Applications of Polymeric Membranes Ultrafiltration Process on the Retention of Bentonite Suspension

LAURENTIU TATARU¹, VALENTIN NEDEFF¹.², NARCIS BARSAN¹*, ANDREI VICTOR SANDU³.⁴, EMILIAN MOSNEGUTU¹, MIRELA PANAINTE-LEHADUS¹, ION SANDU⁴.⁵*

¹Vasile Alecsandri University of Bacau, Department of Environmental Engineering and Mechanical Engineering, 157 Calea Marasesti Str., 600115 Bacau, Romania,

²Gheorghe Ionescu Sisesti Academy of Agricultural and Forestry Sciences, 61 Marasti Blvd., 011464 Bucharest, Romania ³Gheorghe Asachi Technical University of Iasi, Faculty of Material Science and Engineering, 61A D. Mangeron Blvd., 700050 Iasi, Romania

⁴Romanian Inventors Forum, 3 Sf. Petru Movila St., Bloc L11, III/3, 700089 Iasi, Romania

⁵Alexandru Ioan Cuza University of Iasi, Arheoinvest Interdisciplinary Platform, Scientific Investigation Laboratory, 11 Carol I Blvd., 700506 Iasi, Romania

In the field of purification and treatment of waste water resulting from various domestic and industrial activities, classical methods of retention of pollutants almost can no longer eliminate their large quantities, and this signifies a problem related to the environment. Filtration membrane technology has a larger footprint in the removal of these pollutants, and their success is due to virtually the quality of the resulting water. This paper highlights the development of a wastewater ultrafiltration process, containing bentonite, an inorganic compound found in wastewater from industries such as the steel industry, food industry, and so on. This study aims to find the relation between all parameters present in the ultrafiltration process, respectively how these parameters can influence each other. The study is necessary because bentonite, from a common substance, can be a dangerous pollutant, especially if it comes into contact with other compounds that in chemical reactions can harm the environment, and this raises questions to researchers who are experimenting with water purification technologies at a state-of-the-art level. At the same time, the study aims at determining the percentage of bentonite retaining on the membrane filtration surface, and in the final analysis of how bentonite can block the pores of the membrane or deposit on its surface. Throughout the entire ultrafiltration experiment it will be monitorized parameters related to organic membrane with hollow fibers and the results of physico-chemical indicators obtained at the final of the process. The results of the study showed that bentonite can be retained more than 20%. Accumulation of bentonite on the surface of the membrane decreased slightly the volume of permeate at the end of the experiment, resulting in an insignificant decrease in the volume of the liquid in the membrane. The results on bentonite retention efficiency and dependence parameters in the ultrafiltration process will be detailed in the present paper.

Keywords: bentonite, retention efficiency, membrane, ultrafiltration, pilot station

The environment *water* factor is still being discussed as the problem of environmental pollution is increasingly concentrated on deteriorating the quality of water sources. Wastewater filtration can be a quite convenient solution for removing pollutants present in wastewater from different industries as well as using coagulant and flocculants solutions to improve the quality of the filtered water [1-3].

Applying filtration materials with different granulations to efficient effluent filtration has proven to be effective because not only is the ultimate high water quality but is significantly reduced and the cost of acquisition, handling and exploitation of granular materials does not necessarily require qualification high [2, 4-6].

Over time various mathematical models have emerged that highlight the most effective solutions used in wastewater treatment as well as the use of powders and materials of different granulations [2, 4-9].

In recent years, the world's population has increasingly met the problem of water scarcity needed for various activities, especially where water has to be of superior quality such as medicine, industries requiring fairly high purity water, and industry food, problem which was improved as a result of the use of coagulation, flocculation, chlorine disinfection, etc. [10, 11].

Modern studies are increasingly focusing on membrane filtration technology due to multiple advantages both in terms of structural material and cost, not harmful to the environment, and the process of ultrafiltration of water does not take place on several phases [11-14].

The inner membrane construction allows membranes to pass through the membrane pores only with particles smaller than 0.02 μ m, rejecting bacteria, fats, colizons of larger size [15], and desirable that the membrane can retain organic or inorganic matter of the water as much as possible, to have high physical, mechanical and chemical resistance and to allow the flow of as much water as possible through the membrane [16].

For organic fiber membranes, a major disadvantage is fiber breakage during the ultrafiltration process [17, 18], but this can be at least delayed by fastening and gripping the ends of the fibers in such a way that they are not allowed to move or vibration when the flow of fluid flows through them [17, 19, 20].

Over time, to solve the problem of both: membrane fouling and providing a high membrane resistance in

^{*} email:narcis.barsan@ub.ro; sandu_i03@yahoo.com

ultrafiltration processes, studies have been carried out to find optimal optimization solutions, ultimately the ultrafiltration process to ensure not only the quality of the final product but also lower costs and the big disadvantages such as fouling and breakage resistance be improved by providing the membrane with a long lifecycle [21-23].

In the paper on the use of coagulants and flocculants used for the purification of wastewater, Barsan et al. [10] have presented some advantages that support classical processes but the extent and degree of danger of current pollutants are increasingly pronounced. However, even membrane technology faces some problems such as the short life of some membranes, and this involves cost increases, especially where membrane scratching occurs, as Gaurav et al.

But the cost improvement in this context can be pronounced if the adsorption method is used, respectively the use of adsorbents implies a delay in the fouling phenomenon, since these adsorbents have a great ability to clean the deposited material on the surface of the membrane [25].

Until now, ultrafiltration membranes have demonstrated high performance in retaining many organic and inorganic compounds, including bentonite [26-28]. Researchers like Li and Fane [29] have demonstrated that the ultrafiltration process can be quite effective in bentonite retention, but to achieve increased performance, they have used the transversal vibration method that aims to vibrate the membrane fibers, which leads to *disturbing* the material deposited on the walls of the membrane pores [29].

Due to the fact that bentonite has small particle sizes around 6 μ m, it can easily disrupt the good functioning of the ultrafiltration process, but besides the membrane fiber vibration method, Buetehorn et al. have presented in their paper a method of aerating the fibers, whereby the pressure of the air induced to the surface and inside the pores leads to the large displacement of deposited bentonite particles [30].

The evolution of the increase or decrease of the flow of liquid entering the membrane, the influence of the concentrated and permeated pressure, the transmembrane pressure as well as the dependence of the operating parameters in the ultrafiltration process with the hollow fiber organic membrane will be discussed in detail in this paper in the following chapters.

The purpose of this study is to highlight the time when the volume of permeate is decreasing as a result of the membrane fouling. It is also necessary to know the fluctuations of the parameters such as temperature, pressure and flow entering the membrane as a result of the decrease of the permeate when it is decline.

Experimental part

Materials and methods

The installation that serves the ultrafiltration process, containing an organic fiber membrane module is shown in figure 1.

According to the user guide, the ultrafiltration plant is composed of the PAN organic membrane module, placed in the center of it, three tanks: one bottom right feed, one permeate collector located down the control panel and the other for recirculation concentrate, located parallel to the permeate. On the other hand, the other components are the two flowmeters, one that records the permeate flow, located on the left side of the membrane module, at the outlet of the permeate and the other immediately after the discharge pump, before the liquid flow penetrates the membrane during process [31].

The pressure sensors are located both at the output of the module and at the outlet of the concentrate and another is needed where the backwash is required to wash the membrane.

Materials used in the preparation of the feed solution

Bentonite was purchased from BENTAFLUX S.A. Satu Mare, Romania. The membrane module together with the MP 90 pilot plant was purchased from DELTA LAB, France. The material from which organic fibers are made is polyacrylonitrile (PAN), with a molecular weight of 13,000 Da. Both the inner and outer diameters of a fiber are 0.8 or 1.4 mm, and the membrane pore size is 0.3 µm, according to the pilot station manual [31]. The length of the membrane module is 552 mm, which can ensure the penetration of a large flow of liquid through the membrane, as the maximum flow that can flow through the plant is 3000 L/h when no chemical solutions are introduced into the supply water.

Preparation of the bentonite solution

Bentonite, having particle sizes around $6.15\mu m$, was mixed with tap water and then stirred for 35 min at 300 rpm to give an emulsion solution, then added in the feed tank into which 30 L of water were introduced, and a bentonite concentration of 5 g/L, respectively a concentration of bentonite of 0.5 % was established.

Throughout the experiment, deionized water was used, especially for cleaning the appliances that were used for analyzes of collected samples. Prior to the start of the

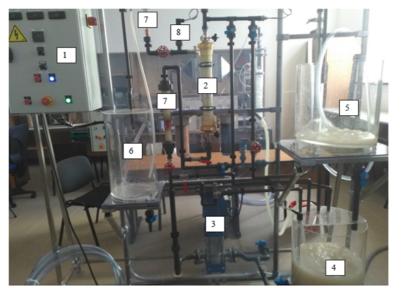


Fig. 1. Experimental installation MP 90 ultrafiltration: 1 - Control panel; 2 - Membrane module; 3 - Peristaltic pump; 4 - Feed tank; 5 - Concentrate tank; 6 - Permeate tank; 7 - Flowmeters; 8 - Pressure sensor

experiment, the solution obtained was recirculated through the installation without penetrating it into the membrane to completely homogenize it.

Description of the operating procedure during the experiment

The experiment was carried out within 5 h, without interruption, with collecting the permeate samples after every 30 min, including taking a sample from the feed tank both before and after the experiment started, to determine the degree of the efficiency of retaining bentonite from the solution proposed for study.

The experiment time is 5 h because it is believed that after this interval, the working membrane surface become agglomerates with the matter particles, namely bentonite, and this causes a significant decrease of the permeate.

The 30 min sampling interval respectively data parameters were chosen to see the process parameters fluctuations in time. In other words, the faster data collection means that the better the fluctuations in the system are observed.

Temperature and pressure recording throughout the system were done automatically, only reading the values on the control panel. The flowmeters recorded the flow of liquid entering the modules, ie the flow of permeate during the whole experiment.

The experiment was monitored step by step in order to avoid unpleasant situations that could hinder the smooth running of the process. Parameters such as transmembrane pressure, flow rates, temperature, retention rate, volume concentration factor, membrane flux density, etc., have been carefully monitored because depending on these, it was possible to establish the dependency relationship that led to the determination of the ultrafiltration membrane efficiency.

The samples that were taken were analyzed using laboratory equipment and the results obtained from the analyzes were further processed in order to show the relationship of dependence between all the parameters of both the installation and those of the ultrafiltration membrane

During the experiment, the freshly collected samples were subjected to the analysis of the main indicators, namely *p*H, dissolved oxygen, conductivity, turbidity using WTW equipment, from the research laboratory. The determination of the indicators was necessary because the relationship between the indicators and the parameters of the installation influences the efficiency of the membrane.

Results and discussions

Figure 2 describes temperature and pressure variation throughout the ultrafiltration process using a PAN organic hollow fiber membrane for 5 h of continuous operation.

The evolution of the temperature in the process is increasing (fig. 2), as the liquid that circulated through the pipelines of the installation always had the tendency to heat as a result of the membrane continuity.

Initially, the process started at 18.4°C, finally reaching 22.5°C. The highest fluctuations were recorded during the first hour, the temperature rising by 2 degrees in just 60 min. This can be explained by the fact that the membrane is most demanded in the early hours because, over time, the material deposited on the membrane sets its position so as to allow the liquid to pass through the membrane pores in a continuous succession.

In other words, it is more difficult for the matter to locate in the walls of the pores at first, then the waste water more easily establishes the trajectory, the flow direction.

Regarding the system pressures, the pressure of the liquid in the membrane, the pressure of the concentrate and the permeate, they did not change because the calculated transmembrane pressure was not too high and the concentration of the bentonite in the supply tank was not high which forced a fluid flow *relaxed*.

In this case, after the interpretation of figure 2, it can be said that the values of the pressure at the concentrate and the permeate recorded a linear continuity.

Transmembrane pressure was calculated with relation [31]:

$$PTM = [(P_{i} + P_{c})/2] - P_{n}, (1)$$

where: PTM represents transmembrane pressure, expressed in bars; Pi - Pressure from fluid inlet to membrane module [bar]; Pc - Pressure of the concentrate [bar]; Pp - Permeate pressure or filtered water pressure [bar].

The transmembrane pressure recorded throughout the ultrafiltration process was 0.8 bar.

It was expected that the temperature would increase much more if pressure varied over time, causing rapid fluid heat, but this situation could only occur if it operated at a transmembrane pressure higher than 1-1.5 bar.

As can be seen in figure 3, the volume of permeate obtained over time has a decreasing trend due to the accumulation of bentonite particles on the membrane.

It was expected that the volume of permeate obtained would be recorded downward during the experiment. A small change was also recorded for the fluid inlet flow. Thus, almost at the end, more precisely, after minutes 270, the input flow started to drop by about 2-3 L/h, and this

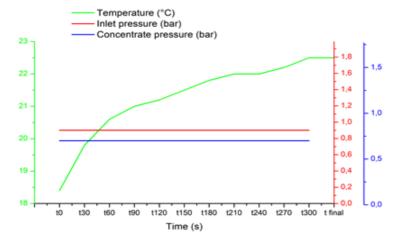


Fig. 2. Evolution of pressure and temperature of liquid flow over time

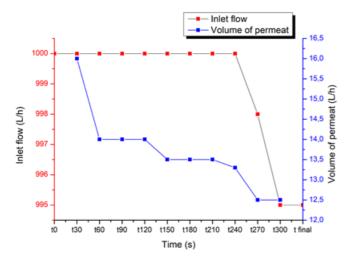


Fig. 3. Variation of permeate flow rate depending on the flow of the incoming liquid

automatically means also the modification of the final product.

Decreasing the flow of the incoming liquid starts to increase after about 4 h because this flow is closely related to the volume of the permeate. This decrease can be explained by the fact that fouling of the membrane causes a decrease in efficiency, ultimately not only changing the volume of permeates, but also its quality.

However, it is normal for there to be a decline in the flow of permeate for two reasons: firstly lowering the inlet flow, the volume of permeate can not remain the same, then, over time, the matter accumulated on the membrane, respectively bentonite can not allow it to pass the same flow of water through the membrane pores.

Finally, the membrane fouling process puts its mark on the productive side and this major disadvantage made it possible to modify all the parameters of both the membrane and the pilot station.

Flow density was calculated using the relationship [31]: J = Q/S

where: J represents the density of the flow of liquid passing through the membrane in the unit of time, relative to the surface of the membrane, expressed in m³/s·m², Q permeate flow rate obtained for each sample collected at 30 min, expressed in L/h, S - Effective membrane filtration area, expressed in m².

The calculated flow density was 0.00528 L/s·m², respectively, during one second, the surface of the 0.6 m² membrane circulated ≈ 0.003168 L of liquid, respectively

A very important role in determining the bentonite retention efficiency of the ultrafiltration process had turbidity (fig. 4).

Turbidity depends on some temperature measure, in this case the influence of temperature did not have a decisive role in the change of the concentration of bentonite in the analyzed samples (fig. 4).

However, the electrical conductivity measured on the basis of the samples collected during the experiment was decisive. It remained somewhat constant, showing small fluctuations, increasing slightly over time.

As the permeate volume decreased, the fluctuations in conductivity continued to be somewhat constant. This means that the fouling of membrane is more pronounced but the quality of the permeate continues to remain the same. Conductivity will influence your retention rate.

Determining the efficiency of bentonite retention by the organic membrane will be done through the relationship [31]:

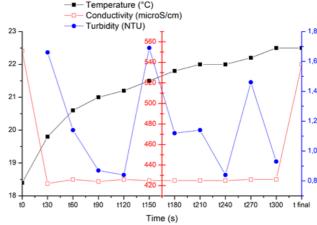


Fig. 4. Determination of conductivity and turbidity depending on temperature

$$TR_{i} = 100 \cdot (1 - CP_{i}/CR_{i}) \tag{3}$$

where: TR, represents the retention rate, expressed in %; CP_i - concentration of substance in the permeate sample [%]; CR, - concentration of substance in concentrate (feed) [%];

The higher the retention rate, the more effective the membrane is because the retention of the substance is basically the superior quality of the final product.

The volume concentration factor was calculated using the relationship [31]:

$$FCV = V_i/(V_i - V_p), \tag{4}$$

where: FCV is the volume concentration factor, expressed

in L/h; V_i - initiale volume; V_p - permeate volume.

In the present study, the determined volume concentration factor was 1.016.

In figure 5, pH values have continuously fluctuated insignificantly since bentonite is not strongly acidic or alkaline, and this has resulted in the recording of pH values ranging from 8-8.4.

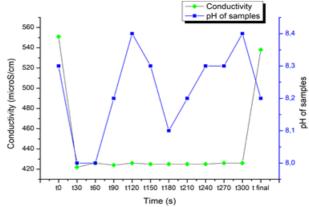


Fig. 5. pH variation according to conductivity values

The fact that the pH did not change too much is also due to the stability of the conductivity in each permeate sample during the ultrafiltration process (fig. 5), which means that the membrane retains bentonite constantly but slightly ascending.

Figure 6 clearly shows a decrease in permeate but at the same time turbidity continues to fluctuate both ascending and descending as the collected permeate samples are not influenced by the change in the amount of decreasing permeate volume.

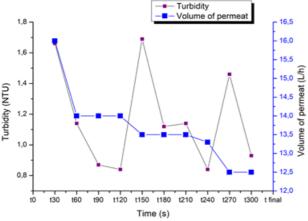


Fig. 6. Turbidity related to the volume of permeate obtained

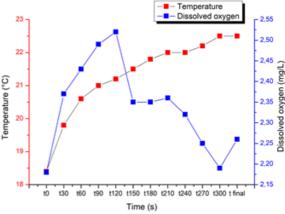


Fig. 7. Changes in dissolved oxygen concentration relative to temperature

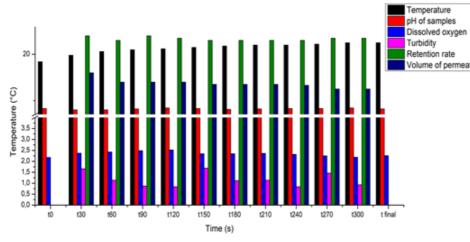


Fig. 8. The reciprocal influence of the chemical indicators between the measured parameters

The dissolved oxygen in each sample shows that the temperature has an influence on the quality of the resulting final product, respectively the permeate.

Figure 7 shows that the dissolved oxygen present in each permeate sample has a slightly decreasing tendency, since after minute 120, from 2.52 mg/L, it tends to drop to 2.19 mg/L. This can be explained by the fact that with the passage of time, the membrane clogging and in the permeate samples the concentration of bentonite increases slightly. If this happens then the dissolved oxygen is decreasing. All of these things lead to lower membrane ultrafiltration performance.

In order to have a clear overview of the membrane parameters, depending on the main chemical indicators analyzed, the variations of each of these parameters and indicators are presented in figure 8.

As the first observation on figure 8, it can be noticed that there are no very large fluctuations in the system, which means that the ultrafiltration process ranged within normal limits, the quality of analyzes and the monitoring of the process itself.

On the other hand, the relationship between the parameters and the chemical indicators can be clearly seen in the same figure as fluctuations in the whole system have the same tendencies, both increasing and decreasing.

It is normal for the retention rate to decrease as much as the conductivity increases or remains somewhat constant, and the volume of permeate is obviously decreasing.

However, in some cases, the permeate quality may remain somewhat the same despite the decrease in permeate volume. This may be mainly due to the type of chemical used as well as its concentration. For the feed sample before and after the experiment was completed, pH, temperature, conductivity, and turbidity values were over 1100 NTU, as this is not highlighted in the graph.

 T_0 is the sample collected from the feed tank and this does not involve calculating the retention rate because this sample is not the permeate sample.

The same can be said about the $T_{\mbox{\tiny final}}$ because this sample is also harvested from the feed tank and is the standard sample rate retention test.

Conclusions

In this paper we highlighted how the MP 90 pilot station parameters interact with the operating conditions of the entire ultrafiltration process. As seen in the previous subchapters, the temperature has played some of the most important role because, depending on this, the entire system has functioned as a result of the temperature evolution.

At the same time, the fact that pressures did not change in any way did not show significant changes and the rest of the parameters worked at low fluctuations throughout the process.

It is clear that the flow of permeate drops from the first hours of work because at the concentration of bentonite of 5 g/L, it has been easily deposited in both the membrane pores, locating in their walls, but also on the effective working surface of the membrane, ultimately favoring the fouling process.

This paper consisted of an experiment that had the role of retaining bentonite by a hollow fiber organic membrane during 5 h of continuous operation. Under normal operating conditions, the membrane retained bentonite in a

satisfactory percentage, most particles passing easily through the pores because of the much smaller dimensions.

The results obtained following the bentonite wastewater ultrafiltration process through a hollow fiber organic membrane have shown that regardless of the nature of the chemical used in the process, the advantages of the membrane cannot be limited. In other words, the high mechanical strength of the membrane fibers proves to be a great advantage, regardless of the waste water flow that entered the membrane module in the time unit.

On the other hand, the membrane worked in good conditions irrespective of the change in the temperature range, regardless of the pH etc. and this did not cause a very high decrease in the flow of permeate over time, as this decrease was about 4 L during the 5 h of work.

If the organic membrane had low mechanical strength, it was very likely that the fiber breakage would be done continuously during the experiment.

The relationship of dependence of parameters and physical-chemical indicators in this paper consisted in the fact that all the parameters within the entire system of the MP 90 pilot system were influenced by each other and the modification of some of them represented the modification of functionality to some extent of the whole process.

The present paper had the main purpose of highlighting the membrane efficiency of bentonite retention in an ultrafiltration process using a PAN organic hollow fiber membrane. The detailed role of this study was to highlight the parameters that have the greatest influence on the process during 5 h of wastewater filtration containing bentonite at a concentration of 5 g/L, respectively 0.5%.

As expected, a sustained decrease in permeate volume was recorded and the time interval of 5 h was sufficient to see this evolution of bentonite retaining on the membrane surface.

This study on the ultrafiltration of bentonite by means of a hollow fiber organic membrane has highlighted the dependence of the membrane parameters and MP 90 pilot parameters respectively, and the bentonite retention efficiency over the selected time interval has shown that this substance can be retained in the processes ultrafiltration by this type of membrane.

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