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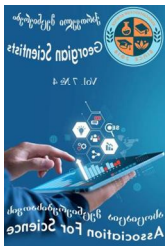

Preparation and study of membranes for the microfiltration process of sterilization of black grape wine material

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Summary

This paper discusses the use of new microfiltration polymer membranes obtained by the phase inversion wet method for the filtration of Saperavi, Kindzmarauli, Khvanchkara, and Mukuzani type wine materials. They were prepared by precipitation of different concentrations of polyethersulfone and polyamide compositions: PES10%/DMF, PES12%/DMF, PA10%/DMF/LiCl, PA12%/DMF/LiCl. It was determined that the obtained microfiltration membranes can be successfully used for the filtration and stabilization of Georgian black wines such as Saperavi, Kindzmarauli, Khvanchkara, and Mukuzani without changing the sensory properties of the wine. It was also found that among the four membranes obtained, red wines filtered on the N2 membrane, which is obtained from a PES12%/DMF polymer composition with a pore size of 0.45 μm and better morphology, have lower turbidity values.

Keywords: Red wine, membrane, microfiltration, turbidity, pore size.

The use of membrane technology in winemaking has revolutionized various stages of winemaking. It represents a precise and efficient alternative to traditional methods. Membranes, characterized by selective permeability, play a crucial role in improving wine quality. During the purification process, microfiltration polymer membranes effectively remove suspended particles and colloids from the wine, allowing for clearer wine without the use of chemical reagents. In the wine stabilization process, ultra- and nanofiltration membranes made of polyethersulfone and polyamides are also capable of selectively removing proteins, polysaccharides, and microorganisms from the wine material, which

leads to an increase in wine stability and shelf life. Some membranes can also be used in the wine dealcoholization process to selectively remove ethanol while preserving the aroma and taste of the wine. This responds to the growing demand for low-alcohol and non-alcoholic wines. In addition, membrane technology contributes to resource recovery and sustainable development in accordance with the principles of the circular economy. Filtration as a method of wine purification has been known since ancient times. After alcoholic and lactic acid fermentation of wine, natural wine is a complex system with a cloudy color, in which three main groups of substances are identified, depending on the size of the contained particles [1,2,]. 1. Dissolved substances (less than 1 nm in size), which include ions, salts, organic acids, and phenolic substances. 2. Colloids (1 nm to 1 mm in size), which include polysaccharides, proteins, polymerized polymeric phenolic compounds, and colloidal substances, aggregates. 3. Particles (larger than 1 mm in size) include microorganisms (yeasts, bacteria), cellular debris, colloidal aggregates, and potassium tartrate crystals.

The viscosity of "black wine" varies considerably, but is typically within the range of other red wines. It is influenced by factors such as alcohol content, sugar (dry extract), glycerol, and polysaccharides. Dry red wines exhibit higher dynamic viscosities than white wines due to their high alcohol and sugar content. Georgian red wines in particular are characterized by their strength, high alcohol and tannin content, which gives them a more intense, sharp taste and higher viscosity compared to rosé wines [3,4]. The dynamic viscosities of Saperavi, Kindzmarauli, Khvanchkara and Mukuzani at 20°C range from 1.83 MPa to 1.615 MPa.

Wine clarity is the first visual characteristic that must remain unchanged under all conditions during the wine's storage period. The main key function of filtration is to ensure wine clarity and microbiological stabilization. Wine clarity is assessed by determining turbidity, specifically nephelometric turbidity units (NTU). After filtration, turbidity should be less than 2 NTU.

In this work, new membranes made of two well-known polymer materials, polyethersulfone and polyamide, were obtained and used for wine microfiltration. Polysulfone membranes are the most commonly used material for red wine filtration, as they are characterized by low protein binding capacity, high permeability, and chemical compatibility, which allows the preservation of wine quality [5,6]. The aim of the study was to test the obtained microfiltration membranes for the sterilization of Saperavi, Kindzmarauli, Khvanchkara, and Mukuzani type wine materials. Also, the goal of the paper is to select and determine membranes with appropriate pore sizes based on the best indicators of wine filtrate turbidity.

To obtain membranes from the above-mentioned polymeric materials, a wet phase inversion method was used. 10% and 12% polyethersulfone and polyamide solutions in dimethylformamide were prepared under constant stirring conditions for 6 h at 50°C [7,8].

As a result, the following membrane preparation compositions were obtained: PE10%/DMF, PE12%/DMF, PA10%/DMF/LiCl, PA12%/DMF/LiCl. From the above systems, 4 new membrane films were respectively deposited on a laboratory filler at a temperature of 20℃. To analyze the chemical structure, the IR spectra of the above membranes were studied. Figure 1 shows the infrared images of the N2 (red) and N4 (blue) membranes.

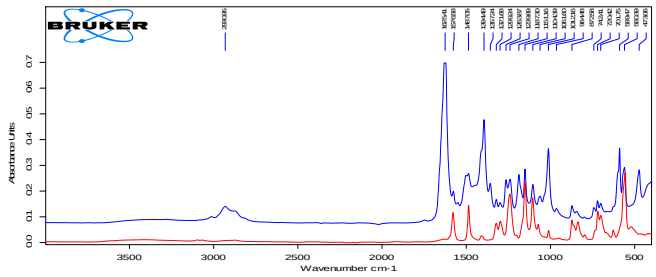

Figure 5. Spectra of N2 (red) and N4 (blue) membranes.

In the N2 image of the polyethersulfone membrane, 621 cm-1 corresponds to C, 882 cm-1 to the unpaired bond C=C of the aromatic nucleus. The peaks at 1150 cm-1, 1235 cm-1 and 1483 cm-1 are attributed to the sulfonic group O=S=O, and 1296 cm-1 to the aromatic ether group C-O-C. The 706 cm-1 peak indicates the presence of a C-S bond. In contrast, the N4 (blue) peak in the polyamide membrane is at 1625 cm-1, which corresponds to a C=N bond [7,8].

The surface morphology of the obtained membranes was studied using a scanning probe microscope. Micrographs of the membranes are shown in Figures 2, 3, 4, and 5.

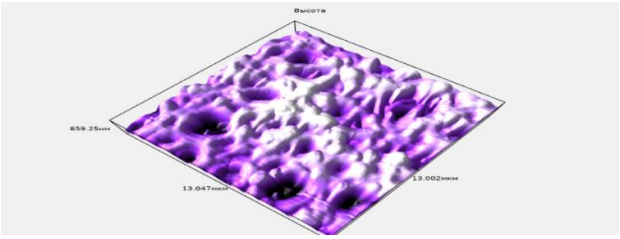

Figure 2. Membrane N1

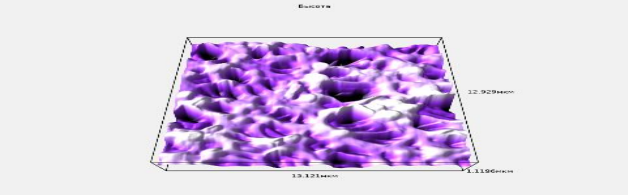

Figure 3. Membrane N2

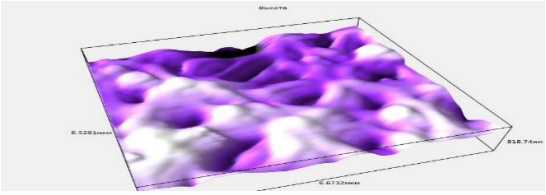

Figure 4. Membrane N3

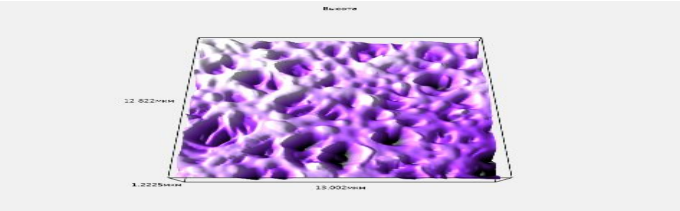

Figure 5. Membrane N4

The structures of membranes (N1, N3) precipitated from 10% polymer solutions were found to be flaky, with uneven pore distribution and surface roughness in both polyethersulfone and polyamide. Membranes N2 and N4, in which the polymer was taken at a content of 12%, have ordered smooth surfaces with almost the same maximum pore size of 0.45 μm-0.55 μm. This is consistent with the result that the wine filtrate tested on N2 and N4 membranes was selected with low turbidity values among the membranes used for wine filtration.

The pore sizes and performances of the obtained membranes were determined on a laboratory device. Table 1 shows the compositions obtained from different polymers and the characteristics of the deposited membranes.

Table 1 Compositions of different systems, characteristics of precipitated membranes.

N	Composition of solutions	Membranes	Pore size, D μm	Productivity l/m2h
1	PES10%/DMF	N1	0,65	220
2	PES12%/DMF	N2	0,45	301
3	Pa 10%/DMF/LiCl	N3	1,0	278
4	Pa 12%/DMF/LiCl	N4	0,55	325

The obtained polymer membranes were used for filtration and testing of Saperavi Kindzmarauli, Khvanchkara, and Mukuzani type wine materials. Table 2 shows the turbidity indicators before and after filtration. As can be seen from the table, the mentioned membranes can reduce turbidity indicators to standard levels. The lowest turbidity indicators were recorded as a result of filtration of wines with the N2 membrane. The pore size of which is 0.45 μm. Table 2 shows the turbidity indicators of Georgian black wines.

Table 2. Turbidity indicators of Georgian black wines before and after filtration.

Wine	Membrane	Turbidity, NTU	
		Before filtering	After filtering
Saperavi	N1	26,45	2,0
	N2		1,71
	N3		1,86
	N4		1,82
Kindzmarauli	N1	20,18	1,63
	N2		1,58
	N3		1,66
	N4		1,61
Khvanchkara	N1	22,34	1,67
	N2		1,62
	N3		1,76
	N4		1,65
Mukuzani	N1	22, 47	1,75
	N2		1,66
	N3		1,73
	N4		1,69

Result and conclusion. Studies have shown that microfiltration membranes obtained by precipitation of polyethersulfone and polyamide compositions of different concentrations can be successfully used for the filtration and stabilization of Georgian black wines such as

Saperavi, Kindzmarauli, Khvanchkara, and Mukuzani without changing the sensory properties of the wine. Membrane samples provide crystal clarity, 100% purity and stability of wine. It also significantly increases storage stability (biological turbidity, repeated fermentation) without clouding and sediment formation. It was also found that wines filtered through the N2 membrane have lower turbidity values which is obtained from a 12% polymer composition of polyethersulfone, the pore size of which is 0.45 μm and has a smooth surface.

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შავი ყურძნის ღვინომასალის სტერილიზაციის მიკროფილტრაციული პროცესისათვის მემბრანების მიღება და კვლევა

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რეზიუმე

აღნიშნულ ნაშრომში განხილულია საფერავის, ქინძმარაულის, ხვანჭკარას, მუკუზანის ტიპის ღვინომასალის ფილტრაციისათვის ფაზური ინვერსიის სველი მეთოდით მიღებული ახალი მიკროფილტრაციული პოლიმერული მემბრანების გამოყენება. ისინი დამზადებული იყო განსხვავებული კონცენტრაციის პოლიეთერსულფონისა და პოლიამიდის კომპოზიციების პეს10%/დმფ, პეს12%/დმფ, პა10%/დმფ/LiCl, პა12%/დმფ/LiCl გამოლექვით. დადგინდა, რომ მიღებული მიკროფილტრაციული მემბრანები შესაძლებელია წარმატებით იქნას გამოყენებული ქართული შავი ღვინოების საფერავის, ქინძმარაულის, ხვანჭკარას, მუკუზანის ტიპის ღვინომასალის ფილტრაციისა და სტაბილიზაციისათვის ღვინის სენსორული თვისებების შეუცვლელად. ასევე დადგინდა რომ მიღებული ოთხი მემბრანიდან ღვინოების სიმღვრივის უფრო დაბალი მნიშვნელობები გააჩნიათ N2 მემბრანაზე გაფილტრულ წითელ ღვინოებს, რომელიც მიღებულია პეს12%/დმფ პოლიმერული კომპოზიციიდან, რომლის ფორის ზომაა 0,45მკმ და გააჩნია უკეთესი მორფოლოგია.

საკვანძო სიტყვები: წითელი ღვინო, მემბრანა, მიკროფილტრაცია, სიმღვრივე, ფორის ზომა.