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Effect of phase inversion temperature on the formation and performance of PES/DMF/PVP polyethersulfone membranes.

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Abstract

The paper studies the preparation of polymer membranes from 15% polyethersulfone solutions of PES/DMP/5%PVP and PES/DMP/7%PVP compositions using the phase inversion method at different precipitation temperatures. Two groups of polyethersulfone membranes were obtained and precipitation was carried out for each at four temperatures (10℃, 20℃, 30℃, 40℃). The particle sizes and degree of polydispersity in the casting solutions were studied on a Malvern analyzer. The optimal temperature for conducting phase inversion at two different PVP concentrations and the influence of the coagulation bath temperature on the performance, structure, and pore size of the resulting membranes were determined. Membranes precipitated from a solution containing 5% PVP showed better performance and structure than membranes precipitated at 20℃, and membranes precipitated from a solution containing 7% PVP showed better performance and structure than membranes precipitated at 30℃.

Keywords: Polyethersulfone, phase inversion, DMF, PVP, productivity

Introduction

In the phase inversion process of membrane preparation, the structure of the solid phase formed during the exchange of solvent and non-solvent in the membrane composition depends significantly not only on the nature of the solvent and non-solvent, but also on the temperature of the coagulation bath. Phase inversion is the process during which a polymer solution is transformed into a solid membrane. Temperature has a significant impact on the phase inversion process of polyethersulfone membranes, affecting both the morphology of the final membrane and the kinetics of the process. High temperatures typically increase the rate of phase separation and consequently lead to rapid dissolution of the polymer solution. This can lead to the formation of a dense membrane with a thin compact selective layer and potentially small pore sizes. At low temperatures, on the contrary, the phase inversion process slows down, giving the polymer chains more time to rearrange and resulting in membranes with porous structures with larger pores [1,2,3].

Analysis In experimental studies, polyethersulfone 5200 in dimethylformamide was used, with polyvinylpyrrolidone as an additive, which, in addition to increasing the hydrophilicity of PES membranes, also plays the role of a pore former in the phase inversion process. The concentration of the PES/DMP/PVP main polymer in the polymer composition was taken as 15%, while two concentrations of 5% and 7% by weight were selected for polyvinylpyrrolidone. Precipitation procedures were carried out separately for each composition at four temperatures. The goal was to determine the optimal temperature for conducting phase inversion at two different PVP concentrations and the effect of coagulation bath temperature on the performance, structure, and pore size of the resulting membranes.

A 15% PES composition in dimethylformamide was prepared in a 100 ml flask at 50℃ for 24 h under constant stirring with a magnetic stirrer. The homogeneity of the solution was checked by polarizing-interference optical microscopy. Polyvinylpyrrolidone was used as an organic additive for the polymer solution, and two more compositions were prepared with the addition of 5% and 7% PVP. Precipitation of both compositions occurred at 10℃ , 20℃, 30℃, 40℃.
The parameters of polymer opening in the membrane compositions, particle size, concentration and degree of dispersion (PDI) of PES/DMF were determined using an instrument (Zetasizer Nano Zen 3690- Malvern Instruments, England). Figures 1, 2 and 3 show the particle intensity curves of PES compositions without additives, containing 5% and 7% PVP. In the PES/DMF composition without additive, the particle sizes are up to 100 nanometers, while in the 5% and 7% PVP-containing compositions, the particle sizes increase from 300 nm to 600 nm with increasing additive concentration.

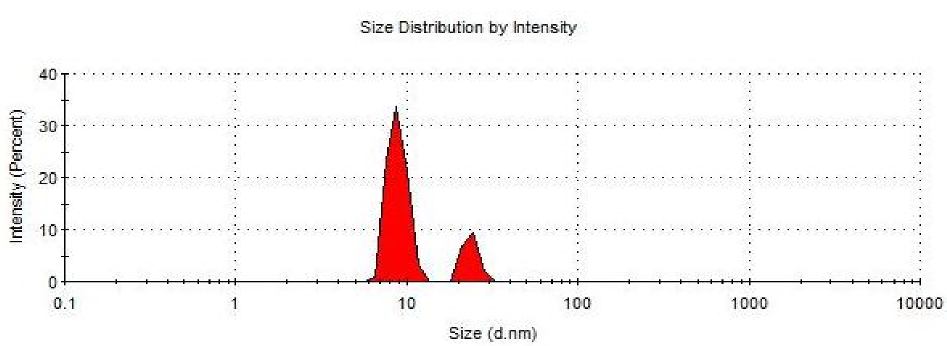

Figure 1. Particle intensity curve in PES/DMP/ solution.

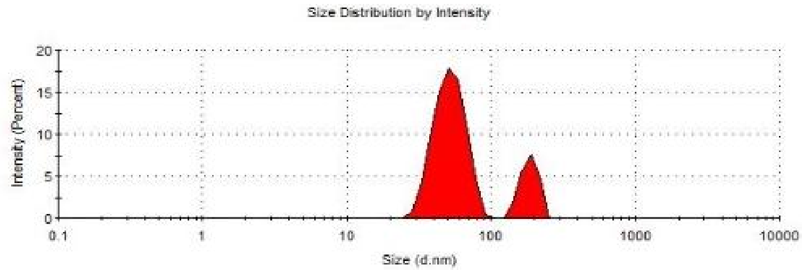

Figure 2. Particle intensity curve in PES/DMP/5% PV solution.

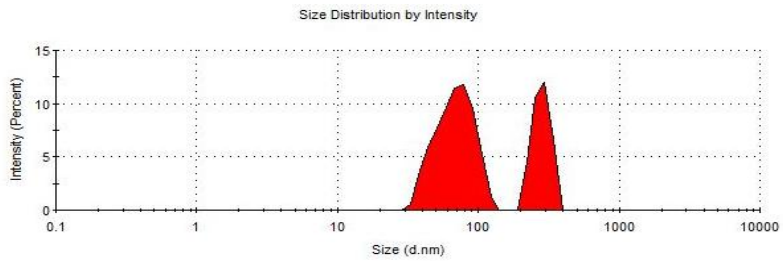

Figure 3. Particle intensity curve in the PES/DMP/7%PVP composition.

The resulting compositions were precipitated in water at 10°C, 20°C, 30°C, and 40°C. Infrared (IR) spectroscopy was used to analyze the chemical structure of the membranes precipitated at different temperatures. No significant differences were observed in the spectra of membranes precipitated at different concentrations and temperatures (Figures 4 and 5).

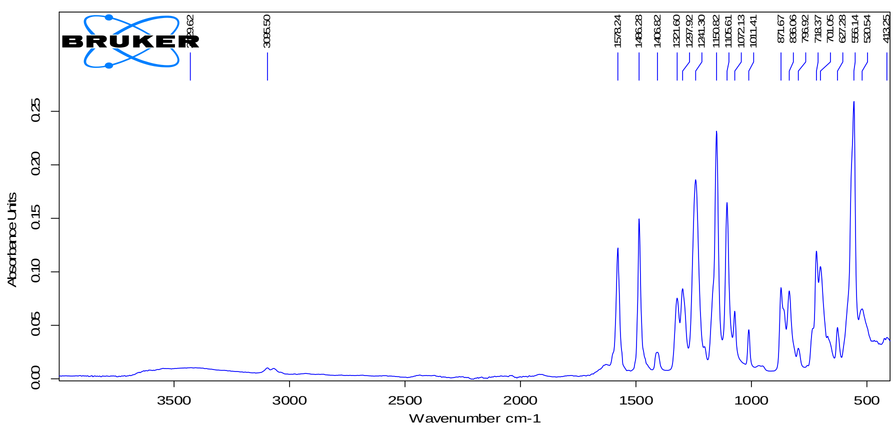

Figure 4. Infrared image of the N2 membrane

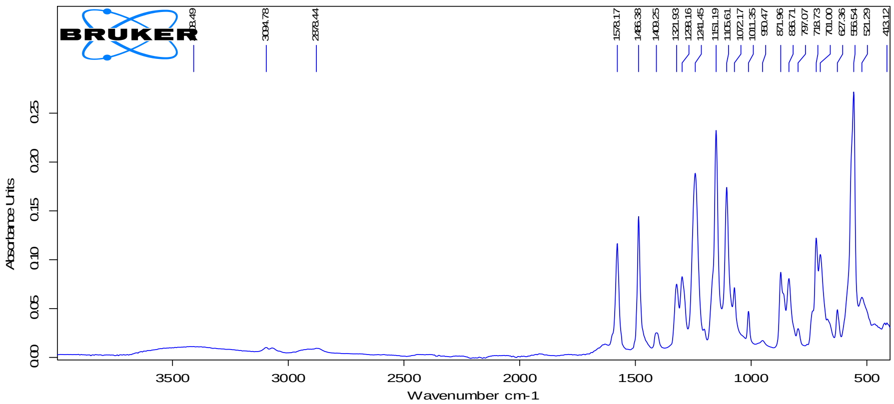

Figure 5. Infrared image of the N6 membrane

All membranes have similar peaks, which is due to the properties of the main polymer polyethersulfone. Because during precipitation the additive is transferred to water. Figures 4 and 5 show the spectra of the membrane samples. 621 cm-1 corresponds to C, 882 cm-1 to the unpaired bond C=C of the aromatic core. The peaks at 1150 cm-1, 1235 cm-1 and 1483 cm-1 are attributed to the sulfonic group O=S=O, while the aromatic ether group C-O-C is at 1296 cm-1. The peak at 706 cm-1 indicates the presence of a C-S bond.

The morphological changes of the surface of membranes deposited at different temperatures were imaged using a scanning probe microscope. The addition of hydrophilic polyvinylpyrrolidone to the deposition solutions resulted in a change in the morphology of the resulting membranes. The surface topography of membranes deposited at a low temperature of 10℃ (Figures 6, 7) with both 5% and 7% PVP addition is worse than that of membranes obtained by performing phase inversion at 20℃ and 30℃ (Figures 8). On the surface of membranes deposited at 10℃ and 40℃, roughness is observed, porosity decreases, and despite the increase in pore size, there is a decrease in performance.

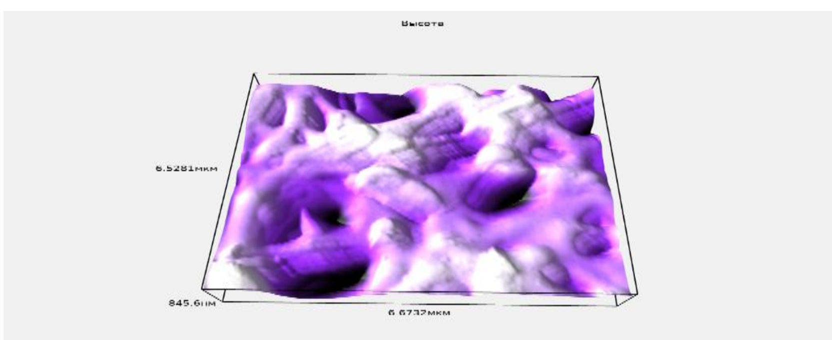

Figure 6. N1 membrane

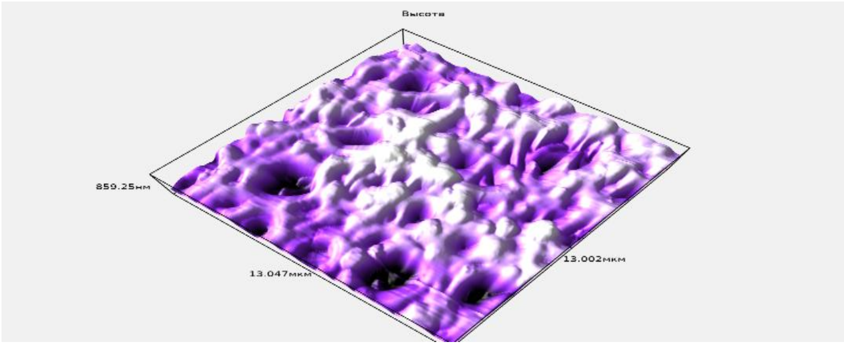

Figure 7. N5 membrane

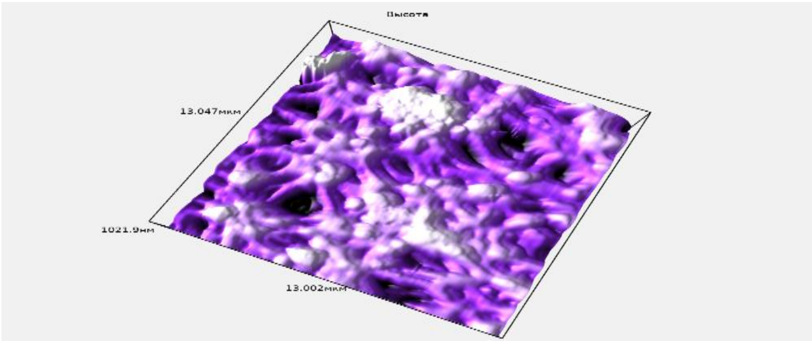

Figure 8. N2 membrane

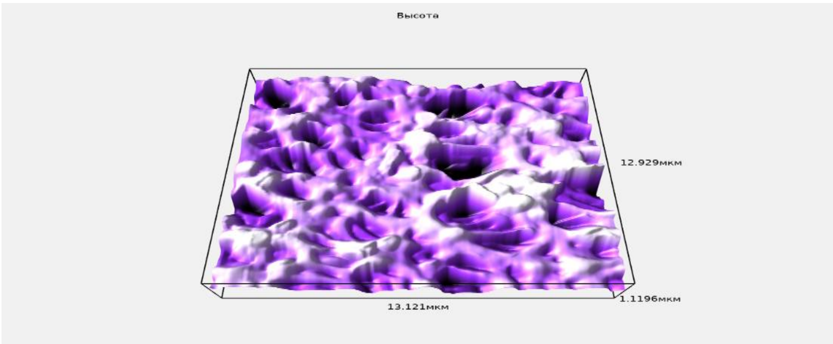

Figure 9. N7 membrane

The characteristics of membranes deposited from 15% polyethersulfone polymer solutions containing 5% and 7% PVP at different temperatures are given in Table 1.

Table 1. Characteristics of membranes deposited at different temperatures from PES/DMP/5%PVP and PES/DMP/7%PVP systems.

Polymer composition	PVP, concentration%	Precipitation temperature, °C	Membrane	Productivity l/m2h	Pore size, D µm
PES/DMP/PVP	5	10	N1	198	0,45
		20	N2	321	0,57
		30	N3	298	0,76
		40	N4	163	0,77
PES/DMP/PVP	7	10	N5	142	0,63
		20	N6	318	0,56
		30	N7	347	0,61
		40	N8	176	0,89

Result and conclusion Experimental studies have shown that the temperature of the coagulation bath is a critical parameter for the production of phase inversion polyethersulfone membranes. By controlling the temperature and other parameters, it is possible to adapt the properties and structure of the membranes to specific separation processes. Analysis of the results revealed that among the membranes precipitated from solutions containing 5% PVP, the membrane precipitated at 20℃ has a better structure and performance, while among the membrane samples obtained from solutions containing 7% PVP, the membrane precipitated at 30℃ has a better structure and performance. The result indicates that both the nature and concentration of the additive, as well as the phase inversion temperature, have a significant impact on the physicochemical characteristics of the membranes.

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ფაზური ინვერსიის ტემპერატურის გავლენა პეს/დმფ/პვპ პოლიეთერსულფონური მემბრანების ფორმირებაზე და წარმადობაზე.

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რეზიუმე

ნაშრომში შესწავლილია 15%-იანი პოლიეთერსულფონის პეს/დმფა/5%პვპ და პეს/დმფა/7%პვპ შედგენილობის ხსნარებიდან პოლიმერული მემბრანების მიღება ფაზური ინვერსიის მეთოდით გამოლექვის განსხვავებულ ტემპერატურაზე. მიღებულია ორი ჯგუფი პოლიეთერსულფონური მემბრანებისა და თითოეულისთვის გამოლექვა ჩატარებულია ოთხ ტემპერატურაზე (10°C , 20°C, 30°C, 40°C). დასასხმელ ხსნარებში შესწავლილია ნაწილაკის ზომები და პოლიდისპერსობის ხარისხი ანალიზატორ Malvern-ზე. დადგენილია ფაზური ინვერსიის ჩატარების ოპტიმალური ტემპერატურა პვპ-ს ორი განსხვავებული კონცენტრაციის პირობებში და საკოაგულაციო აბაზანის ტემპერატურის გავლენა მიღებული მემბრანების წარმადობაზე, სტრუქტურაზე და ფორის ზომებზე. 5%პვპ-ს შემცველი ხსნარიდან გამოლექილი მემბრანებიდან უკეთესი წარმადობა და სტრუქტურა აღმოაჩნდა 20°C-ზე გამოლექილ მემბრანას, ხოლო 7%პვპ-ს შემცველი ხსნარიდან გამოლექილი მემბრანებიდან 30°C-ზე მიღებულ მემბრანას.

საკვანძო სიტყვები: პოლიეთერსულფონი, ფაზური ინვერსია, DMF, PVP,ხვ.წარმადობა