PARAMETERS OF THE DYNAMIC MEMBRANE REGENERATION PROCESS

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Abstract: The main components in the composition of the oil-water emulsion are hydrocarbons, an increase in the intensity of the absorption bands at 690, 2856, 2930, 3020-3100 cm^{-1} corresponds to vibrations of the C-H bond, at 1372, 1410, 1460 cm^{-1} to vibrations of $-CH_3$. To restore the initial performance of the membranes, the membranes were washed by washing with a 5% sodium dodecyl sulfate solution was restored by 99%. This circumstance is due to the poor wettability of the surface of the dynamic membrane compared to the original membrane of nylon, due to the hydrophobicity of polystyrene, oil products remain on the surface of the dynamic layer and are easily removed when washing the membranes.

Keywords: FT-IR spectroscopy, scanning electron microscopy, nylon, polystyrene, an oil-in-water emulsion, concentration polarization, membrane regeneration, sodium dodecyl.

1 Introduction

In the process of membrane separation of water-oil emulsions, in particular, microfiltration and ultrafiltration, a concentration polarization phenomenon are observed with an increase in the concentration of oil products in the surface layer of the membrane (Fazullin & Mavrin, 2017: Fazullin et al, 2016: Fazullin et al, 2017: Fazullin et al, 2018: Lee, 1984: Mohammadi et al, 2003). The consequences of this phenomenon are manifested by a decrease in the specific productivity of the membranes as a result of the formation of a gel layer on the surface and clogging of the pores of the membranes. Membrane regeneration is carried out by backwashing, washing membranes with surfactant solutions, mechanical removal of contaminants.

The phenomenon of concentration polarization during ultrafiltration of a water-oil emulsion was studied in (Sablani et al, 2001). According to the results of the study, the authors found that the oil content in the gel layer is approximately 40 vol. %. It was also established that the membrane is contaminated due to oil adsorption on the membrane structure, as a result of which the critical surface tension and wettability of the membrane increase and the effective pore diameter also decreases, which leads to a decrease in membrane permeability. The membrane was regenerated by micellar solutions of sodium dodecyl sulfate -n-pentanol-water, which turned out to be sufficiently effective and not expensive.

To prevent the phenomenon of concentration polarization, a turbulent flow is created, rotational mechanisms are installed in front of the membrane surface, the structure of the membrane elements (roll, tubular membranes) is changed, preliminary suspension of suspended substances and oils is carried out. To increase the resistance of membranes to polarization phenomena, chemical and physical modification of the membranes is carried out.

In (Lobo et al, 2006), the effect of the hydrogen exponent and flow rate on the phenomenon of concentration polarization was investigated. The experiments were carried out using tubular ceramic membranes with two pore sections (50 and 300 kDa) in the operating pressure range of 0.05-0.4 MPa. The polarization of the concentration was observed at low transverse flow rates. The authors found that the pH of the emulsion does not affect the particle size of the dispersed phase. But the membrane permeability and chemical oxygen demand (COD) sharply decreased at low pH values, since the membranes acquired a positive charge and anionic surfactants were adsorbed on the membrane surface, which made it more hydrophobic and caused a decrease in membrane performance. The separation efficiency of the emulsion in terms of COD was 92%.

Of all types of membranes, dynamic membranes have the advantage of separating water-oil emulsions. So while reducing the permeability of the membranes, you can replace the dynamic layer with a new one, which will restore the original performance. Using different particle sizes of the applied material or by obtaining several dynamic layers, the required pore size of the membranes can be achieved. Depending on the physicochemical properties of the applied material, various wettability and surface roughness of the membranes can be obtained.

In (Sung Yong et al, 2015), a regenerable antifouling membrane was obtained by applying a dynamic layer of polyethylene glycol (PEG) to the surface of a polytetrafluoroethylene membrane (PTFE) membrane. And the authors of (Chanchan et al, 2010). obtained a non-fouling membrane from a nonwoven material (NWF) modified by coating with chitosan. The modification improved specific productivity and made membrane regeneration more efficient.

For the regeneration of ceramic membranes, a solution consisting of NaOH and sodium dodecyl benzenesulfonate (SDBS) was used in (Pengli et al, 2015: Bruno et al, 2016). An important role during cleaning is played by hydrolysis of NaOH and solubilization of SDBS micelles, which reduce the adhesion between contaminants and the surface of the membrane. The membrane cleaning efficiency was dependent on the concentration of SDBS (C-SDBS) and the temperature of the solution, as well as on the working pressure, cross-flow rate and filtration time. The optimal parameters of membrane regeneration were established: surfactant content 0.30% by mass, pressure 0.10 MPa, flow rate 3 m / s, solution temperature 60 ° C and membrane washing time 80 min.

The main problem in the membrane separation of water-in-oil emulsions is a decrease in specific productivity due to the formation of a gel layer on the membrane surface as a result of the phenomenon of concentration polarization. Therefore, the work aims to study the process of regeneration of a dynamic membrane with washing solutions.

2 Methods

A microfiltration polymer membrane made of nylon with an average pore size of 0.45 μm and a diameter of 47 mm was used as the initial substrate, on the surface of which a dynamic layer was applied. A dynamic layer was obtained by forming on the surface of the porous base a semipermeable layer of suspended polystyrene microparticles present in the filtered aqueous solution of acetone in dynamic equilibrium with the solution. The polystyrene content in the membrane was determined by the gravimetric method, by the weight of the membrane before and after modification.

The IR spectra of the samples were studied in an «InfraLUM FT-02» brand IR-Fourier spectrometer. IR-Fourier spectrometer allows to obtain high-resolution absorption bands.

The change in the surface structure of the membranes was recorded using a scanning electron microscope brand "LEO-1430 VP" manufacturer Carl Zeiss. Samples were glued onto aluminum plates, the surface of the membranes was sprayed with gold, by cathodic deposition in argon, and viewed in high vacuum.

As the main indicators of the membrane separation of the emulsion, we considered the specific productivity, which was determined as the ratio of the amount of filtrate formed to the product of the membrane area and the process time in terms of $dm^3/ m^2 \cdot$ h, and the degree of separation of the emulsion, which was calculated as the ratio of the content petroleum products (NP), determined using a KN-3 concentrator, emulsions before and after separation.

For membrane separation, 1% emulsion of freshly prepared coolant with an oil concentration of more than 10 g / dm^3 was used as water-oil emulsion. During the separation of distilled water and emulsions, the working pressure of 0.1 MPa was applied, and the temperature of the liquid was 25 ° C.

With a decrease in the specific productivity of the membrane by 2 times, to restore the initial productivity, they were washed with 5% sodium dodecyl sulfate solution, followed by washing with distilled water. Membrane washing conditions: pressure 0.10 MPa, solution temperature 40 ° C, membrane washing time 10 min.

3 Results And Discussion

After applying a dynamic layer of polystyrene to the surface of the original nylon membrane, the surface of the membrane becomes hydrophobic, which is determined by the increase in the contact angle of the membrane with a drop of distilled water from 59.6 ° to 106.2 °.

The results of the study of the membrane surface by scanning electron microscopy with a magnification of 2000 times are presented in Figure 1.

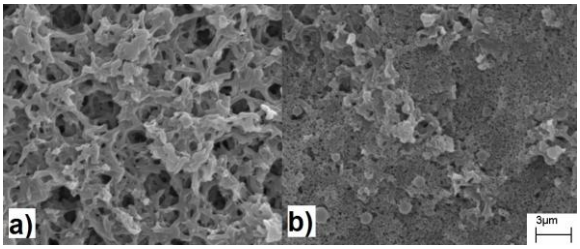

Figure 1. Microphotographs of the membrane surface: a) Initial membrane of nylon; b) the membrane after the application of a dynamic layer of polystyrene (Magnification 2000 times).

According to Figure 1, it can be seen that polystyrene particles are located on the surface and in the pores of the nylon membrane.

Using an «InfraLUM FT-08» brand Fourier transform infrared spectrometer, spectra of the studied membrane samples were obtained in the frequency range 600-4000 cm^{-1}. Figure 2 shows the IR spectra of the nylon-PS membrane before and after the separation of the oil-water emulsion.

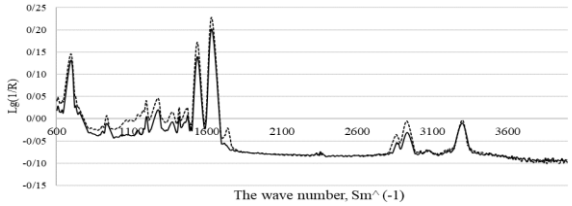

Figure 2. IR absorption spectra of the investigated membrane samples: nylon-PS (solid line) and nylon-PS after separation of the water-oil emulsion (dotted line).

After the separation of the oil-water emulsion, an oily stain remains on the surface of the membrane. According to the IR spectrum of a nylon-PS membrane after separation of a water-oil emulsion (Fig. 2.) there is an increase in the absorption intensity for all bands. The main components in the composition of the oil-water emulsion are hydrocarbons, an increase in the intensity of the absorption bands at 690, 2856, 2930, 3020-3100 cm^{-1} corresponds to vibrations of the CH bond, at 1372, 1410, 1460 cm^{-1} to vibrations of $-CH_3$. The new absorption band at 1740 cm^{-1} corresponds to the vibrations of the limiting aliphatic aldehydes $-CH_2$-CHO.

The results of studies on the specific productivity of the membranes are presented in the table 1.

Table 1. Specific productivity of distilled water

Membrane	The content of polystyrene,% (by weight)	The specific capacity of membranes, $dm^3/ m^2 \cdot h$	
		initial	after separation of the emulsion
Nylon	-	3845	2157
Nylon-PS	3.4	266	104

After applying a layer of polystyrene to the surface of the membrane, the specific productivity of the membranes decreases by an order of magnitude due to the deposition of polystyrene particles in the pores and surface of the membrane. The obtained dynamic nylon-PS membranes in terms of specific productivity are not inferior to the UPM-100 polysulfonamide membranes (1.2 - 4.2 $dm^3/ m^2 \cdot h$), to the UAM-150 cellulose acetate membranes (5.4 $dm^3/ m^2 \cdot h$). After the separation of the oil-water emulsion with a concentration of oil 10 g / dm^3, the permeability of the dynamic membrane decreases by 2.6 times.

To restore the initial performance of the membranes, the membranes were washed by washing with a 5% sodium dodecyl sulfate solution and then washing with distilled water.

Table 2. Specific productivity of membranes after regeneration

Membrane	The content of polystyrene,% (by weight)	The specific capacity of membranes, $dm^3/ m^2 \cdot h$	
		initial	after regeneration
Nylon	-	3845	3249
Nylon-PS	3.4	266	263

Figures 3 and 4 show photographs of the initial and dynamic membranes after separation of water-oil emulsion and after washing with a washing solution.

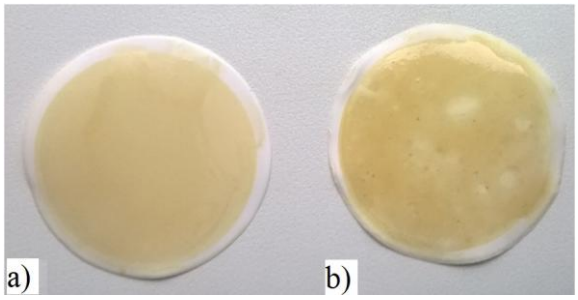

Figure 3. Photographs of membranes after separation of water-oil emulsion: a) initial from nylon; b) dynamic "nylon-PS".

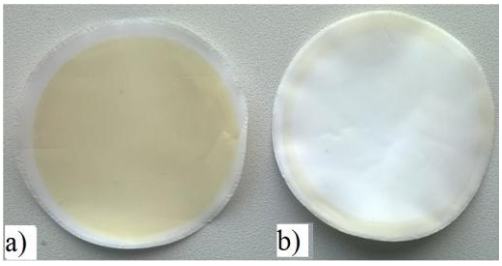

Figure 4. Photographs of membranes after regeneration with a washing solution: a) initial from nylon; b) dynamic "nylon-PS".

Membrane regeneration after separation of water-oil emulsion was carried out by washing with a washing solution. Figures 3 and 4 show photographs of the initial and dynamic membranes after separation of water-oil emulsion and after washing with a washing solution. An oily stain remains on the original membrane after washing (Sedysheva, 2011).

It was determined that, on average, the specific productivity of the initial membrane recovered from the initial parameters by 84 %, and the specific productivity of the dynamic nylon-PS membrane was restored by 99 %. (table 2). This circumstance is due to the poor wettability of the surface of the dynamic membrane compared to the original membrane of nylon, due to the hydrophobicity of polystyrene, oil products remain on the surface of the dynamic layer and are easily removed when washing the membranes.

4 Summary

After separation of the water-oil emulsion, an oily stain remains on the membrane surface, the permeability of the dynamic membrane decreases by 2.6 times. According to the IR spectrum of the Nylon-PS membrane, after separation of the oil-water emulsion, an increase in the absorption intensity for all bands is observed. The main components in the composition of the oil-water emulsion are hydrocarbons, an increase in the intensity of the absorption bands at 690, 2856, 2930, 3020-3100 cm^{-1} corresponds to vibrations of the CH bond, at 1372, 1410, 1460 cm^{-1} to vibrations of $-CH_3$. To restore the initial performance of the membranes, the membranes were washed by washing with a 5% sodium dodecyl sulfate solution and then washing with distilled water. It was determined that, on average, the specific productivity of the initial membrane recovered from the initial parameters by 84%, and the specific productivity of the dynamic nylon-PS membrane was restored by 99%. This circumstance is due to the poor wettability of the surface of the dynamic membrane compared to the original membrane of nylon, due to the hydrophobicity of polystyrene, oil products remain on the surface of the dynamic layer and are easily removed when washing the membranes.

5 Conclusions

As a result of the separation of the oil-water emulsion by the dynamic nylon-PS membrane, a decrease in specific productivity is observed up to 2.6 times. To restore the initial permeability of dynamic membranes, regeneration is carried out by reverse or direct washing with hot water, steam and surfactant solutions with the addition of alkali. To restore the initial performance of the membranes, the membranes were washed by washing with a 5% sodium dodecyl sulfate solution and then washing with distilled water. It was determined that, on average, the specific productivity of the initial membrane recovered from the initial parameters by 84%, and the specific productivity of the dynamic nylon-PS membrane was restored by 99%. Thus, the dynamic polystyrene layer prevents the adsorption of oil in the pores of the membrane, forming an oil layer on the surface, which is easily removed after washing the membrane with a washing solution.

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