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# Research on the processes of phase inversion and membrane partitioning of polymer of different compositions

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Abstract

The paper examines the process of obtaining 13% polyethersulfone (PES) microfiltration membranes by phase inversion using DMF (dimethylformamide) as the solvent and PEG400 (polyethylene glycol) with different mass percentages as the additive. The intensity and IR spectra of particle sizes in the cast composite solutions have been studied. Inversion of the compositions has been carried out at 30°C and 50°C. The influence of the coagulation bath temperature on the characteristics of membranes precipitated from solutions containing different amounts of PEG has been determined. Membranes have been tested. The surface morphology of the obtained membranes was also studied using a scanning probe microscope. Experimental studies have shown that membranes deposited at 50°C from solutions of the same composition have higher performance and pore size than membranes deposited at 30°C. Binodal curves were constructed when the system was subjected to phase inversion at 30°C and 50°C.

**Keywords:** membrane, PES, PEG 400, binodal, Performance

Conducting the phase inversion process is one of the important stages in the creation of membranes. During the mentioned process, a liquid-liquid dispersion, i.e. a homogeneous polymer solution, is transformed into a solid membrane, simultaneously evaporating and diffusing [1,2,3,4]. In the coagulation bath, the solvent and pore-forming additive are completely expelled and a non-solvent-impregnated membrane is formed. This process involves complex, rapid changes in the time interval of 1-2 seconds, accompanied by dispersion breakdown and coalescence and ultimately a decrease in the free energy of the system. The course of the process is influenced by parameters such as polymer type and concentration, solvent, additives, humidity, membrane thickness, composition and temperature of the coagulation bath, and the angle and speed of sample entry into the bath [5,6].

The paper discusses the changes in the characteristics and morphology of membranes deposited from polymer compositions of different compositions under different temperature conditions of the coagulation bath. It is known that temperature is a critical parameter when conducting phase inversion, it affects both the kinetic (exchange rate) and thermodynamic (dissolution) processes occurring during the inversion. Increasing the bath temperature reduces the viscosity of the composite solutions and accelerates the solvent-nonsolvent exchange and the solidification process[7,8,9]. The morphology of the membrane is significantly dependent on the solvent diffusion rate, which means that the mass transfer rates of the solvent and nonsolvent are of decisive importance during phase separation. At higher temperatures, the viscosity of the casting solution decreases, the exchange rate and the solidification process are accelerated. Precipitation at high temperatures leads to an increase in pore size and performance.

The membrane-forming polymer polyethersulfone, dissolved in dimethylformamide and supplemented with varying amounts of PEG 400, was chosen as the object of study. The polyethersulfone was dissolved in dimethylformamide in a 100 ml flask by heating at 55°C for 24 h under constant stirring with a magnetic stirrer, and a 12% PES/DMF composition was obtained. PEG 400 was added to the resulting polymer solution in amounts of 3%, 5%, 7% and 9% (wt%) and stirring was continued for another 1 hour. The particle sizes and particle distribution intensity in the compositions were studied on a Malvern analyzer. Figure 1 shows the particle size distribution curves in the composite solutions.

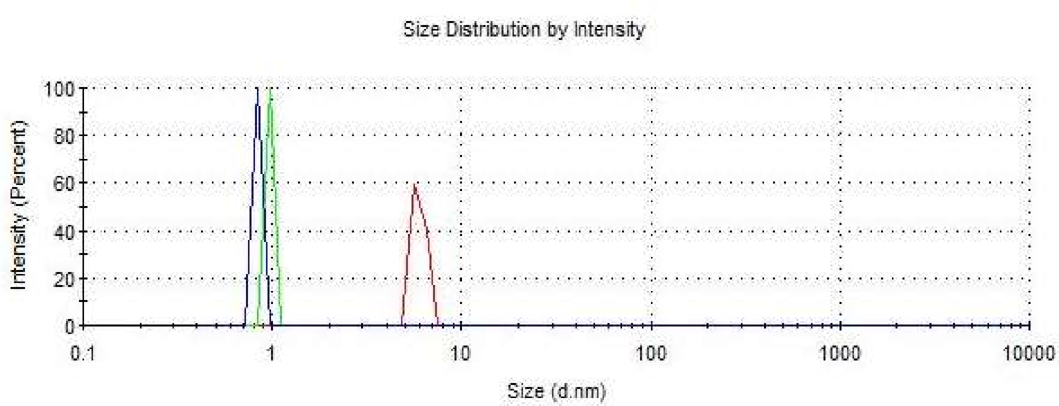


Figure 1. Particle size distribution in PES/DMF/5%PEG400, PES/DMF/10%PEG400, and PES/DMF/20%PEG400 compositions. 1. Curves of PES/DMF/5%PEG400.

In the polymer compositions, the particle sizes and intensities vary to match the change in PEG concentration, from 1 nm to 8 nm. In the composition of PES/DMF/20% PEG400, the intensity of 8 nm particles is 60%.

Infrared (IR) analysis of the resulting compositions was also performed. Figures 2 and 3 show IR spectra of the composite solutions of PES/DMF/5%PEG400 and PES/DMF/20%PEG400.

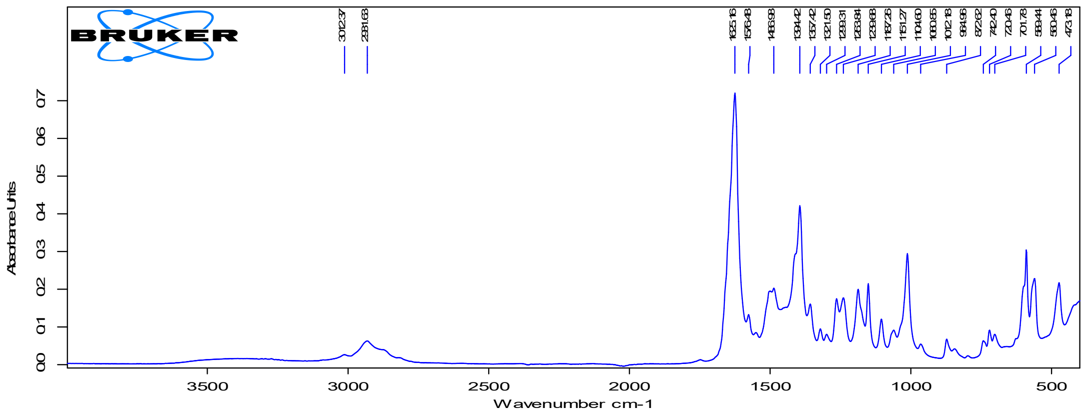


Figure 2. IR spectrum of the PES/DMF/5%PEG400 composition

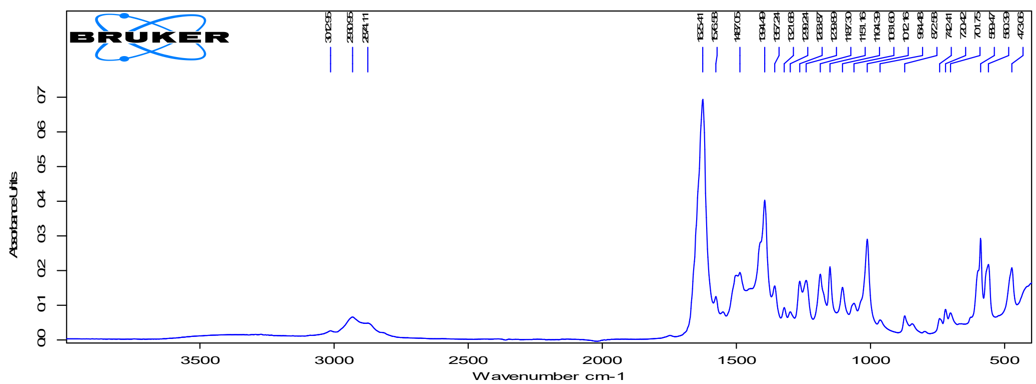


Figure 3. IR spectrum of the PES/DMF/20% PEG400 composition

For solutions containing 5% and 20% PEG the images of the polymer compositions are almost identical. The absorption band of aromatic nuclei in solution for both compositions is 1666 cm-1. The (C-O-C) aromatic ether group corresponds to 1280- 1150 cm-1, the absorption band of the aromatic (C=C) group is 1483 cm-1 and 1510 cm-1, the absorption band of the (O=S=O ) groups corresponds to 1151 cm-1, 1239 cm-1, 1483 cm-1, the peak at 621 cm-1 corresponds to C.

The incomplete binodal of the 13% PES/DMF/PEG400 system, which is incomplete due to the constant concentration of PES, has been studied. Figure 4 shows the composition of the compositions, and Figure 5 shows the binodal segment for the given concentrations of PEG and PES.

| N | PES, mass % | DMF mass % | PEG mass % |
|---|---|---|---|
| 1 | 13 | 82 | 5 |
| 2 | 13 | 77 | 10 |
| 3 | 13 | 72 | 15 |
| 4 | 13 | 67 | 20 |

Figure 4. Composition of the polymer composition PES ▲ )

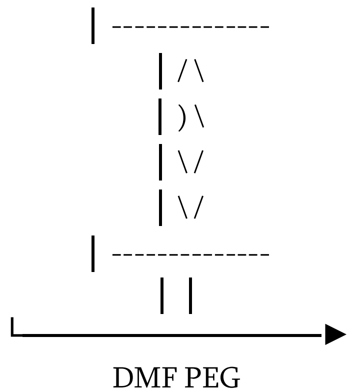


Figure 5. Incomplete binodal segment of the PES/DMPA/PEG400 system.

Three-component phase diagrams were constructed for this system in relation to precipitation temperatures of 30 °C and 50 °C. On figure 6 three-component phase diagrams of the 13% PES/DMF/PEG400 system during precipitation at 30 °C and 50 °C are given.

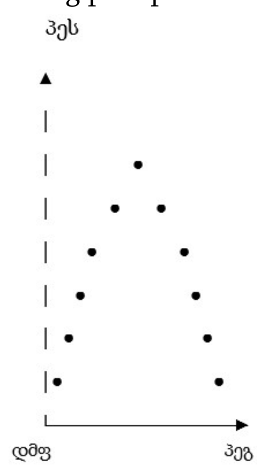


Figure 5. Phase diagram of the 13% PES/DMF/PEG400 system during precipitation at 30 °C.

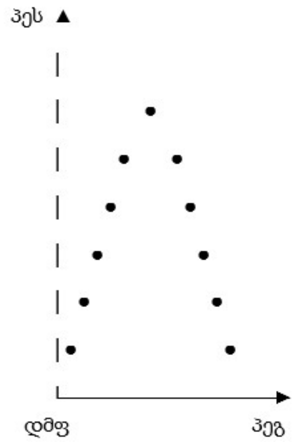


Figure 6. Phase diagram of the 13% PES/DMF/PEG400 system during precipitation at 50 °C.

Comparing the binomials, it can be seen that a low concentration of PES requires a larger amount of PEG for phase separation, while a high concentration of PES requires a larger amount of PEG, and a high concentration of PES requires a smaller amount of PEG. At high precipitation temperatures, the spinodal contracts and moves toward the solvent. Separation occurs due to nucleation, while at low precipitation temperatures, the spinodal expands and moves toward the nonsolvent, the system becomes more unstable, and the spinodal breaks down.

The surface topography of the membranes obtained by phase inversion was studied using a scanning probe microscope. The results showed that the N4 membrane had the best topography among membrane samples obtained from solutions containing different amounts of PEG at different temperatures. With increasing PEG concentration, the surface structure of the membranes changes from a dense, fine-pored to a more macroporous surface structure with interconnected channels. The N4 membrane is distinguished by its surface uniformity and pore frequency. Figure 7 and 8 show micrographs of the N4 and N8 membranes.

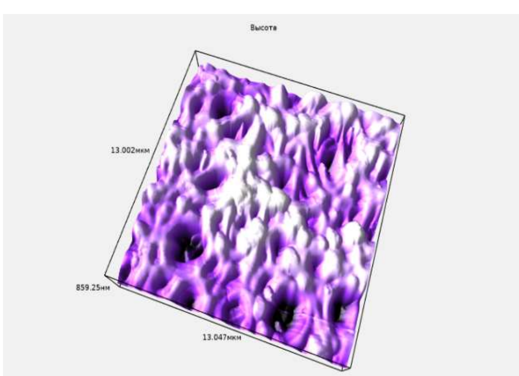


Figure 7. Micrograph of N4 membrane

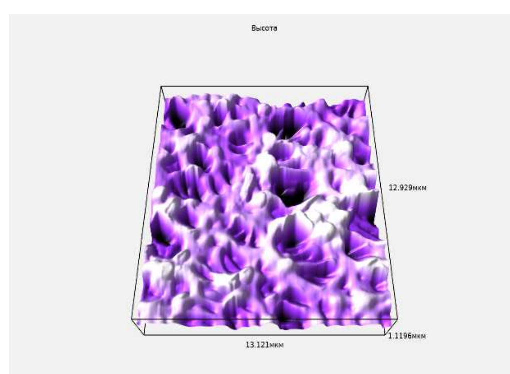


Figure 8. Micrograph of N8 membrane

The phase inversion process of the four resulting compositions was carried out at 30°C and 50°C. The composition of the compositions and the characteristics of the precipitated membranes when tested on water are given in Table 1. The aim was to determine the effect of the additive concentration in the PES/DMF/PEG400 solution and the phase inversion temperature on the properties of the resulting membranes.

| Composition of 13% PES | PEG mass % | Precipitation temperature°C | Membrane | Pore size, μm | Performance l/m2h | Porosity, % |
|---|---|---|---|---|---|---|
| PES/DMF/PEG400 | 5 | 30 | N1 | 0,42 | 745 | 15±3 |
| PES/DMF/PEG400 | 10 | 30 | N2 | 0,53 | 921 | 20±2 |
| PES/DMF/PEG400 | 15 | 30 | N3 | 0,67 | 1006 | 25±4 |
| PES/DMF/PEG400 | 20 | 30 | N4 | 0,7 | 1128 | 54±2 |
| PES/DMF/PEG400 | 5 | 50 | N5 | 0,58 | 804 | 33±1 |
| PES/DMF/PEG400 | 10 | 50 | N6 | 0,61 | 1232 | 42±3 |
| PES/DMF/PEG400 | 15 | 50 | N7 | 0, 67 | 1534 | 39±2 |
| PES/DMF/PEG400 | 20 | 50 | N8 | 0,9 | 1723 | 46±1 |

Membrane testing showed that increasing the PEG concentration at the same precipitation temperature increases the pore size and performance of the precipitated membranes. Increasing the coagulation bath temperature from 30°C to 50°C resulted in a more significant increase in pore size and performance for the precipitated membranes. This is also confirmed by the three-phase diagrams. A better porosity index was found for the N4 membrane. Of the membranes precipitated at 30°C, the N4 membrane had better characteristics, and of the membranes precipitated at 50°C, the N8 membrane. Three-phase diagrams of polymer compositions for a constant concentration of PES during precipitation at different temperatures have been studied. A semi-empirical thermodynamic model has been created.

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# განსხვავებული პოლიმერული კომპოზიციების ფაზური ინვერსიის და მემბრანული დაყოფის პროცესების კვლევა

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რეზიუმე

გამოკვლეულია 13%-იანი პოლიეთერსულფონის(პეს) მიკროფილტრაციული მემბრანების ფაზური ინვერსიით მიღების პროცესი გამხსნელად დმფ-ის(დიმეთილფორმამიდი) და დანამატად განსხვავებული მასური პროცენტით პეგ400 (პოლიეთილენგლიკოლი) გამოყენების შემთხვევაში. შესწავლილია დასასხმელ კომპოზიციურ ხსნარებში ნაწილაკების ზომების ინტენსივობა და იწ სპექტრები. კომპოზიციების ინვერსია ჩატარებულია 30-ზე და 50-ზე.დადგენილია საკოაგულაციო აბაზანის ტემპერატურის გავლენა განსხვავებული რაოდენობით პეგ-ის შემცველი ხსნარებიდან გამოლექილი მემბრანების მახასიათებლებზე. ჩატარებულია მემბრანების ტესტირება. ასევე მასკანირებელი ზონდური მიკროსკოპით შესწავლილია მიღებული მემბრანების ზედაპირული მორფოლოგია. ექსპერიმენტული კვლევებით დადგენილია, რომ ერთნაირი შედგენილობის ხსნარებიდან 50-ზე ტემპერატურაზე გამოლექილ მემბრანებს გააჩნიათ უფრო მაღალი წარმადობა და ფორის ზომა ვიდრე 30-ზე გამოლექილ მემბრანებს. აგებულია ბინოდალური მრუდები სისტემის ფაზური ინვერსიის ჩატარებისას 30-ზე და 50-ზე.

**საკვანძო სიტყვები:** მემბრანა, პეს, პეგ 400, ბინოდალი, წარმადობა.