufficient to offset the decline in selectivity caused by mechanical compression of the membrane, which alters solute transport behaviour.

NB: The “driving force” refers specifically to the transmembrane pressure itself, which pushes the solvent and small solutes (including ethanol) through the membrane.

Based on the above one can conclude that for Alfa Laval NF99HF membrane the optimal rejection range is 20 bar transmembrane pressure for Asenovgrad wine and 30 bar for Harmanli wine. Pressures higher than that lead to worse rejection due to membrane fouling, concentration polarization, and membrane stress. Wine from Harmanli shows consistently higher ethanol rejection than that from Asenovgrad, due to lower density and the stronger interaction of the constituent ethanol with the membrane.

The morphological changes of the membrane active layer as a result of filtration are illustrated in SEM microphotographs (Fig. 3A, B) with two different magnifications. Comparing the obtained images before and after filtration, we observed slight distortion of the active layer surface of the membrane, as well as expansion and significant deformation of the pores. The cut of the unused membrane displays a sharp edge while the outer contour of the used membrane is softer most likely due to the additional layer formed by fouling. In addition, par-

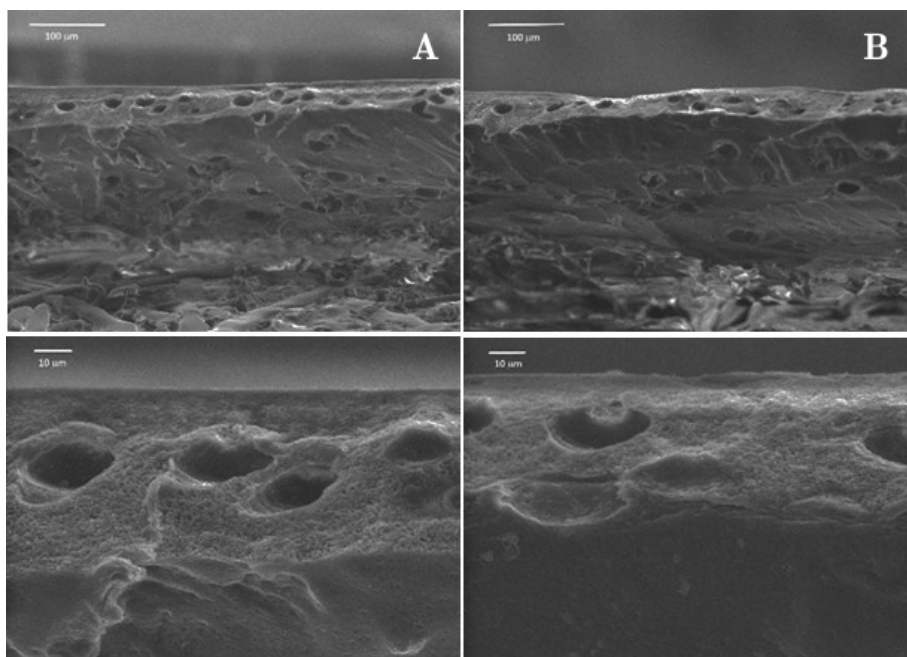


Fig. 3. SEM images of the membrane before (A) and after (B) nanofiltration, acquired via scanning electron microscope JEOL SEM T-200 in SEI mode (at low and high magnification)

tial pore occlusion due to accumulation of macromolecular sediments is observed after nanofiltration process.

Conclusions. The experimental results demonstrate that both pressure and wine origin play significant roles in membrane filtration performance. While increasing pressure enhances flux, higher operating pressures (40–50 bars) may also lead to increased fouling, attributed to membrane pore blocking and cake formation [12]. An increase in pressure leads to a decreasing flow rate due to the accumulation of retained dissolved substances on the membrane surface (concentration polarization). The Harmanli Mavrud wine shows a slight advantage in flux and lower fouling potential, due to lower density and more favourable physicochemical composition regarding membrane permeability.

The nanofiltration membrane Alfa Laval NF99HF partially rejects ethanol from wine, but its effectiveness is strongly transmembrane pressure dependent. Optimum ethanol rejection occurs at 20 bars for Asenovgrad wine and 30 bars for Harmanli wine. Excessively low pressure gives poor rejection due to membrane compression. Wine from Harmanli shows consistently higher ethanol rejection than that from Asenovgrad, due to lower density and the stronger interaction of the constituent ethanol with the membrane.

The conducted experiments confirm the Alfa Laval NF99HF membrane as suitable for partial dealcoholization during nanofiltration of wine in concentration mode. It combines high permeate flux, stable over time, and high ethanol retention. It was shown that as a result of nanofiltration, changes in the morphology of the membrane were observed – expansion and deformation of the pores.

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*Georgi Nadjakov Institute of Solid State Physics, Bulgarian Academy of Sciences,
72 Tsarigradsko Shosse Blvd, 1784 Sofia, Bulgaria*
e-mails: alex.georgiev@issp.bas.bg, viktor.prodanov@issp.bas.bg,
maria.todorova@issp.bas.bg, biolayer@issp.bas.bg, julia.genova@issp.bg