

Original Article: Isolation and Removal of Halostonitrile from Water by Hybrid Adsorption and Nano Filtration System

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ABSTRACT

In the present study, a hybrid method has been used to completely remove these compounds. This method is a combination of 3 different separation processes including ultra-filtration, fixed bed adsorption and nano filtration. The membranes used were made by Sepro USA and were made of Polyacrylonitrile and polyamide with 0.0162 and 0.0165 cm thickness and 0.05 and 0.003 μm pore sizes, ultra and nano types were used, respectively. The results showed that none of these processes alone can reduce the concentration to the standard level and eliminate them completely. The results of the hybrid combination of these processes showed that the use of ultra-filtration at the beginning and as a pretreatment increases the flux and thus increases the treatment rate. In general, the use of this combined method increased the speed of the treatment operation, reduced the choking rate and had a higher efficiency of the treatment operation. The results obtained from the study of adsorption models showed that the adsorption of halostonitriles on adsorbent particles conformed with the temkin equation. Optimal adsorption conditions of halostonitriles in continuous adsorption operation were obtained in a tower with a diameter of 1.6 cm with an adsorbent height of 75 cm.

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Introduction

Water is the backbone of the global economy, whose high quality and sustainability are of particular importance for agriculture, industry, recycling processes, energy production and urban consumption [1-3]. In recent decades, due to increasing demand and high utilization rates, as well as pollution of natural water resources, they have become vital. In addition, the growing population of the planet has provided the precondition for increasing demand for drinking water. Therefore, the improvement of water pollution removal technologies that are in line with environmental considerations has become one of the most important concerns of the 21st century [4]. Disinfection of drinking water is essential in providing safe and secure drinking water for urban use [4-6]. Disinfection processes have expanded since the beginning of the twentieth century. These processes are performed with different techniques and using different materials, and during these processes, microbes and microorganisms present in the water are killed or are largely inactivated [7-9]. Chlorine and its compounds are one of the most common disinfectants used for water. The low cost and long shelf life of this material justify its common use. After disinfection in treatment plants, chlorine remains in the water for a long time and prevents re-contamination of water in the distribution system [10-12]. The use of disinfectants reduces the risk of microbial diseases, but studies have shown that

disinfectants cause by-products of the disinfection process in water [13]. These substances are caused by chemical reactions between disinfectants (especially chlorine), water-soluble organic and inorganic compounds, and have adverse effects on human health, causing cancer. These substances are known to be toxic substances by water quality control agencies and should be removed from the water. Halvastonitriles are a by-product of the disinfection process present in water. Contaminant separation methods include microfiltration, ultrafiltration, Nano filtration, reverse osmosis and adsorption processes [14]. In this research, in order to determine the amount and extent of halostone nitrile compounds through the hybrid Adsorption-Nano filtration process, the effect of contact time, contaminant concentration and adsorbent concentration parameters has been investigated [15].

Standard values

Standard values are by definition values that express the concentration of components to such an extent that they do not endanger human health during a long period of water consumption. Institutions such as the World Health Organization (WHO) have introduced rules for the amount of halvostonitriles in drinking water [16]. According to the latest standard announced by the WHO in 2010, the permissible levels for halvostonitriles are listed in Table 1.

Table 1: Standard values declared by WHO

Halvastonitrile	Permissible amount (micrograms per liter)
DCAN	20
DBAN	70
TCAN	1
MCAN	80
MBAN	12

Materials and research methods

The experiments were performed separately on the ultrafiltration and Nano filtration membrane separation system and

discontinuous adsorption. After calculating the discontinuous adsorption data and thermodynamic and kinetic analyzes, the adsorption test was performed on fixed substrates. The efficiency of each method for

removing halostonitrile was selected for hybrid tests, the order with the highest efficiency was selected and the hybrid system tests were performed.

Batch adsorption was performed in the central laboratory of Abadan Oil Refining Company to observe the adsorbent behavior, draw adsorption isotherms, perform kinetic and

thermodynamic analyzes all membrane and hybrid tests.

Preparation of granular activated carbon

In this research, granular activated carbon used in Abadan refinery was used, the specifications of which are given in Table 2. Before using activated carbon, they were prepared.

Table 2: Physical characteristics of granular activated carbon

Value	Parameter
0.425	Bulk density (g/ml)
4.04	Solid density (g/ml)
0.52	Moisture content (%)
6.5	Ash content (%)
0.6 - 1.1	Particle size (mm)
0.67	Porosity (dimensionless)
413.3	BET surface area (m ² /g)
0.02	Surface acidity (meq.v/g)
2.34	Surface basicity (meq.v/g)

Performing the test

Adsorption tests were performed to determine the adsorption of halostonitriles by granular activated carbon adsorbent. All experiments were performed at 25 and 15 °C. For this purpose, samples of 100, 200, 300 and 400 micrograms per liter of halostone nitrile solution in water were prepared by double distillation [16-18].

Solution

The required concentrations for the experiments are 100, 200, 300 and 400 μg / lit of halostone nitrile in double distilled water. In order to study the adsorption behavior of halostonitriles and to prevent the adsorption of other compounds present in water on granular activated carbon and their effect on the adsorption of halostonitriles, double-distilled water, which is free of other compounds, was

used. During solubilization, the solutions were prepared in such a way that equal proportions (by weight) of the 5 halostone nitrile compounds were present in the samples. Due to the easier solubility of halostonitrile compounds in methanol, the specified amount of these compounds was dissolved in methanol and after creating an intermediate solution, this solution was diluted with distilled water [19]. Gouisti et al. showed that hydrophilic and water-miscible compounds such as methanol have a very low tendency to adsorb on granular activated carbon. The volume of methanol was selected to be approximately three times as much as the volume of halostone nitrile in the sample. In order to prepare test solutions with the mentioned concentrations, first a standard solution with a concentration of 10 gr / lit of halostonitrile in distilled water was prepared. Then, by diluting this standard solution, solutions with lower concentrations were prepared [20].

Table 3: Laboratory materials used in adsorption tests

a matter	Place of purchase	Density (g / ml)
MBAN (monochloroacetonitrile)	Aldrich	1/72
MCAN(monobromoacetonitrile)	Aldrich	1/19
DCAN(dichloroacetonitrile)	Aldrich	1/36
DBAN(dibromoacetonitrile)	Aldrich	2/29
TCAN (trichloroacetonitrile)	Aldrich	1/44
CH3OH	Merck	0/79

Study of absorption process

To investigate the effect of contact time, this test was performed at a constant initial concentration of 100 µg/lit, stirring speed of 110 rpm and with an adsorbent of 30 g l⁻¹ at a temperature of 25 ° C. In this test, the optimal contact time was 120 min.

Halvastonitrile uptake at any time was calculated using the following equation:

$$\text{Removal\%} = [(C_{in} - C_{out}) / C_{in}] * 100$$

In this equation, C_{in} and C_{out} are the input concentration and the output concentration of halostone nitrile at time t, respectively.

$$q_e (\mu\text{g } m\text{g}^{-1}) = \frac{C_0 - C_e}{M} * V$$

C₀ is the initial concentration and C_e is the final concentration at each stage of the experiment, V is the volume of the solution containing HAN and m is the adsorbent mass (g).

Table 4: Specifications of membranes used in ultrafiltration and nano filtration process

Firm	Model	Material	Thickness (mm)	Pore Size (µm)	Process pH	Pmax (MPa)	Tmax (oC)
Sepro	PAN-350	PAN	0.162	Size (µm)	10-3	8.4	100
Sepro	Nano filtration -1	PA	0.165	0.05	10-3	8.4	50

Two types of membranes were used for ultrafiltration and nano filtration processes. The membranes used for ultrafiltration and Nano filtration processes were made of polyacrylonitrile and polyamide, respectively [21-23]. The characteristics of membranes used in ultrafiltration and Nano filtration processes are given in Table 4.

Due to the nature of the present research project, some equipment was used to implement it, which will be explained in the following [24-26].

Membrane separation pilot

The most important tool for the experiments was a membrane separation pilot that was used to carry out the present project (Fig. 1). This pilot was made and used in the central

laboratory of Abadan refinery. The different parts of this membrane pilot are:

- Tanks: a tank is for storing food and a second tank for storing detergent. The tanks are made of stainless steel and both have a height of 1 meter and a diameter of 32 centimeters. The aqueous solution used in the present project contains a mixture of halostonitriles in pure water;
- Pump: A three-phase high-pressure diamond pump of OS-30A type made in China with a power of 3 hp (2/2 kw) was used to create the required driving force [27];
- Heat exchanger: It is a tube type with a length of 50 cm and a radius of 10 cm.;
- Thermometer: It is a hand type made by RTL company [28];

e) Barometers: They are of oily hand type made by TGI company. One of the barometers is installed at the input of the membrane module and the other at the output of Natrava. The correct amount of pressure on the membrane surface is equal to the average of the numbers shown by these barometers. Because the permeate was discharged to the feed tank at atmospheric pressure, there was no need to install a barometer at the permeation outlet and the pressure difference between the two sides of the membrane was equal to the pressure on the membrane surface [29]; and

f) Membrane module: It is one of the most important parts of the membrane pilot. This module is of disc type and made of stainless steel and the membrane is placed in a circle with a diameter of 11 cm. In addition to the above components, pipes, hoses and valves are also used in the membrane pilot. Pipes and valves are all made of stainless steel for working conditions at different pHs, and high-pressure hoses are used [30].

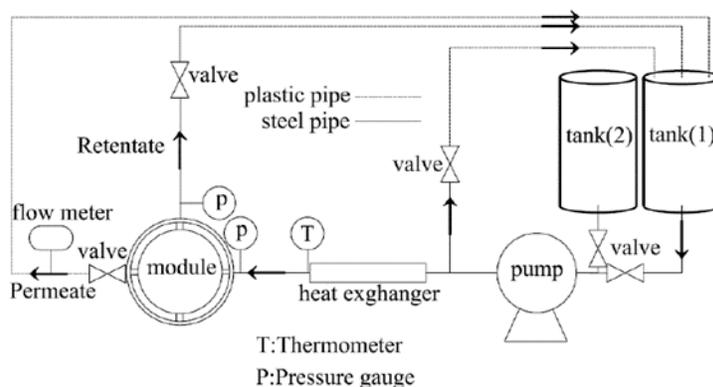


Figure 1: Schematic structure of the membrane pilot

The experimental process was based on two general steps. The first step was to investigate the ability of halostonitrile to remove from the initial synthetic input drinking water for each separation process separately [31-33]. In this way, each of the discontinuous and continuous adsorption experiments, ultrafiltration and Nano filtration were performed separately and independently on the input synthetic aqueous solution and the effect of each of these methods was investigated separately. The second series of experiments was to investigate the ability of halostonitrile to remove from drinking water in a primary synthetic aqueous solution as a hybrid combination of these processes, which is the main purpose of this study. The reason for dividing the experiments into two parts was that the results of the first series of experiments made it possible to decide to obtain a logical relationship and the order of using separation methods in the second phase of the experiments, which is the hybrid phase [34-36].

The parameters studied in the first step of the first part of the research, which was the study of

the adsorption process in the discontinuous state, included the effect of the amount of adsorbent, time, temperature and initial concentration of the contaminant until the adsorption was complete and the final concentrations reached equilibrium concentration. These experiments were performed to investigate the adsorbent behavior and determine the appropriate pattern of adsorption prediction and using the obtained data, isotherm constants were obtained. These data were used for thermodynamic, kinetic and mass transfer analysis of the adsorption process. After performing these experiments and initial analyzes, the feed was passed through a fixed bed and by studying this process and reaching the necessary parameters for the best separation efficiency, ultrafiltration and Nano filtration membrane processes experiments were performed [36]. Prior to the start of the membrane test, a series of initial tests with distilled water and the initial synthetic aqueous solution were performed to identify possible system problems, to see how the membrane

pilot operated, and to prevent errors during the main tests. After eliminating the mentioned defects, the main tests began. The experiments were performed in such a way that the separation ability of each of the ultrafiltration and nano filtration membrane processes was tested separately to remove halo acetonitriles in the initial input synthetic solution. Each of the ultrafiltration and nano filtration experiments was performed separately on the input synthetic aqueous solution. Process pressures for each of the ultrafiltration and nano filtration membrane processes were considered to be 7 and 20 bar, respectively. Initially, before sending the main synthetic aqueous solution into the membrane pilot, the permeate flux for distilled water was obtained as a function of the pressure difference on the membrane surface. The importance of the relationship between permeate flux and pressure difference is due to the fact that the obtained values are a criterion for determining whether the membrane is clogged or not. After this step, experiments were started to determine the efficiency of the membranes in the treatment of the desired synthetic aqueous solution. After starting the machine with the main feed and

bringing it to the required pressures, for each process, the amount of permeate volume was recorded every 4 minutes until the permeate flux was fixed, and as soon as a constant and uniform state in the permeate flux was ensured, samples feed, the permeate and non-permeable were removed for analysis to be sent to the water laboratory of Abadan refinery for necessary tests and after performing the relevant tests, the effect of each of these methods was examined. In order to maintain the feed concentration in each experiment, both the permeate and non-permeate were returned to the feed tank. All experiments were performed at 30 °C. After completion of the experiments, the membrane and all parts of the pilot were thoroughly washed with distilled water. According to the results of the initial stage of the experiments, which was the study of different processes separately, it was decided to start the hybrid process with ultra-filtration as pre-treatment, followed by treatment with adsorption process as the main stage of treatment. Finally, separation by nano filtration was used as a post-purification or final purification step.



Figure 2: View of the hybrid process pilot



Figure 3: View of the membrane modulus

Adsorption and synthetic isotherms

Adsorption isotherms were used to investigate the adsorbent behavior and determine the equilibrium pattern. The adsorbent capacity was investigated at different times using kinetic models and finally the mechanisms of penetration of halostonitrile particles into the adsorbent and diffusion coefficient were determined using mass transfer analysis.

The quasi-first-order synthetic equation is as follows:

$$\text{Log}(q_e - q_t) = \text{Log}(q_e) - (t \cdot k_1) / 2.303$$

Quadratic velocity equation:

$$\frac{1}{(q_e - q_t)} = \frac{1}{q_e} + k_2 \cdot t$$

Crime transfer

The mechanism of penetration of pollutant particles consists of two stages. The first stage is the penetration of the film from the solution to the adsorbent surface and the second stage is the penetration into the particle. Analysis and understanding of the mechanism of influence in the adsorption process is possible by using experimental data and adapting them to the following equation:

$$q_t = k_p \cdot t^{0.5}$$

This relationship is presented by Weber and shows the mechanism by which the influence took place.

Result and discussion

Results of non-hybrid tests

The separating power of halostonitriles in the initial input synthetic aqueous solution was investigated separately for each of the separation methods. It was therefore measured. This was done to assess the occurrence and extent of membrane occlusion. Figure 1 shows the time permeation flux for distilled water in two ultrafiltration-nano filtration processes at 7 times the operating pressure difference. To do this, the permeation flux was measured every 2 minutes from the beginning of the experiment. As can be seen in Figure 1, over time, practically no change in the values of the permeation flux was observed. The reason for this can be attributed to the absence of distilled water from any solutes, suspended solids and organic matter. The amount of permeate flux in terms of time for distilled water in the two processes of ultrafiltration and Nano filtration is 250.29 and 10.119 liters per square meter, respectively.

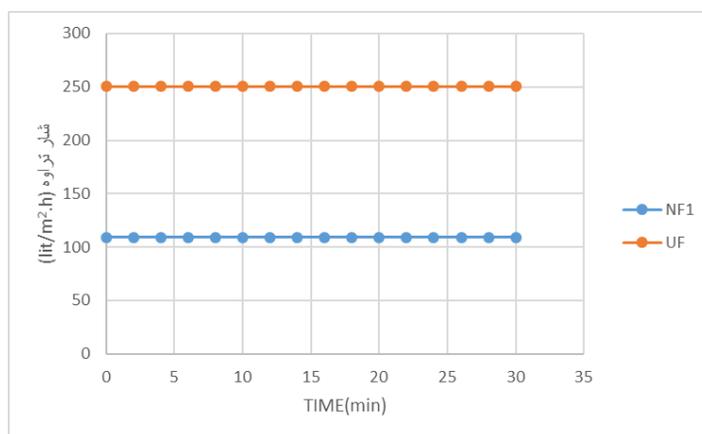


Figure 4: Time permeation flux for each of the UF and NF processes with distilled water at a pressure difference of 7 bar

Before starting the experiments, membrane permeability was measured at 25 °C for distilled water. In Figure 2, the permeation flux is plotted according to the pressure difference applied to the membrane. The linear relationship of the

permeate flux with the pressure difference indicated that the membranes followed Darcy's law:

$$J_v = L_p \cdot \Delta P$$

In the above relation, J_v is the permeability flux ($\text{m}^3/\text{s}\cdot\text{m}^2$), and L_p permeability ($\text{m}^3/\text{s}\cdot\text{m}^2\cdot\text{bar}$) and ΔP are the applied pressure difference (bar). According to Figure 4-2, L_p for ultrafiltration membrane is 5×10^{-6} $\text{m}^3/\text{s}\cdot\text{m}^2\cdot\text{bar}$ and nano

filtration membrane is 1×10^{-6} $\text{m}^3/\text{s}\cdot\text{m}^2\cdot\text{bar}$. These numbers were used as a reference to assess the likelihood of clogging as well as the efficiency of the washing process of each membrane.

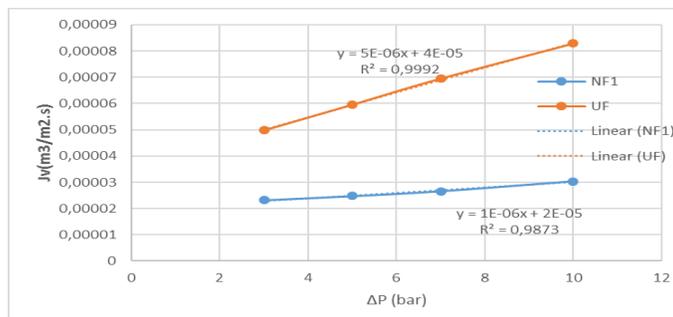


Figure 5: Permeation flux in terms of pressure difference applied to nano filtration and ultrafiltration membranes

Investigation of the effect of ultrafiltration on the treatment of synthetic aqueous solution

The percentage of pollutant reduction by the ultrafiltration process is shown in Figure 3. As can be seen, the ultrafiltration process is not able to bring the number of contaminants in the initial synthetic aqueous solution closer to the standard accepted in Iran shown in Table 5. It seems that the use of ultrafiltration membrane as a pre-treatment of the nano filtration process can help increase the efficiency of the hybrid process to better purify the synthetic aqueous solution. Figure 4 also shows the time-varying permeation flux in the ultrafiltration process when used alone. To measure the permeate flux at this stage, the flux rate was measured every 4 minutes from the beginning of the experiment.

By studying this diagram, it is not possible to see a very significant drop in permeation flux over time as the synthetic aqueous solution passes through the ultra-filter membrane. Figure 4 shows that the rate of drop of the permeate flux was not significant with the operation time, and finally after about 60 minutes from the start of the test, its intensity was slightly reduced. This flux drop can be due to the effect of concentration polarization and the formation of a thin layer of cake on the membrane surface. The reason for the formation of the cake layer and as a result of clogging can be the accumulation of contaminants and the joining of contaminant molecules in the synthetic aqueous solution as well as the size of the fine cavities of the ultra-filter membrane.

Table 6: Effect of feed concentration on permeation concentration in the ultrafiltration membrane used

Permissible amount in drinking water ($\mu\text{g}/\text{lit}$)	Permeation concentration ($\mu\text{g}/\text{lit}$)	Feed concentration ($\mu\text{g}/\text{lit}$)
60	270	1000
60	215	750
60	93	400
60	72	250

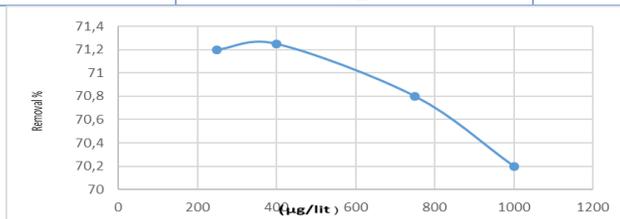


Figure 7: Percentage of halostatone removal at different feed concentrations at 7 bar pressure difference for the NF process in a series of non-hybrid experiments

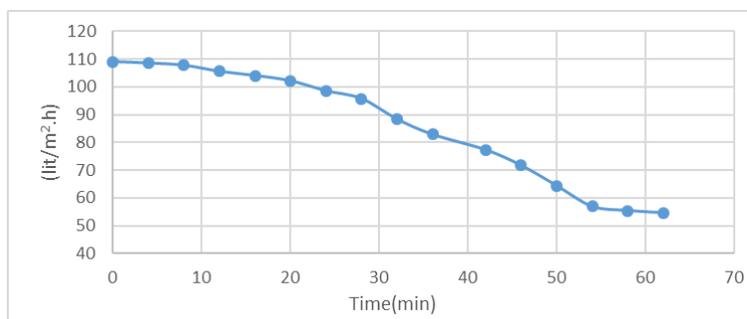


Figure 8: Time permeation flux for NF process at feed concentration of 250 $\mu\text{g/l}$ and pressure difference of 7 times in a series of non-hybrid experiments

Results of hybrid tests

As can be seen from Table 7, the characteristics of the effluent from each of the non-hybrid processes failed to meet the standards of aqueous solution disposal of industrial synthetics as well as drinking water standards provided by the Iranian Institute of Standards and Industrial Research, the Environmental Protection Agency and the World Organization. At this stage, the tests were performed as a hybrid to test the ability to remove

contaminants in this series of tests. According to the results obtained from the series of non-hybrid experiments, it can be concluded that in the series of hybrid experiments, which is a combination of the mentioned processes, ultrafiltration filtration is used as pretreatment and then adsorption in the fluidized bed as the main stages of purification. And, nano filtration membrane process was used as post-filtration or final purification steps. The results of this series of experiments are presented in Table 7.

Table 7: Feed and product characteristics by hybrid separation process

Halvastonitrile concentration ($\mu\text{g/l}$)				
250	400	750	1000	Primary feed
227	368	695	930	Ultrafiltration
-	162	-	434	Ads
72	93	215	270	Nano filtration
29	44	74	105	UF + Ads + NF
60	60	60	60	Iranian drinking water standard
40	40	40	40	EPA drinking water standard
60	60	60	60	WHO drinking water standard

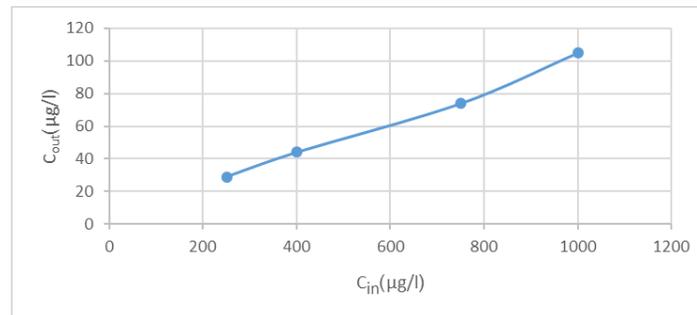


Figure 9: Output concentration of UF + Ads + NF hybrid process at different feed concentrations

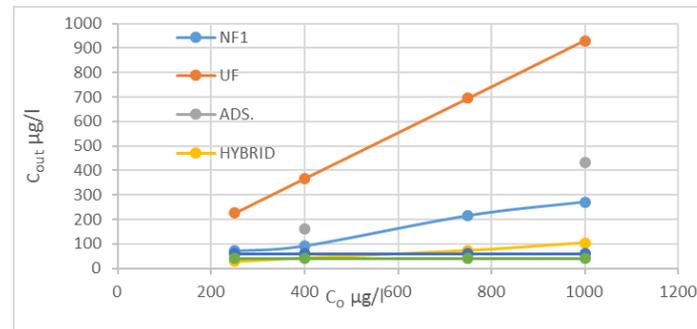


Figure 10: Output concentration of NF1, UF, ADS process individually and hybrid and comparison with Iranian and European standards in different feed concentrations

Investigation of the effect of tandem ultrafiltration processes, adsorption on a fixed bed and Nano filtration

According to the results presented in Table 7, this hybrid process, in addition to being able to bring the level of halvastonitrile pollution index completely to the standards of Iranian drinking water at concentrations below 500 µg / l, was also able to reach concentrations above that, approaching Iran's drinking water standard and reaching the standards of WHO in concentrations below 500 µg / l, and in concentrations below 250 µg/l, even complying with the European environmental protection standard.

Conclusion

Adsorption experiments showed that the adsorption percentage increased with increasing the amount of adsorbent. With increasing contact time, initially the removal of halostonitriles increased rapidly until it finally reached equilibrium. From that time on, there was no significant increase in the separation rate with increasing contact time. This efficiency occurred in the pH range of 7.5-8, with the

highest removal rate. The results were somewhat satisfactory by microfiltration and ultrafiltration membrane processes. It was found that in the ultrafiltration process, by increasing the concentration from 250 to 1000 micrograms per liter, the amount of separation percentage decreases by more than 1 percent.

In the nano filtration process, we saw a much better efficiency than ultrafiltration, so that in the initial feed concentration of 250, micrograms per liter of permeate concentration decreased from 227, micrograms per liter in the ultrafiltration process to 72, micrograms per liter in the permeability of the Nano filtration process, which shows the use of Nano-membranes can be a great help in water purification, but economic considerations are a barrier to the use of nano-filtration process, because in this case the amount of membrane used and the number of times it needs to be replaced will increase sharply. Pre-treatment is therefore required.

The hybrid process showed good results by using the ultra-filtration process as a pretreatment for adsorption and nano filtration. By increasing the concentration of halostonitriles in the feed from 250 to 1000,

micrograms per liter, it was determined that the concentration of halostonitriles in the output of the hybrid process reach 29 to 105 micrograms per liter and approximately up to a concentration of 500, micrograms per liter were observed. The output concentration of the process complies with the standard of the Ministry of Health and even exceeds the standards of drinking water in Iran and the pollution indicators of halostonitriles to the global standards of drinking water provided by the Environmental Protection Agency and the WHO. It is hygienic, of course, in concentrations above 1000, micrograms per liter; this hybrid system can no longer be used.

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