Original Article

Synthesis of a New Ion Exchange Membrane based on Aniline and Nitrocellulose

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Abstract - *In this research work, the synthesis of polyanilinenitrocellulose (PANS) with the participation of aniline(AN) and nitrocellulose(NS) was carried out, and an ion exchange membrane was obtained based on it. The substances were synthesized at different mole ratios and at different temperatures, as well as at different time intervals, and the optimal process conditions were determined. As a result, aspects of using PANS as new ion exchange membranes were studied based on their physico-chemical properties. In addition, based on the results of IR-spectrum, SEM, mechanical test, and thermal analysis of the synthesized PANS, its analysis data were presented.*

1. Introduction

Protection of water resources and their rational use is one of the most global problems today. Membrane, electrochemical, and other physico-chemical methods of water quality improvement and wastewater detoxification, which enable water reuse in technological processes, are very important [1], [2]. Reusing water in production processes allows for reducing the consumption of fresh water and the discharge of polluted water and return to production of previously produced valuable components[3], [4]. The use of multifunctional high-capacity ion exchangers and membranes allows solving the current problems of import substitution in the development of waste-free sorption technologies and electrodialysis [5]. Technologies such as baramembrane, membrane ion exchange, and electromembrane desalination have been developed to ensure water safety. Ion exchange membranes produce clean water unfit for consumption [6], IEMs are membranes with charged groups that attract counterions and repel ions from ionic solutions. IEMs that conduct anions are called anion-exchange membranes (IEMa), and those that conduct cations are called cation-exchange membranes (IEMk) [7]. To be effective, IEMs must have a high Ion Exchange Capacity (IEC), be highly conductive and highly ion-storing, and be chemically stable [8]. When polymeric ion-exchange membranes are classified according to the method of introduction of ion-exchange groups, they are divided into homogenous and heterogeneous. For homogeneous IEMs,

the ion exchange groups are chemically attached directly to the polymer bonds; these membranes are chemically more stable. Heterogeneous IEMs have a dispersed distribution of independent ion exchange groups in the polymer structure [9]. The advantages of heterogeneous IEMs can be tuned by changing the loading of functional group ions that give them ion exchange properties. Obtaining a homogeneous material by adding different proportions of ion-exchange additive particles and polymers has lower production costs than other IEMs [10]. More interfacial nanotechnology versatile approaches are required to incorporate ion exchange functional groups into heterogeneous IEMs. Using nanomaterials as ion exchange additives is a known and acceptable strategy. Compared with other IEMs, IEMs developed by adding additives to polymer membrane matrices have been easier to synthesize [11]. The formation of nano and micropores and ion exchange groups in homogeneous IEMs shows significant advantages in ion exchange, mechanical strength, and other properties. One of the most popular classes of polymers that form IEMs is cellulose and its derivatives, such as cellulose esters. These include mixed esters of cellulose acetate, cellulose triacetate, cellulose tripropionate, ethylcellulose, and cellulose nitrate [12]. Membranes and filters based on cellulose and its derivatives are used not only for microfiltration and ultrafiltration but also for reverse osmosis, gas separation, protein separation, and electrodialysis. Cellulose esters represent a very important class of basic membrane materials

[13]. This is because the main ingredient, cellulose, is a polysaccharide that can be obtained from plant materials [14]. Cellulose derivatives such as cellulose nitrate and cellulose acetate have been partially used for microfiltration and ultrafiltration, while cellulose triacetate shows good properties for reverse osmosis membranes in water desalination. Despite their excellent membrane properties, cellulose ethers are very sensitive to thermal, chemical, and biological effects.

Therefore, processing is required to obtain the necessary properties from the membranes. Due to the limited ion exchange capacity of IEMs obtained using different materials, it is possible to obtain selective IEMs by introducing functional groups into them. Excessive additives will reduce the ion exchange capacity of IEMs. In addition, additives can be used to improve their morphology [15]. Strategies to improve the properties of IEMs have been explored to increase separation efficiency and reduce energy consumption of electromembrane desalination processes. It has been proven that the permeability of membranes can be increased based on polyaniline [16].

The main problem with IEMs is that their physicochemical properties decrease or disappear if they do not have the necessary substances and environment for their formation. During the synthesis of the IEM polymer found in many literatures, its morphology and ion exchange possibilities were discussed. Considering the physicochemical properties of polyaniline, we also synthesized a new PANS membrane using polyaniline and an in situ polymerization method. The properties of PANS, such as high surface area, mechanical stability, electrical conductivity, and sufficient thermal stability, have shown their suitability for use in the field of IEMs. In addition, PANS is easily dispersed in the solvents commonly used to shape the membrane. Therefore, the availability of PANS-based membranes attracts attention as universal IEMs. As a result of the non-homogeneous distribution of the used pore-forming substances, failure parts appear in the structure of the membranes and prevent their practical use. We propose another option for developing porous IEMs: copolymers are obtained based on the chemical bonding of PA and NS. PANS is a membrane-forming copolymer characterized by ease of production and processing. The synthesized PANS membrane was tested to achieve the highest possible loading, and its physicochemical properties were studied.

2. Experimental Part
2.1. Material and Methods
The following materials and equipment are needed to prepare the membrane. Nitrocellulose (NS), aniline(AN) 99%, ethyl ether(ETS) 99%, Diglycidylamine (DGA) 20%, acetone (AT) 96%, ammonium persulfate (Amps) 96%, glycerin (GLT) 96%, N-methyl-2-pyrrolidone (NMP) 98%, isobutyl alcohol (IBS) 99%, ethylene glycol (ETG) 98%, hydrochloric acid 24%, distilled water. Flasks and mixing devices are used to carry out reaction processes. Nitrocellulose was first synthesized by nitrating cotton cellulose in sulfuric acid at low temperatures. The obtained substance was treated with 5% nitric acid, neutralized with distilled water, and dried at room temperature for reaction.

2.2. Synthesis of PANS Raw Materials
PANS were copolymerized with nitrocellulose and aniline using the in situ polymerization method in the presence of ammonium persulfate and hydrochloric acid catalysts. First, nitrocellulose was taken and dried at 45 °C for one day, then placed in a three-necked. Then, aniline was introduced into the reactor and mixed with nitrocellulose at low temperatures. As a catalyst, hydrochloric acid and ammonium persulfate solutions were slowly added dropwise.

The substances were continuously stirred during the reaction time. After the catalysts had been thoroughly introduced, they were stirred for another 2.5 hours, and blue-colored PANS were obtained and neutralized by washing them with distilled water. Then, the product was dried in the oven at a temperature of 40-45 °C for 8 hours. During the experiment, reactions were carried out by introducing aniline and nitrocellulose raw materials in different proportions. The synthesis reaction scheme of the synthesized PANS copolymer is shown in Figure 4. In the reaction of nitrocellulose and aniline with a mass ratio of 1:1.25, the process yield was 70% (Figure 1). The influence of the reactants' concentration, time, and temperature on the product yield was studied, and the results are presented in Table 1.

Table 1. To study the effect of concentration, time, and temperature of reactants on product yield

A mass ratio of initial products	Duration of the reaction (hours)	Temperature dependence of the reaction, °C	Reaction yield %
ANILINE-NITROCELLULOSE			
1:0,5	2,5	8	30
1:0,75	3,5	7	40
1:1	4	5	50
1: 1,25	4,5	0	70
1:1,5	5	-2	70
1:75	5,5	-4	70

Mass ratios are in grams

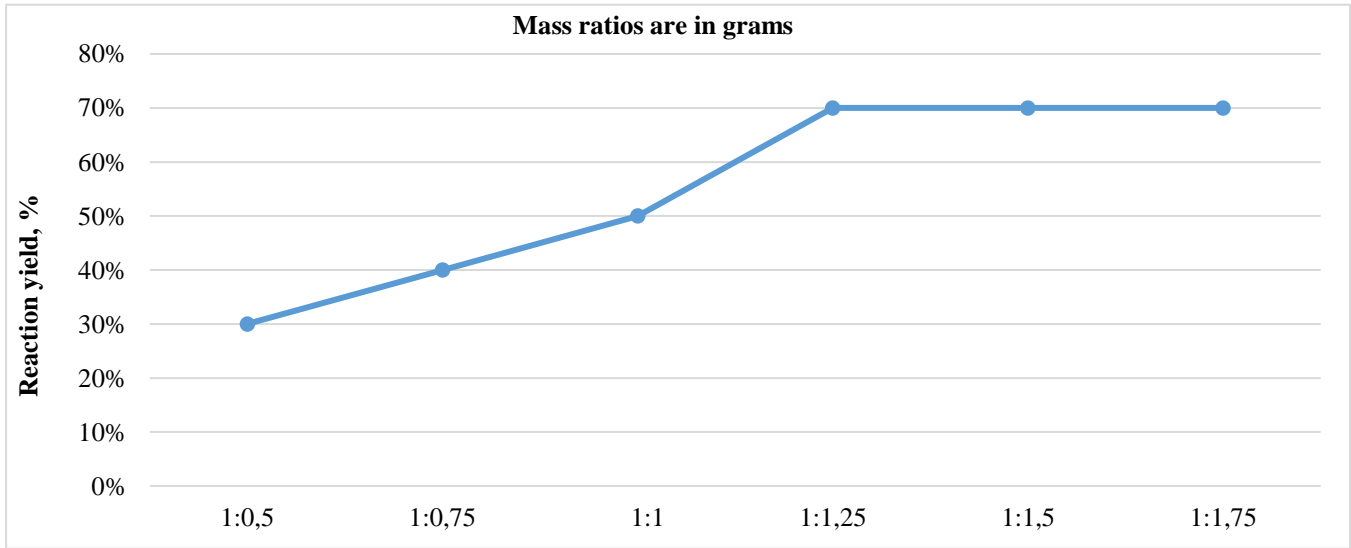**Fig. 1 The effect of mass ratios of starting materials on reaction efficiency during the synthesis of PANS raw materials**

Reaction temperature,°C

Fig. 2 The effect of temperature on the efficiency of the reaction during the synthesis of PANS raw material

Reaction time in hours

Fig. 3 The effect of time on the efficiency of the reaction during the synthesis of PANS raw materials
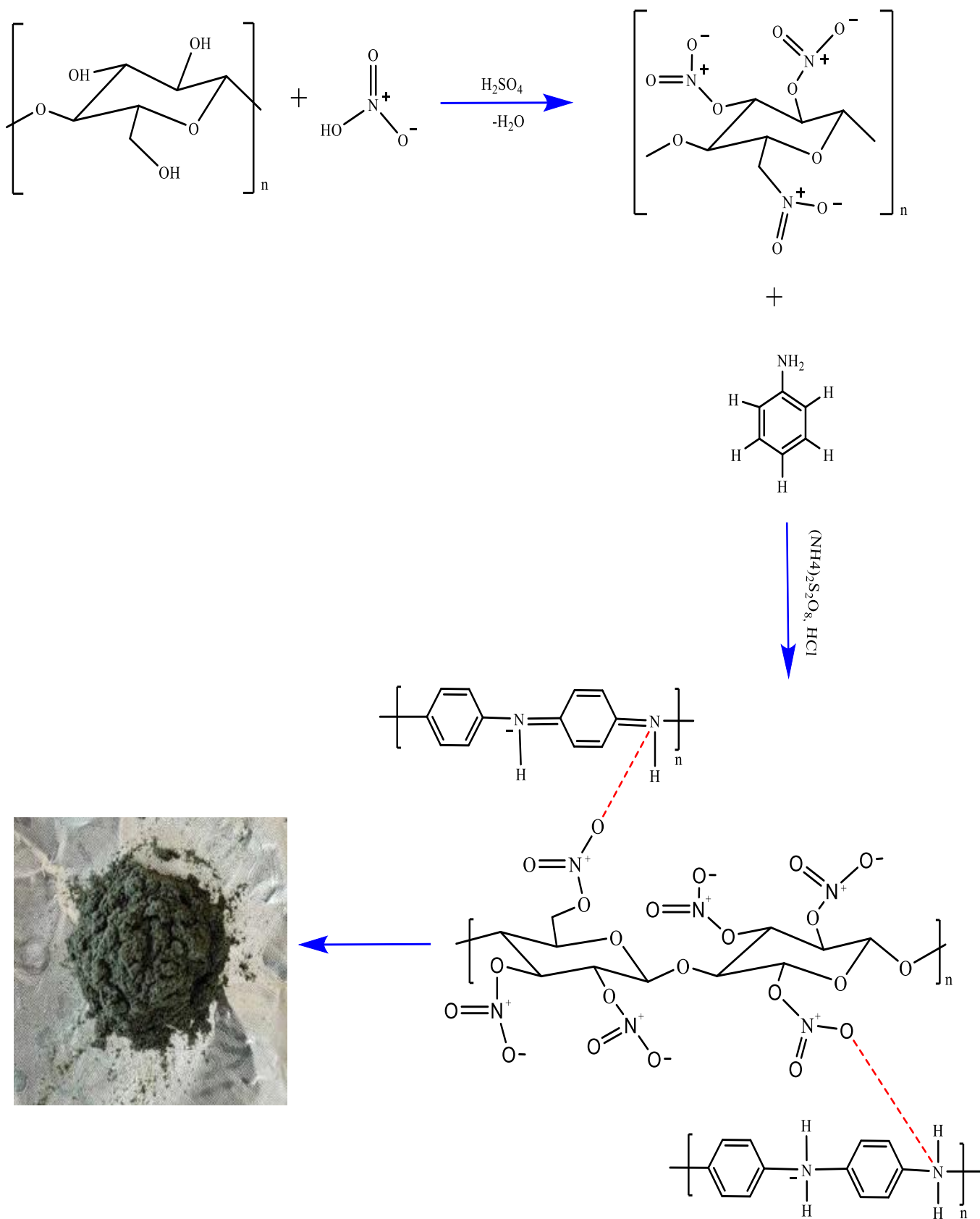
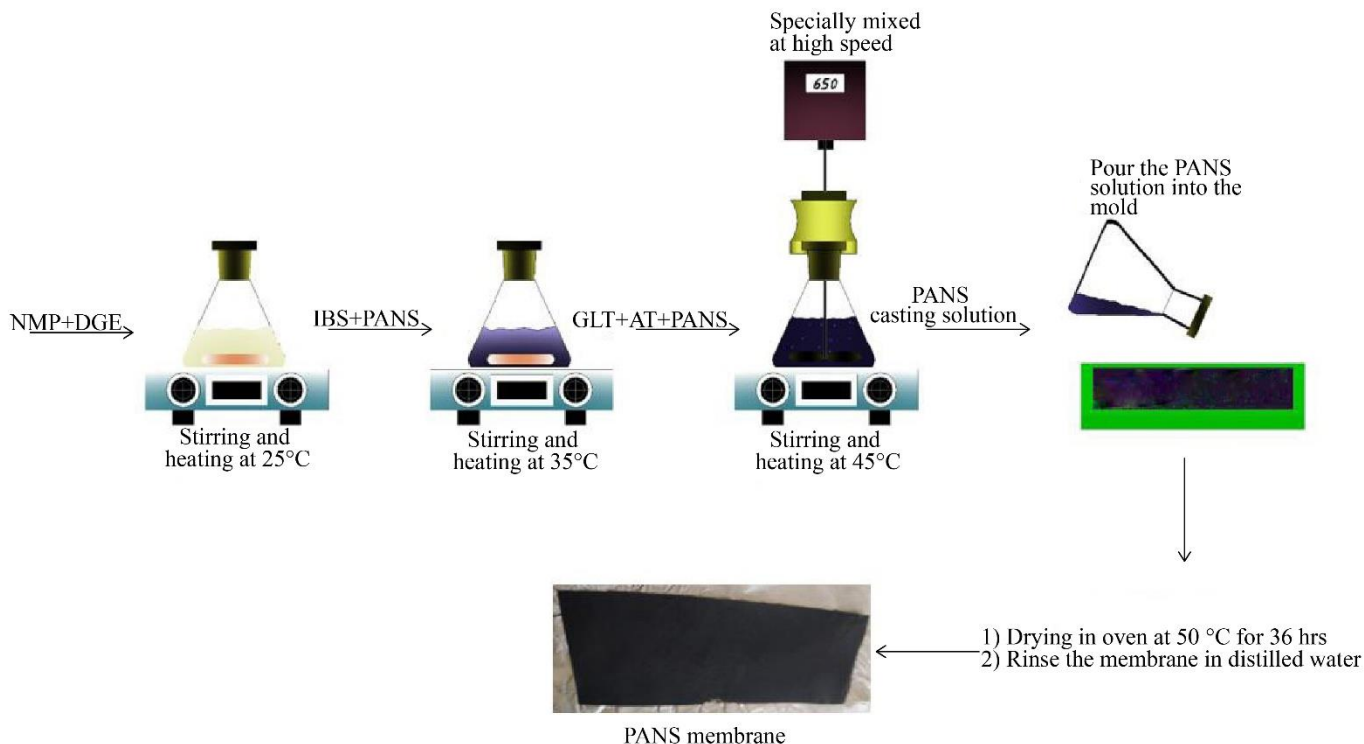**Fig. 4 Schematic representation of PANS raw material synthesis**

Fig. 5 Schematic of PANS ion exchange membrane preparation

2.3. Membrane Preparation

The PANS membrane was manufactured following the following steps (Figure 5). The first reaction flask was placed on a magnetic stirrer, then a solution of 25.35 g of NMP and 7.5 g of DGE was added and stirred at 25 °C for 35 min. In the next step, 3.5 g of IBS and 2 g of PANS were added to the solution and mixed for 1 hour at 35-37 °C to dissolve in a homogeneous state. At this stage, the color of the bulk solution changed from white-yellow to light brown. Then 1.65 g of 9.61% solution of GLT and another 1 gram of PANS were added to form a uniform dark blue bulk solution.

Then AT was added, equal to 22% of the total solution. The product was stirred for 2 hours at 45 °C in the next step. In this, the final composition of the Nm_4 membrane is 25.35 g NMP, 7.5 g DGE, 3.5 g IBS, 3 g PANS, 0.15 g GLT, 1.5 g water and 18 g ATs. Water was used to form the glycerin solution. The resulting solution of each PANS membrane was poured into a polyvinyl chloride mold made based on a casting solution. Membrane thickness can be controlled by controlling the volume of solution poured into the mold. Then, to evaporate the solvent of the resulting sample, it was dried in a special oven at a temperature of 50 °C and air humidity of 50-55%. The PANS membrane was immersed in DI water for cleaning when its weight was reduced by 55-60%. Then, it was dried again in the oven at 45-50 °C for 4 hours. Synthesis of the PANS membrane was also performed under different conditions and fillers. Its preparation scheme is shown in Figure 5, and its composition in Table 2.

In preparing ion-exchange NAIM membranes, the proportions of the substances listed in Table 2 were correctly distributed and synthesized in an exceptional environment and temperature under constant stirring.

Table 2. Cast composition of PANS ion exchange membrane

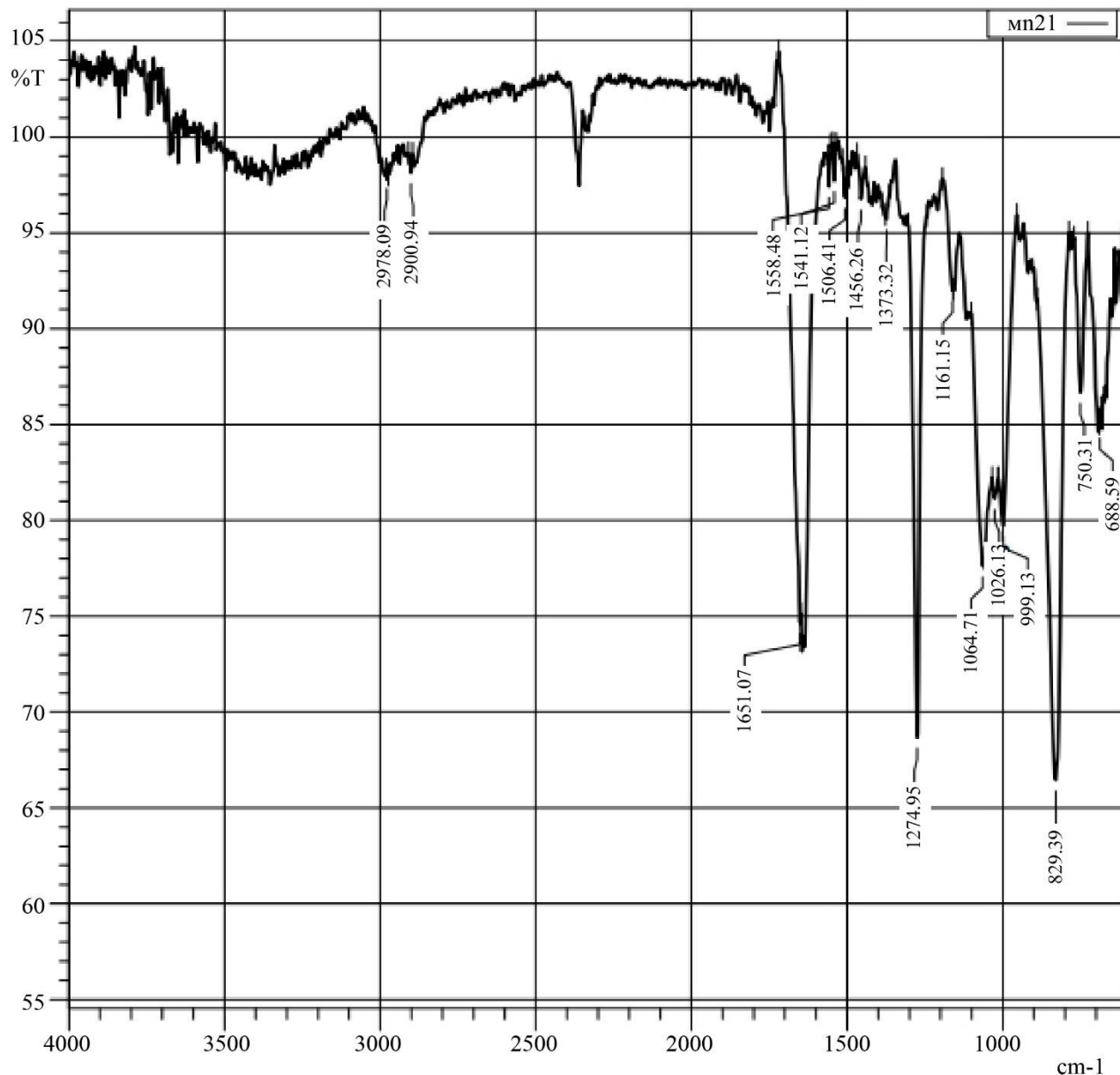| Component	Additive composition %					
	N_{m1}	N_{m2}	N_{m3}	N_{m4}	N_{m5}	N_{m6}
PANS	5	3	4,5	6	6	4.5
NMP	30.5	43	47	50,7	60,5	50,5
DGE	15	14	6	15	5	20
IBS	23	13.8	12	7	15,5	15
At	2	0,2	12,9	18	2	5
GLT	-	-	0,2	0.3	-	-
ETS	3	4	5	-	5	3
Suv	1,5		2	3		1
ETG	20	22	10	-	6	1

Fig. 6 IK-spectrum of raw material aniline

3. Characterization

3.1. Infrared Spectrum (IR) of synthesized PANS membrane and raw materials

Initially, the analysis of the infrared spectrum of substances was studied based on the necessary literature [22]. The IR spectrum of synthesized PANS membrane and raw materials NS and PA was analyzed by SHIMADZU IR-Fure spectrophotometer manufactured in Japan at 400 and 4000 cm-1. The analysis of the results obtained based on the IR spectrum is presented in Figures 6-8.

3.2. Scanning Electron Microscope (SEM)

The morphological structure of the membrane was studied using a JSM-Scanning in TouchScope electron microscope (SEM) from China JEOL Ltd. Porosity, and contact angle of flat membranes are determined by the attached bubble method. The analysis of results based on SEM is given in Figure 8.

3.3. Mechanical testing

The mechanical properties of the membrane sample were performed using the AUTOGRAPH AGS-X 10Kn SHIMADZU universal testing machine. The sample dimensions were 50 mm long, 10 mm wide, and 0.15 mm thick. Tensile strength and elongation for the samples were measured up to 30% at 1 mm/min and then at 20 mm/min. An average of three samples was recorded for all prepared membranes, and mechanical properties were determined.

3.4. Thermal analysis of the synthesized membrane.

Thermogravimetric (TGA) and differential thermal (DTA) analysis of the synthesized PANS membrane was performed by a SHIMADZU DTG-60 Derivatograph instrument manufactured in Japan. 2,002 mg of PANS membrane was analyzed, and the process was studied in a temperature range of 10-600 °C in an argon atmosphere. The results of the thermal analysis of the membrane are shown in Figure 8.

4. Results and Discussion

4.1. IR spectroscopic analysis of substances

The IR spectrum of polyanilinenitracellulose, aniline, and nitrocellulose used for its synthesis was obtained. The analysis of the obtained results is shown in Figures 6-8. In the values of Figure 6, we can see the presence of valence vibrations of Ar–NH_2 bonds in the area range of 3427.51 cm^{-1} and valence vibrations of –C=C– bonds in the area range of 1618.28 cm^{-1}

In the values of Figure 7, we can see the presence of valence vibrations of –O–NO_2 bonds in the area of 1645.28 cm^{-1}, valence vibrations of >CH_2 bonds in the 1319.31 cm^{-1} area, valence vibrations of –C–O–C– bonds in the area of 1273.02 cm^{-1}, valence vibrations of >CH– bonds in the field range of 1109.07 cm^{-1}. This shows that all the valence fluctuations in the results belong to the nitrocellulose polymer.
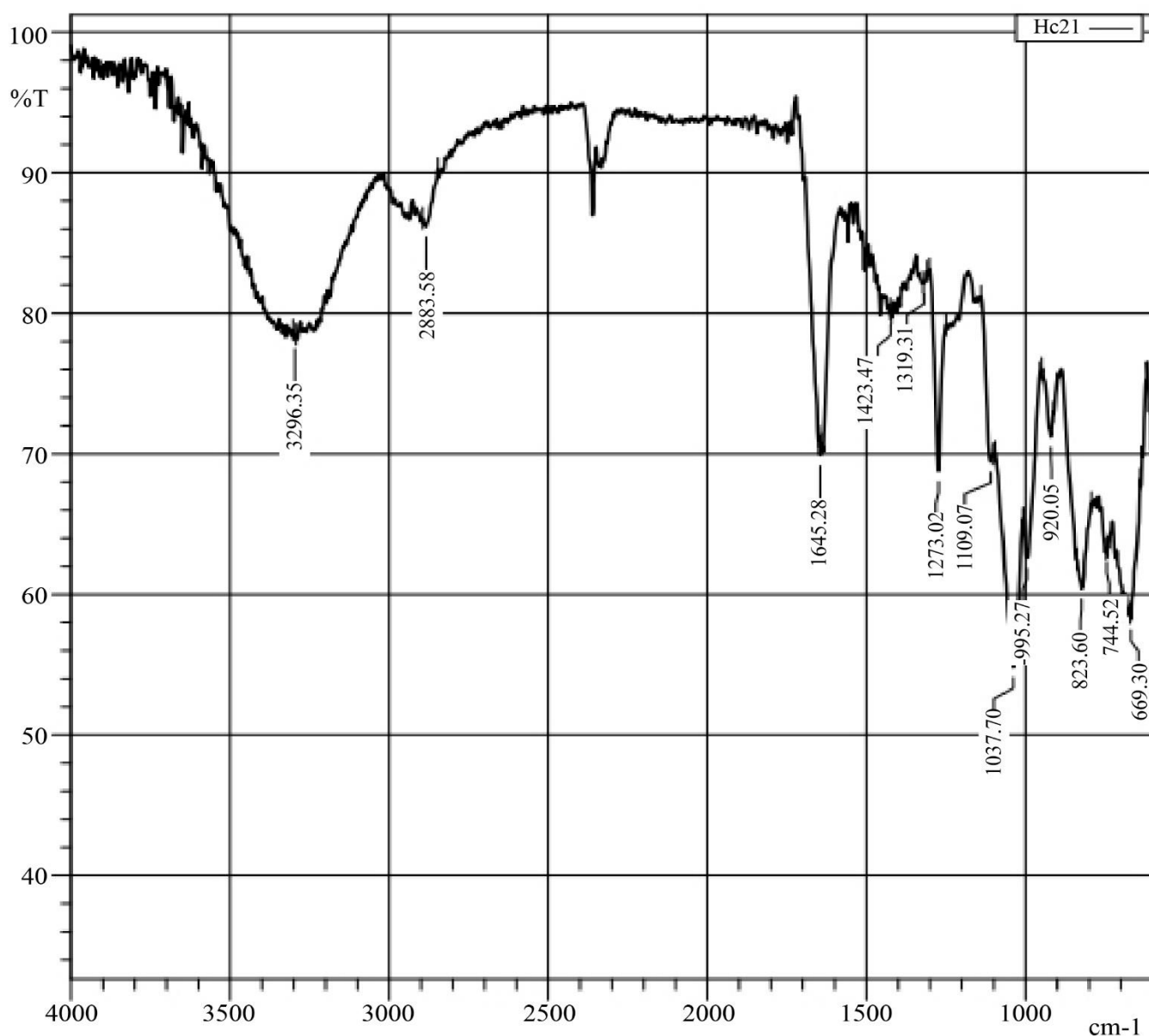
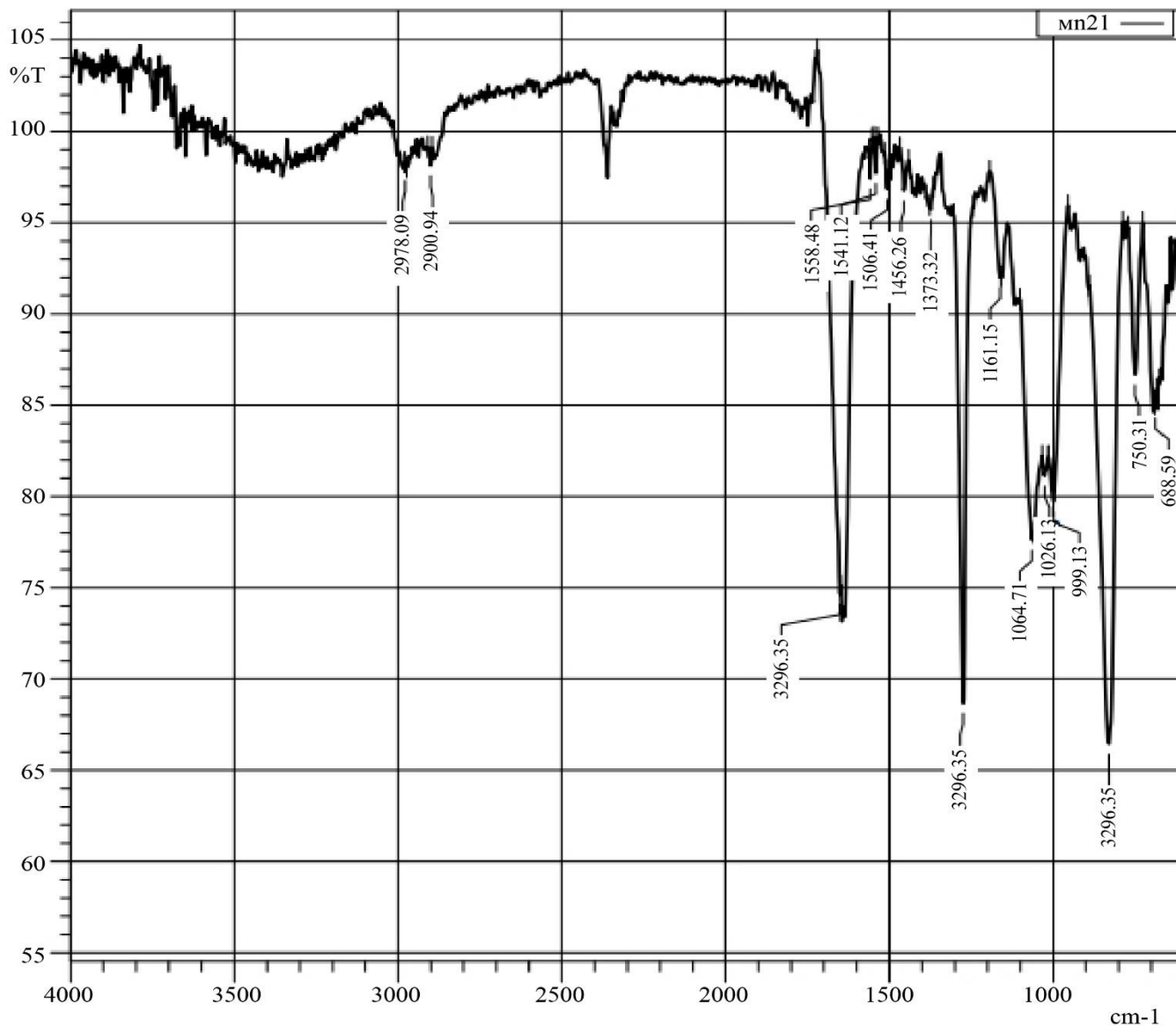**Fig. 7 IK spectrum of nitrocellulose raw material**

Fig. 8 IK-spectrum of PANS synthesized based on modification of nitrocellulose with aniline

In the values of Figure 8, we can see the presence of Valence vibrations of –CH= bonds in the area of 2900.94 cm^{-1}, Valence vibrations of $–O–NO_2$ bonds in the area of 1651.00 cm^{-1}, Valence vibrations of Ar –NH– bonds in the area of 1558.48 cm^{-1}, Valence vibrations of >C=NH–bonds in the area of 1506.41 cm^{-1}, Valence vibrations of $>CH_2$ bonds in the area of 1373.32 cm^{-1}, Valence vibrations of –C–O–C– bonds in the area of 1274.95 cm^{-1}, Valence vibrations of >CH– bonds in the area of 1161.15 cm^{-1}. We can see that the given valence vibrations correspond to the chemical formula of the substances we recommend.

4.2. Scanning Electron Microscopic (SEM) Analysis of PANS Ion Exchange Membranes

The surface morphology, pore structure, and elemental analysis of the ion exchange membrane were studied using a Scanning Electron Microscope (SEM) device. The resulting Nm_3 and Nm_4 ion exchange membranes were selected for SEM analysis. In Figures 4-5, the Nm_3 and Nm_4 membranes synthesized based on PANS showed porous structures on the surface and cross-sections.

If we look at the results, we notice that irregular and large pores were formed in the membrane due to the partial addition of more in the general pore-forming solution during the synthesis of the Nm_3 membrane. As a result, problems may arise in distributing substances that are transferred through the membrane and absorbed.

Figure 9 shows the surface morphology and pore size of the synthesized Nm_3 membrane in the top, bottom, and cross sections.
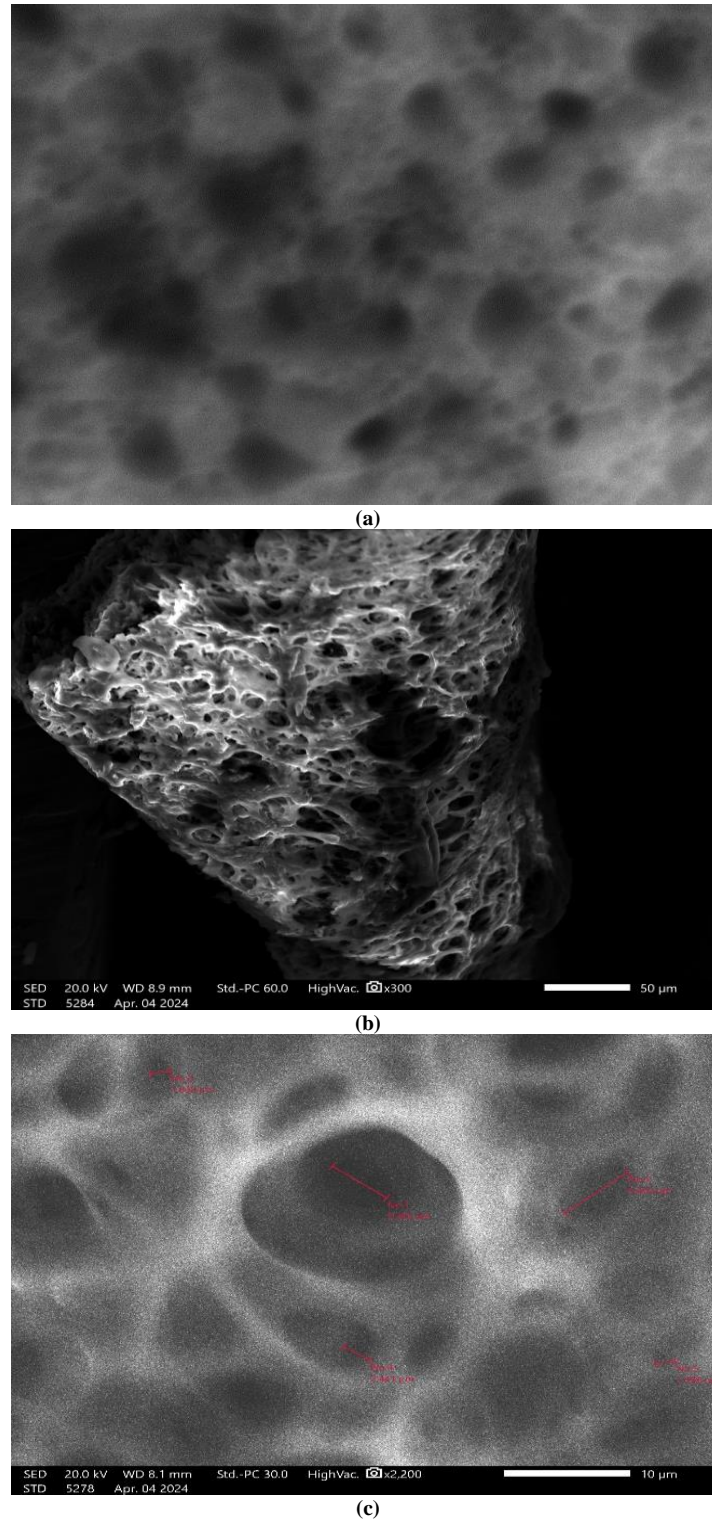
Fig. 9 SEM photographs of the prepared Nm$_3$ membrane, a) SEM photo taken from the top of the membrane, b) SEM photo of a cross-section of the membrane, c) SEM photo of the underside of the membrane

As a result of the SEM analysis of the Nm$_4$ membrane, we can see that the membrane contains nano and micropores of the same size and order. In synthesizing the Nm$_4$ membrane with isobutyl alcohol and glycerin, it acts as an auxiliary filler in forming nano and micropores. We can see the surface morphology and pore size of the synthesized Nm$_4$ membrane in the upper, lower, and cross-sectional parts in Figure 10.
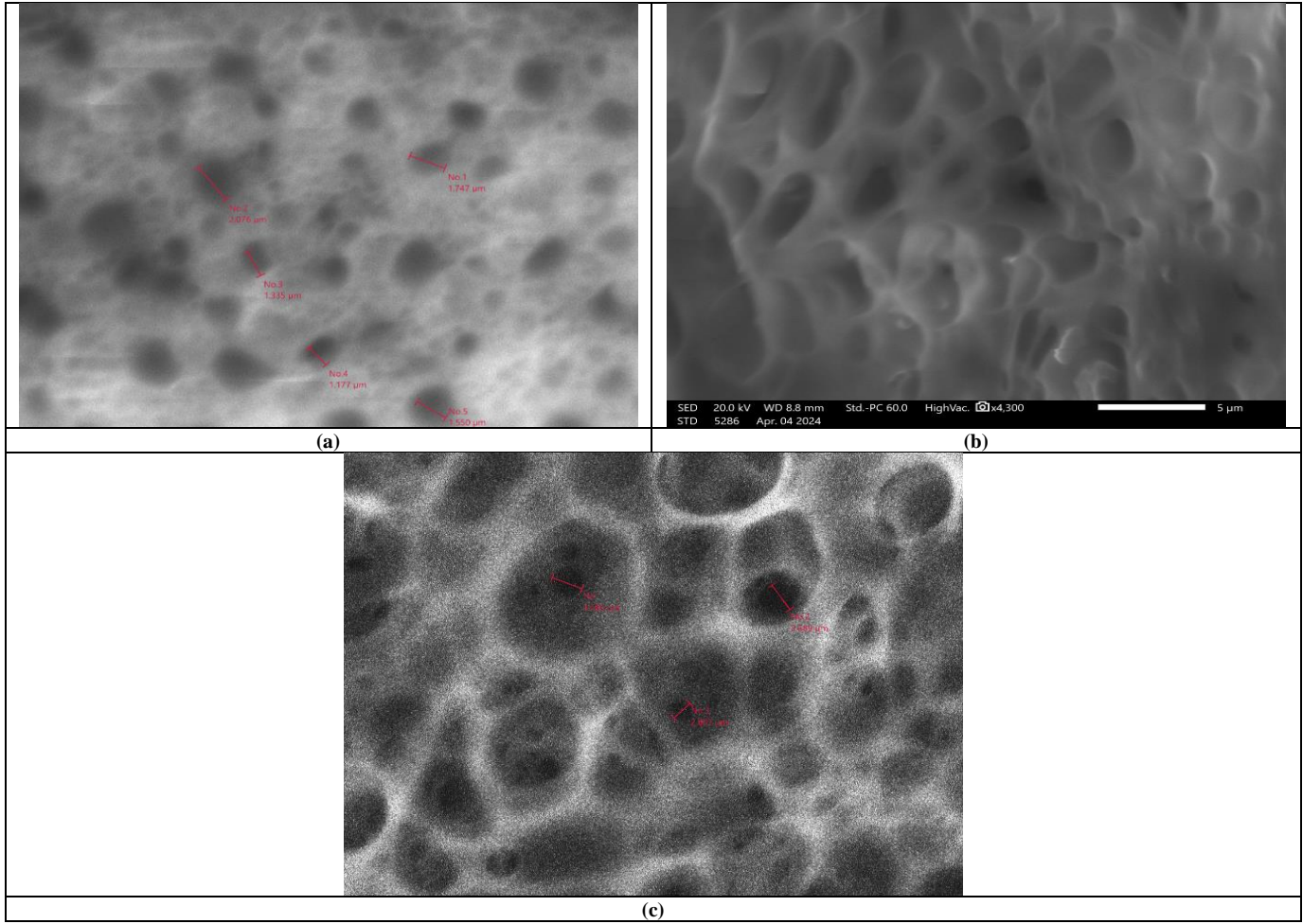
Fig. 10 SEM photographs of the prepared Nm4 membrane, a) SEM photo taken from the top of the membrane, b) SEM photo of a cross-section of the membrane, c) SEM photo of the underside of the membrane
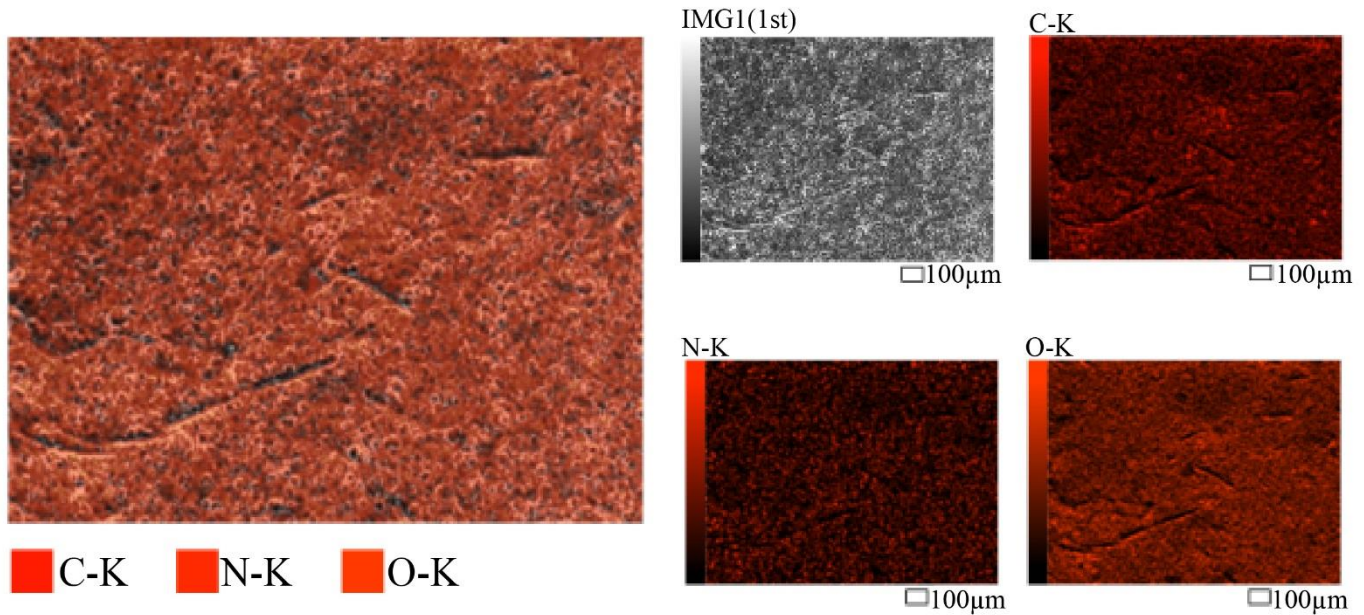

Fig. 11 SEM photos of the prepared Nm$_4$ membrane IMG1 (1st) 100-micrometer view of the general membrane:View of C-k carbon, N-k nitrogen, and O-oxygen mass
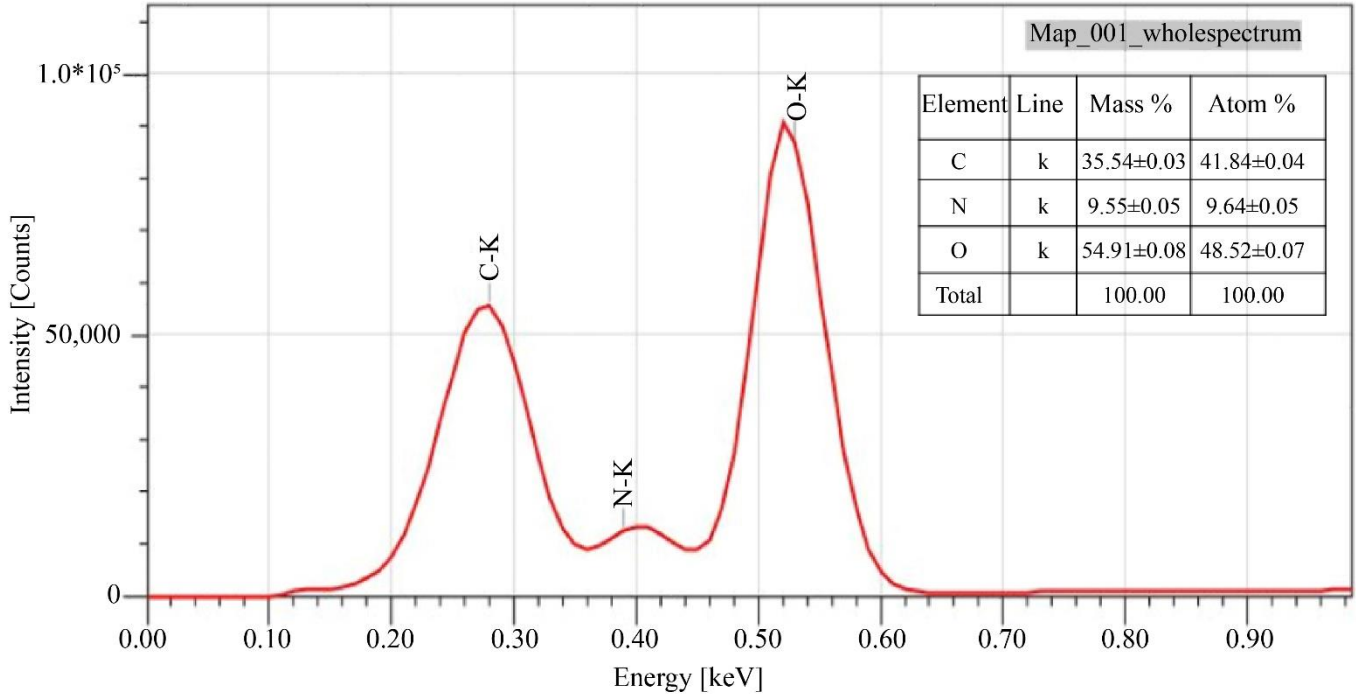
Fig. 12 Energy dispersive spectrum and compositional element ratios of the ion exchange membrane synthesized based on polyanilinenitrocellulose

Table 3. Elemental analysis of newly synthesized PANS membrane

No	Elements	Mass % view of elements
1	C	35.54
2	O	9.55
3	N	54.91
General		100

There may be some residues of additional solvents, but they are very small and do not affect the synthesis of the membrane and its properties. Proper selection, distribution of pore-forming substances in the membrane, and optimization of the environment show that it is possible to create standard porous ion exchange membranes.

4.3. Mechanical Test Analysis of PANS Membranes

The sample was cut to the required size and placed in the testing machine. An average of three samples was recorded for all prepared membranes. All membrane samples were tested three times, and the average result was obtained. For analysis, Nm_3 and Nm_4 membranes were taken and tested on a testing machine based on a force of 1-60 newtons. Initially, when the force acting on the sample Nm_3 increased to 41.7725 newtons, it showed an elongation property of up to 1.4%. Then, it was found that its interruption point occurred slowly at a force of 41.13004 newtons. In the Nm_4 sample, when the force acting on it increased to 53.8826 newtons, it showed the property of stretching up to 1.2%. It can be observed that its interruption point occurred slowly at a force of 52.9687 newtons. The results of the ratio of the force acting on the membrane to the elongation are shown in Figure 13.
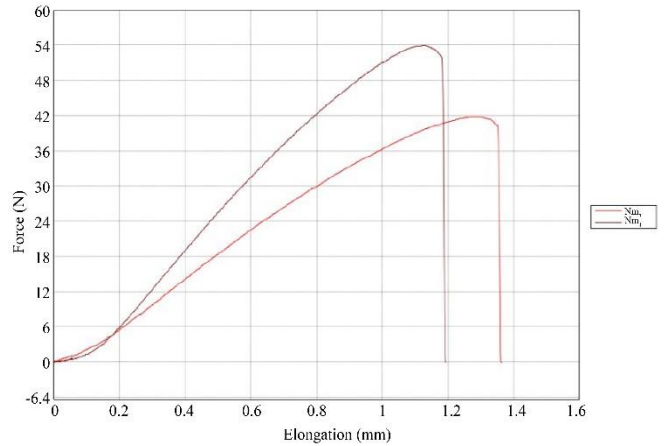

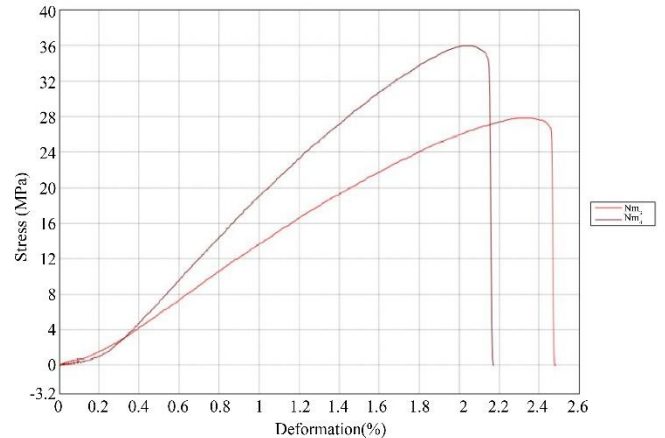**Fig. 13 The ratio of the force exerted on the membrane to the elongation**

Fig. 14 Ratio of speed to deformation

It can be observed in Figure 14 that sample Nm$_3$ is deformed up to 2.5% at a stress of 681.801 MPa, and sample Nm$_4$ is deformed up to 2.15% at a stress of 333.033 MPa. From the analysis given below, we can observe that sample Nm$_4$ is more resistant to external force than Nm$_3$. Our samples Nm$_3$ and Nm$_4$ also show the possibility of use in ion exchange membranes.

4.4. Thermogravimetric (TGA) and Differential Thermal (DTA) Analysis of PANS Membrane

Analysis of the thermogravimetric curve of the PANS ion exchange membrane shows that the TGA curve mainly occurs in the temperature range where 3 intense mass losses occur. The 1st mass loss range was 22.63 – 109.44 °C, the 2nd mass loss range was 109.44 – 226.0 °C, and the 3rd mass loss range was above 226 °C. The analysis shows that in the 1st mass loss interval, the mass loss decreased by 0.165 mg, i.e. 8.242%, due to the complete removal of moisture and pore formers from the membrane.

In the 2nd mass-loss interval, an intensive decay process took place. Due to the decomposition of the copolymer, the mass decreased by 1.809 mg, i.e. by 90.36%. In the 3-mass loss range, the mass loss was 0.117 mg, i.e. 5.844%. A charred residue was left at the end of the process. The obtained results are shown in Figure 15.
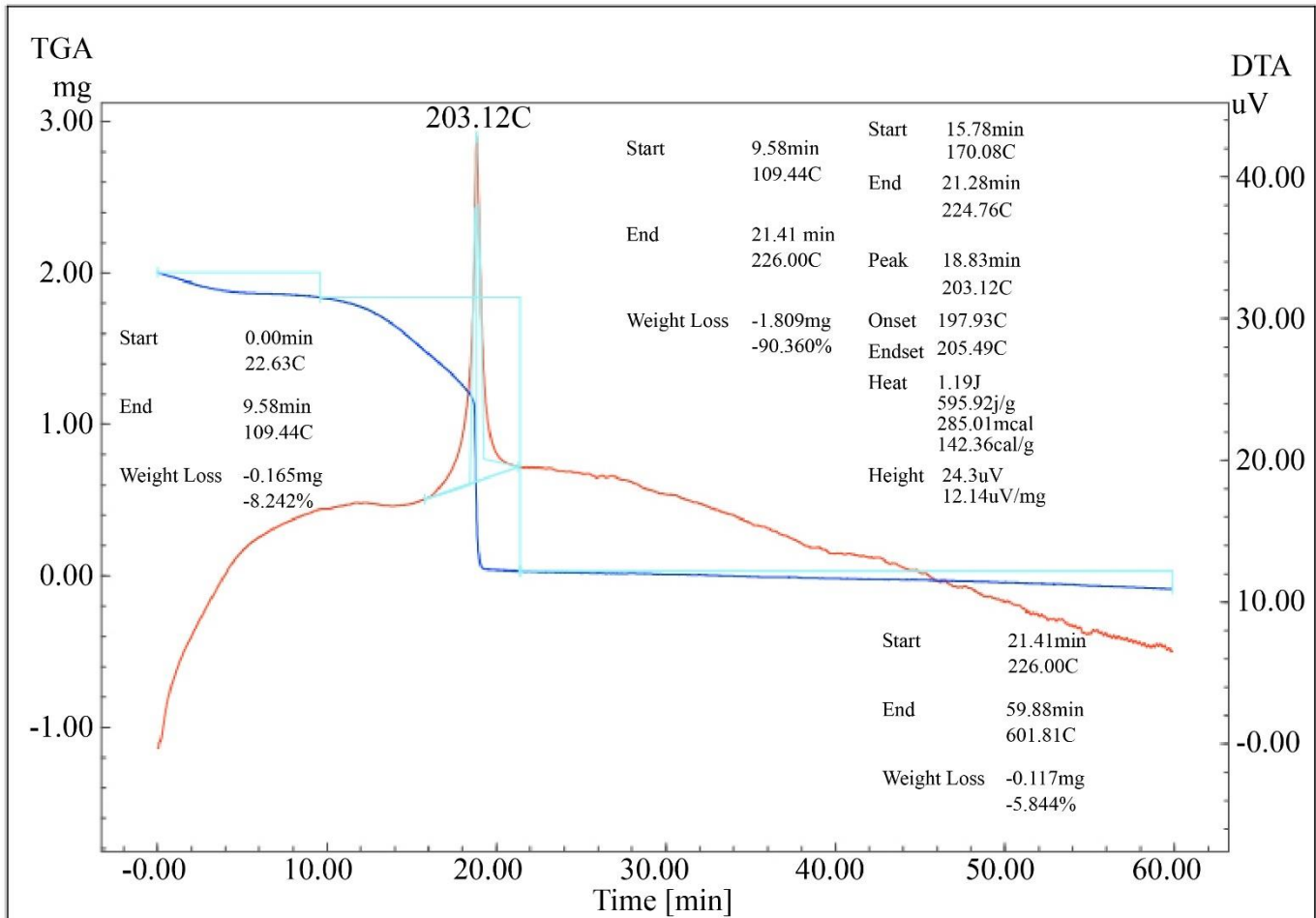

Fig. 15 Results of thermogravimetric (TGA) and differential thermal analysis (DTA) of PANS ion exchange membrane.

Table 4. Cast composition of PANS ion exchange membrane

No	T, °C	Lost mass, mg (2,002 mg)	Amount of energy consumed (µV*s/mg)	Time spent (min)	DW (mg)	DW/DT (mg/min)
1	100	0.154	16.1	8.61	1.848	0,0179
2	200	0.835	30.47	18.6	1.167	0,0449
3	300	1.97	17.92	28.9	0.032	0.068
4	400	1.982	13.79	39.1	0.02	0.051
5	500	1.983	10.22	49.4	0.019	0.040
6	600	1.991	6.72	59.7	0.011	0.033

The differential thermal analysis of the PANS ion exchange membrane found that initially, the exothermic effect occurred in the range of 26,63 – 226,0 °C. The analyses show that the peak of the intensive decay process occurred in the 2-mass-loss range. A detailed analysis of the thermogravimetric and differential thermal analysis curves is given in Table 4. As can be seen from the derivatogram of the membrane presented above, with the increase in temperature, the mass of the polymer gradually decreases, and exo-effects are formed. The decrease in the mass of the membrane with the increase in temperature occurs due to the loss of adsorbed water and the decomposition of substances contained in the membrane.

and nitrocellulose produced more than 70% yield in a 1:1.25 mass ratio process for 4.5 h from 0 °C to -4 °C. Then, the PANS ion exchange membrane was synthesized from it. The morphology, surface area, porosity, and element analysis of the synthesized PANS ion exchange membrane were studied in a scanning electron microscope (SEM). The bonding nature of atoms was determined by infrared spectroscopy (IR), Derivatograph determined thermal properties, and resistance to mechanical effects was determined using universal test probes. Due to the ease of obtaining and reforming the PANS membrane and the presence of an aniline compound, which gives high permeability, it gives good results in ion exchange membranes.

5. Conclusion

Polyanilinenitracellulose was synthesized based on aniline and nitrocellulose using the in situ polymerization method. The effect of temperature, concentration, and time on the reaction yield of PANS synthesis was studied. Aniline

Acknowledgments

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