



## Original Research Article

# Haloacetonitriles (HANs) Removal Assessment from Aqueous Solution by Hybrid Adsorption/Membrane Filtration Process

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## ARTICLE INFO

## Article history

Submitted: 2020-06-07

Revised: 2020-8-26

Accepted: 2020-09-18

Manuscript ID: CHEMM-2009-1278

DOI: [10.22034/chemm.2021.118773](https://doi.org/10.22034/chemm.2021.118773)

## KEYWORDS

Haloacetonitrile

Nano-filtration

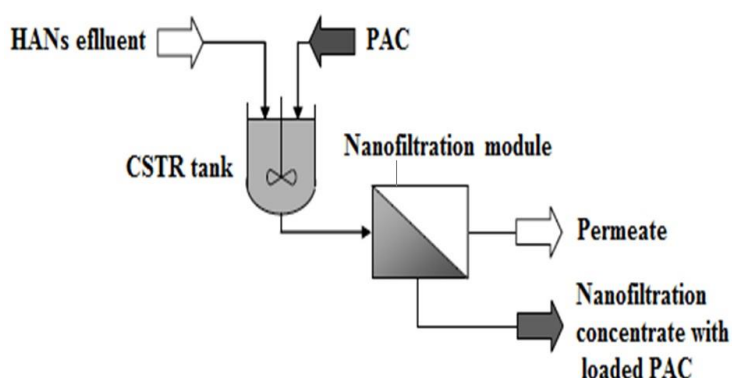
Adsorption

Hybrid adsorption

## ABSTRACT

The haloacetonitriles (HANs) exist in drinking water exclusively as byproducts of disinfection. HANs are found in drinking water more often with higher concentrations. Human exposure occurs through consumption of finished drinking water, oral and dermal contact as well as when showering, swimming and other activities. The purpose of this study was to investigate the novel hybrid adsorption/ PBS membrane filtration process used to remove haloacetonitriles from aqueous solution. The efficiency of the PAC adsorbent was also evaluated at different pH, which confirmed efficacy of PAC at pH of 8. A maximum removal of DOC (98.7%) was obtained with 150 mg/L PAC dosage at a solution pH of 8. HANs sorption process followed the pseudo second order kinetic model and the Langmuir isotherm gave the best correlation of adsorption.

## GRAPHICAL ABSTRACT



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## Introduction

The haloacetonitriles (HANs) exist in drinking water exclusively as byproducts of disinfection. HANs are found in drinking water more often with higher concentrations. Human exposure occurs through consumption of finished drinking water, oral and dermal contact as well as when showering, swimming and other activities [1-3]. Due to their high cytotoxicity and genotoxicity, HANs require more consideration and various processes, such as adsorption [4-7]. Recently, among membrane technologies, nano filtration and reverse osmosis have attracted a great deal of attention for use in water softening and the removal of various contaminants from drinking-water sources. However, these technologies have inherent drawbacks; they have lower permeated flux in nano filtration and higher trans membrane pressure in reverse osmosis, thus lower process efficiency and higher cost of investment and operation. Recently, hybrid membrane filtration systems are used as an alternative way to achieve a high removal efficiency of organic matter in a cost-effective manner [8-15].

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].

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 increase in the Earth's population 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 [1].

Disinfection and disinfection of drinking water is essential in providing safe and secure drinking water for urban use. Disinfection processes have been expanded since the beginning of the 20th 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. 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. The use of disinfectants reduces the risk of microbial diseases, but studies have shown that disinfectants cause by-products of the disinfection process (DBPs) in water. These substances are caused by chemical reactions between disinfectants (especially chlorine), water-soluble organic and mineral compounds, and have adverse effects on human health, causing cancer. They are considered as toxic substances by water quality control institutions and should be removed from the water. Haloacetonitriles are a group of DBPs present in water. Contaminant separation methods include microfiltration, ultrafiltration, nanofiltration, reverse osmosis and adsorption processes.

This study addressed the removal of HANs from aqueous solution by adsorption- nano-filtration hybrid process. For a better understanding of the adsorption mechanism, the adsorption isotherms fitted on the experimental data of the studied system. In addition, kinetic study was carried out in order to determine the controlling mechanism of the process. Finally, the effect of PAC on poly (butylene succinate) (PBS) membrane performance was investigated [16-19].

### *Nano-filtration (NF)*

Nano-filtration was referred to as hyper filtration in the 1980s and has always been associated with the RO process and the like. Today, NF is used as a completely separate process with special application properties and is fundamentally different from the RO and UF processes. In other words, the membranes used in the NF process have a porous structure of micro type with apertures of less than 2 nm and are mainly made of polymeric materials, which in most cases have an ionic charge. The mechanism of their separation is screening, solution dispersion and drift [2]. Because of the high

pressure and differences commonly required in RO membranes, significant energy costs were required, so the construction of such membranes was a major breakthrough in the membrane separation industry [3]. In addition, due to the reduced costs of pumps, pipes and tanks at low pressures, the NF process required less initial investment compared with RO. Due to its high similarity with UF, RO and electro dialysis processes, NF process can be a suitable alternative to these processes in most of their applications

## Material and methods

Commercial standards including mono chloroacetonitrile (MCAN), mono bromo acetonitrile (MBAN), dichloroacetonitrile (DCAN), dibromoacetonitrile (DBAN), trichloroacetonitrile (TCAN) were purchased from Aldrich. Wooden PAC was purchased from Shanghai activated carbon Company (Shanghai, China). The grain size distribution of PAC used this study was >74 nm-77 percentage, >34 nm-90 percentage. Other chemicals used in this study were of analytical grade and procured from Merck, India. Further, distilled water was used throughout the solution preparations.

### *Durability of halo acetonitrile in the environment*

Halogenated acetonitriles including dichloroacetonitrile and dibromoacetonitrile and bromochlorostonitrile are hydrolyzed in water. Dichloroacetonitrile and di bromoacetonitrile are very persistent in soil. In wet alkaline soils dichloroacetonitrile and dibermostonitrile may be hydrolyzed. In aqueous medium, debermostonitrile is hydrolyzed, although its hydrolysis rate is higher in alkaline waters and in the presence of chlorine. Approximately 10 to 60% of dichloroacetonitrile and 5 to 20% of dicermasto-nitrile were hydrolyzed over 10 days at pH 6 to 8. Due to these conditions, damage to the environment is reduced to some extent and their deposition in the environment is reduced to some extent.

### *Preparation and characterization of biodegradable PBS membrane*

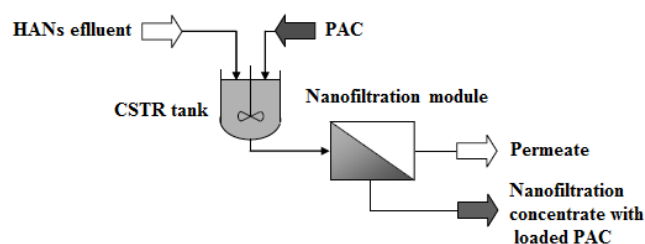
The preparation of asymmetric flat sheet membrane of PBS was carried out by the phase inversion method. At first, PBS (17 wt. %) was dissolved in 1-methyl-2 Pyrrolidone (NMP). After 4 h of stirring, the solution was cast with 250  $\mu\text{m}$  casting knife onto a glass plate and then immersed in water coagulation bath ( $T=50\text{ }^{\circ}\text{C}$ ). After that, the membranes transferred to distilled water for 24 h at room temperature to remove the remaining solvent from the membrane. Finally, the membrane was dried at  $40\text{ }^{\circ}\text{C}$  by vacuum oven [20, 21]. Detailed properties of the PBS membrane are presented in Table 1.

**Table 1:** Properties of prepared PBS membrane

Property	Value
Porosity (%)	45
Contact angle( $^{\circ}$ )	76
Pure water permeability ( $\text{Lm}^{-2}\text{ h}^{-1}\text{bar}^{-1}$ )	18.3
Average pore size(nm)	43
Tensile strength (MPa)	53.2

### *Process design*

The schematic diagram of PAC/NF hybrid process is shown in Figure 1. The different dosages of powdered activated carbon are added to water sample including HANs molecules in a CSTR tank (contact time: 1h). After that, the suspension was fed to the nano-filtration unit. The PBS membrane was used for solute rejection analyzing with an effective filtration area of  $50.24\text{ cm}^2$ . The membrane operated with a cross flow velocity of  $1.2\text{ m/s}$  and a recovery of 75 %. The permeate flux was measured by collecting the volume of permeate (20 ml) at applied pressure of seven bars in a measuring vessel over a given period. The collected permeate solution was analyzed by measuring of DOC in a TOC analyzer (Shimadzu, TOC V-CPH) and ultraviolet absorbance at 254 nm (UV254) in an UV-visible spectrophotometer (PerkinElmer, Lambda 35).



**Figure 1:** Schematic of the PAC/NF process

### Adsorption Study

All adsorption experiments were performed at room temperature ( $\sim 25^\circ\text{C}$ ). 0.025 gr of PAC was mixed with 30 mL of distilled water and then stirred for 30 min by rapid mixing at 100 rpm, and the ionic strength IS was fixed at  $10\text{ m mol L}^{-1}$  using phosphate buffer. The water sample was filtrated by a filter (pore size:  $0.45\text{ }\mu\text{m}$ ). After that, the equilibrium concentrations of HANs in the supernatant solution were measured by a gas chromatography-mass spectrometry (GC-MS) (GCMS-QP2010 SE, Shimadzu, Japan) according to EPA method 551.1 [22]. The percentage of HANs adsorption was given by the following equation 1:

$$\text{HANs adsorption (\%)} = \frac{C_f - C_p}{C_f} \times 100 \quad (1)$$

Where,  $C_f$  and  $C_p$  are the concentrations of HANs at the initial time and at time  $t$  ( $\mu\text{g L}^{-1}$ ) respectively. The amount of HAN adsorbed ( $\mu\text{g HAN/g adsorbent}$ ) was determined by following equation:

$$q \left( \frac{\text{mg}}{\text{g}} \right) = \frac{(C_f - C_p)V}{m} \quad (2)$$

Where,  $V$  is the volume of HAN containing solution and  $m$  is the mass (g) of adsorbent.

### Adsorption isotherms and kinetics

Three kinetic models were considered to investigate the mechanism of adsorption [23]. These models included pseudo-first order, pseudo-second order and intraparticle diffusion models.

The pseudo first order model is given by following equation 3 [24]:

$$\ln(q_s - q_t) = \ln q_s - k_1 t \quad (3)$$

The pseudo-second order kinetic rate equation 4 is expressed as [25]:

$$\frac{t}{q_t} = \frac{1}{k_2 q_s^2} + \frac{t}{q_s} \quad (4)$$

The Intraparticle diffusion model is given by equation 5 [26]:

$$q_t = k_d t^{1/2} + C \quad (5)$$

In these equations  $k_1$  ( $\text{min}^{-1}$ ),  $k_2$  ( $\text{g } \mu\text{g}^{-1} \text{ min}^{-1}$ ) and  $k_d$  ( $\mu\text{g g}^{-1} \text{ min}^{-1/2}$ ) are the rate constants of pseudo-first

order, pseudo-second order and intraparticle diffusion models.  $q_e$ ,  $q_t$  ( $\mu\text{g g}^{-1}$ ) are the adsorption uptake at equilibrium and at time  $t$  (min). In addition,  $C$  constant represents the boundary layer thickness. The adsorption isotherm models such as those of Langmuir and Freundlich models represent the equilibrium distribution of HANs molecules between the aqueous and solid phases [23]. The Langmuir adsorption isotherm is shown in following equation 6:

$$q_s = \frac{q_m \cdot K_L \cdot C_s}{1 + K_L C_s} \quad (6)$$

Where  $q_m$  is the maximum adsorption capacity ( $\mu\text{g g}^{-1}$ ) and  $K_L$  is the Langmuir constant.

The Freundlich equation 7 is [27]:

$$q_s = K_F C_s^{1/n} \quad (7)$$

Where  $K_F$  and  $n$  are the Freundlich constants.

### Result and discussions

#### Effect of adsorbent dosage on the adsorption of HANs

Figure 2 presents the effect of PAC dosage on the adsorption of HANs molecules. As shown in figure 2, by increasing the PAC dosage more than  $150\text{ mg/L}$ , the percentage sorption increased and the adsorption uptake decreased. This can be attributed to the availability of more active adsorption sites on the PAC with increasing the dose of the adsorbent. On the other hand, the decrease in the adsorption uptake is due to unsaturated sites on the PAC at a high dosage. A maximum uptake of HANs molecules was obtained using  $150\text{ mg/L}$  of the adsorbent.

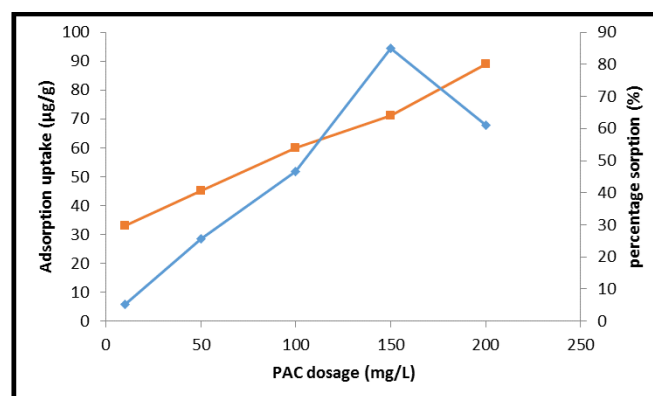
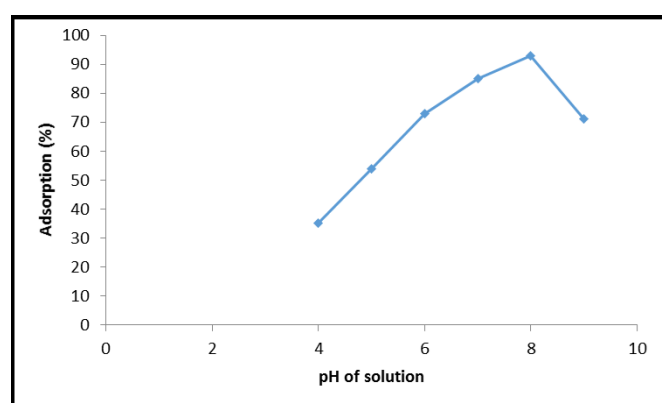


Figure 2: effect of PAC dosage on the adsorption of HANs

### Effect of pH on DCAN adsorption

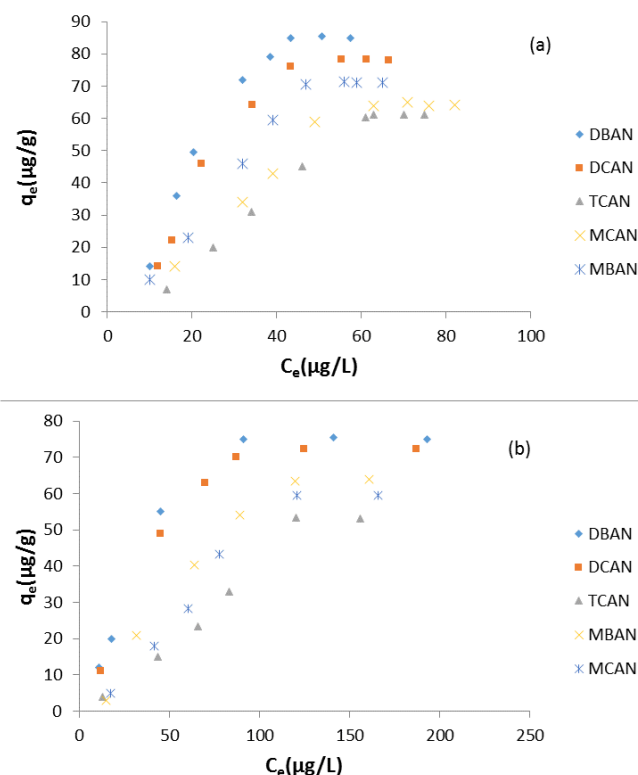
Investigating the effect of initial pH of solution on the removal of DCAN by PAC is represented in Figure 3. It can be observed that the removal percentage increased with the increasing of pH up to eight and then decreased with the increasing of Ph. The maximum DCAN removal was observed at PH 8. The DCAN gives positively charged ions when dissolved in water. At pH of 9, PAC surface will be negatively charged and lead to increasing electrostatic interaction between the positive dipole of the H atom of DCAN molecules which favored the uptake of DCAN molecules. At pH<seven the positively charged surface of sorbent tends to oppose the adsorption of the cationic adsorbate.



**Figure 3:** effect of solution PH on DCAN adsorption

### Selective adsorption of HANs in solution

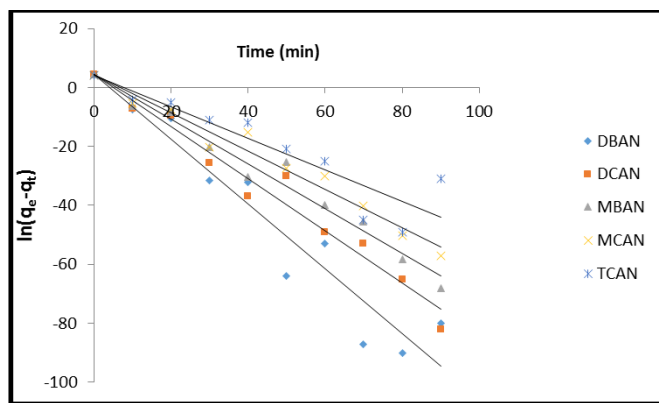
The results showed that the order of HANs adsorption on PAC was DBAN > DCAN > TCAN  $\cong$  MBAN  $\cong$  MCAN in the single-solute solution (Figure 4a). This might be explained by the fact that the HANs molecules with high molecular weight i.e. tri and di-HANs could have been more easily adsorbed than the smaller ones. The adsorption capacity of di-HANs is more than from tri-HANs. The adsorption capacity of each HAN in the mixed solute solution on PAC was slightly less than that in the single-solute solution due to the high concentration of mixed HANs solution and segregation of active sites for HANs (Figure 4b).



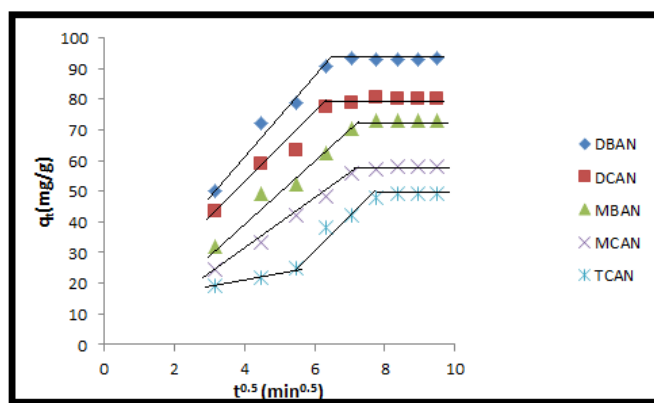
**Figure 4:** (a) Adsorption isotherms of HANs, (at: pH= 8, IS= 10 mmol L<sup>-1</sup>, and T =25°C), (b) In a single-solute solution, In mixed-solute solution (at: pH= 8, IS= 10 mmol L<sup>-1</sup>, and T =25°C)

### Adsorption Kinetics

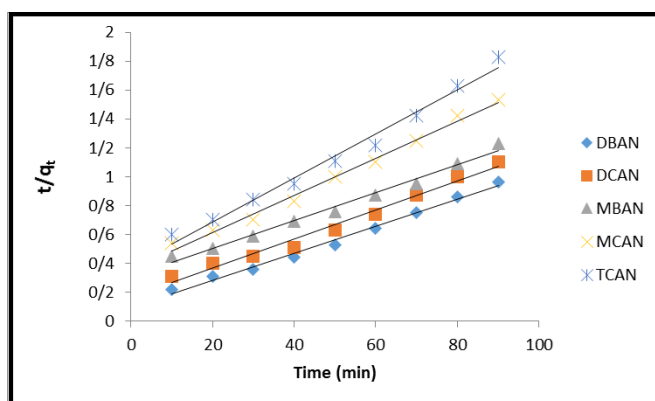
The adsorption kinetic results are illustrated in Figures 5-6 and listed in Table 2. The results showed that the adsorption of HANs on to the PAC followed the pseudo second-order kinetic model. It was discovered that this model gave a better straight line with square of correlation coefficient of  $R^2 > 0.97$ , compared with that of the pseudo first-order model [28-30]. The usual adsorption process over porous adsorbents comprised three steps; external diffusion, film diffusion, inter-pore diffusion and site adsorption steps [31]. The data-fitting curves for intraparticle diffusion model are shown in Figure 7. As can be seen, the  $q_t$  vs.  $t^{0.5}$  relations for TCAN displays multi-linearity, indicating that multiple adsorption steps are involved in the adsorption process. TCAN adsorption on PAC showed at least two phases but did not plateau out.



**Figure 5:** Pseudo-first order adsorption kinetics of HANs onto PAC adsorbent



**Figure 7:** Intraparticle diffusion model for the HANs adsorption PAC



**Figure 6:** Pseudo-second order adsorption kinetics of HANs onto PAC adsorbent

**Table 2:** The first and second order kinetic constants of BSA nanoparticle adsorption

HAN	First-order kinetic			Second-order kinetic		
	$q_e$ ( $\mu\text{g}\cdot\text{g}^{-1}$ )	$k_1$ ( $\text{min}^{-1}$ )	$R^2$	$q_e$ ( $\mu\text{g}\cdot\text{g}^{-1}$ )	$k_2$ ( $\text{g}\cdot\mu\text{g}^{-1}\cdot\text{min}^{-1}$ )	$R^2$
DBAN	93.2	1.1	0.92	107.2	1.12	0.99
DCAN	90.2	0.88	0.96	99.4	1.09	0.99
MBAN	91.3	0.76	0.96	102.2	0.94	0.98
MCAN	70.2	0.64	0.96	78.12	0.9	0.99
TCAN	56.4	0.53	0.83	65.3	0.87	0.97

**Table 3:** Kinetic parameters of HANs adsorption onto the PAC adsorbent using the intraparticle diffusion model

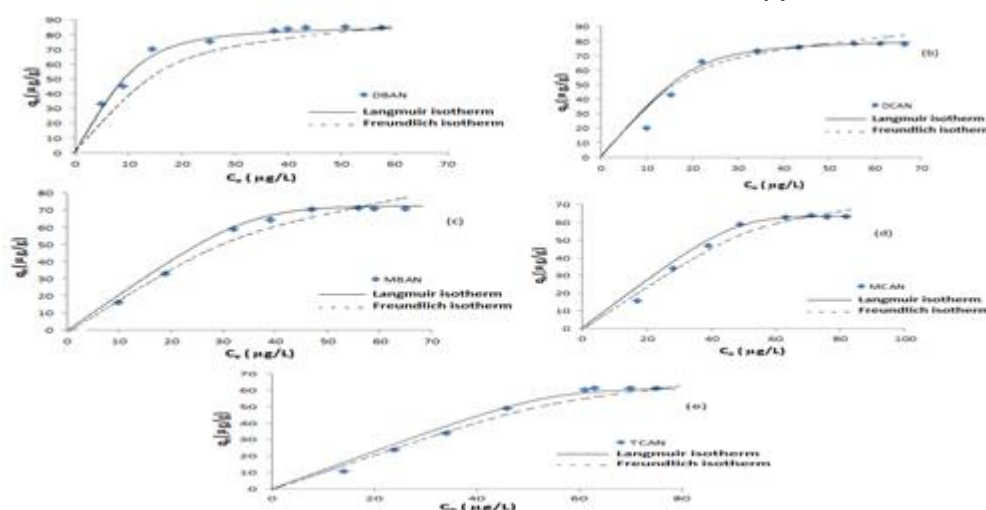
HANs	$K_{d1}$	$C_1$	$R^2$	$K_{d2}$	$C_2$	$R^2$
DBAN	13.05	9.02	0.98	–	–	–
DCAN	10.69	8.17	0.97	–	–	–
MBAN	9.47	1.2	0.97	–	–	–
MCAN	7.7	0.5	0.99	–	–	–
TCAN	1.1	0.97	0.99	6.58	1.15	0.98

### Isotherm models

Adsorption isotherms including the Langmuir and Freundlich were explored as shown in Figure 8. The best fitting for HANs adsorption on PAC

was supported by the  $R^2$  values for the two isotherms (Table 4). The exponent  $1/n$  value for HA can be seen.





**Figure 8:** Comparison of the predicted and experimental data for the equilibrium adsorption of five HANs on PAC, (a) DBAN, (b) DCAN, (c) MBAN, (d) MCAN, (e) TCAN

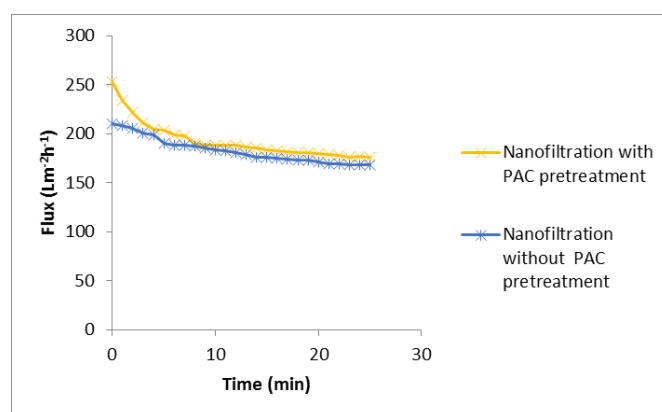
**Table 4:** Isotherm parameters of HANs adsorption on PAC adsorbent

	HANs				
Isotherms	DBAN	DCAN	MBAN	MCAN	TCAN
Langmuir					
$q_m(\mu\text{g g}^{-1})$	533.3	502.1	491.3	334.5	298.2
$K_L(\text{L}\mu\text{g}^{-1})$	9.1	8.36	7.78	7.1	6.3
$R^2$	0.99	0.98	0.99	0.98	0.97
Freundlich					
$1/n$	0.65	0.49	0.48	0.45	0.42
$K_F(\mu\text{g g}^{-1})$	620.2	631.2	602.3	600.3	593.3
$R^2$	0.78	0.83	0.85	0.89	0.93

#### PAC pretreatment effect on Flux of PBS Membrane

In this study, the influences of the PAC pretreatment on the permeation flux and rejection of HANs are shown in Figure 9. As can be seen, the flux decline takes place with time so that this decline is rapid during the first 5 min. The flux then gradually decreases until it reaches a constant value. The concentration polarization and fouling of the membrane can be responsible for this phenomenon [21]. Obviously, the permeation flux increases with usage of PAC. It might be related to the nature of the moderate hydrophilic HANs (except TCAN). On the other hand, PAC particles selectively adsorbed hydrophilic organic matter, so the hydrophilic interaction between the PAC particles and HANs molecules increase. In contrast, the surface of PBS membrane is hydrophobic (contact angle:  $76^\circ$ ). Therefore, hydrophobic adsorption between water and PBS membrane is reduced and then

consequently cannot result in membrane fouling. The membrane/adsorption configuration that introduced in this study could adsorb the HANs and reduce the costs associated with fouling in membrane unit.

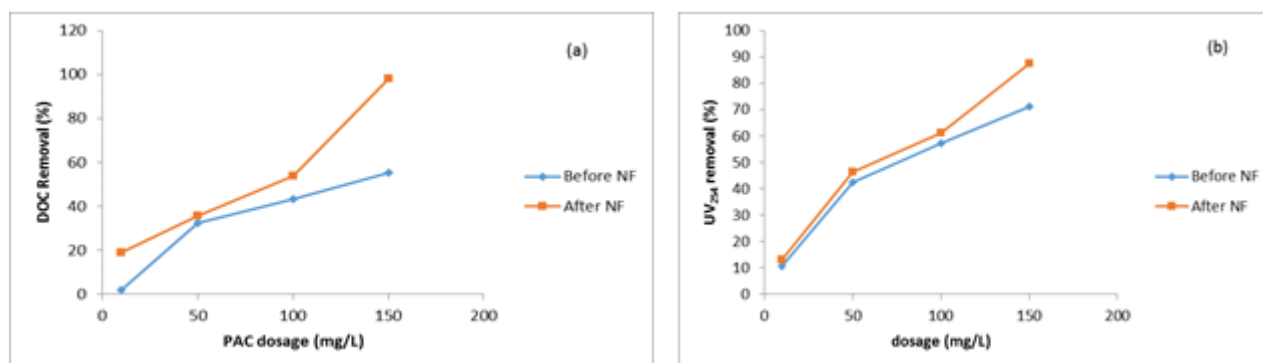


**Figure 9:** Effect of PAC pretreatment on PBS Membrane Flux

### DOC and UV<sub>254</sub> Removal Efficiencies of PAC and NF Process

The percentage of DOC and UV<sub>254</sub> removal after water sample passed through hybrid process is shown in Figure 10. As can be seen, DOC removal increased from 2.0 % to 55.3 % by increasing PAC dosage up to 150 mg/L. With connecting of NF process to the PAC adsorption, the DOC removal of permeate increased from 19.1 % to 98.2 %. Like

DOC removal, a high removal rate of UV<sub>254</sub> was achieved. The UV absorbance removal by PAC adsorption increased up to 71.2 % with the increase of PAC dosage (up to 150 mg/L). After NF process connecting, at higher PAC dosage, the higher UV<sub>254</sub> removal of 87.6 % was achieved. As shown in Figure 10, addition of NF process to the PAC adsorption gently improved the final DOC and UV<sub>254</sub> removal from water.



**Figure10:** DOC and UV<sub>254</sub> Removal vs. PAC dosage in PAC/NF hybrid process

### Conclusion

HANs removal from aqueous solution was investigated by a PAC adsorption/ PBS membrane hybrid process. HANs adsorption on PAC adsorbents followed a pseudo-second order rate kinetics model and the Langmuir isotherm could predict the equilibrium adsorption of five HANs on PAC. Furthermore, DCAN adsorption was highly pH dependent, being favored at a high pH due to having a higher negative surface charge density. The PAC adsorption was added as the pretreatment in to NF membrane process in order to remove HANs and reduce membrane fouling. PAC improved membrane fluxed slightly from 210 to 253 Lm<sup>-2</sup>h<sup>-1</sup>. The results showed that by PAC adsorption, higher DOC and UV<sub>254</sub> removal was achieved in hybrid process. During 10–150 mg/L PAC dosage at hybrid process, DOC removal efficiencies of 19.1 %-98.2 % and UV<sub>254</sub> removal rate up to 87.6 % were obtained.

### Acknowledgements

The study presented in this paper is part of a research project of Mohsen Vaziri (Department of Chemical Engineering, Mahshahr Branch, Islamic Azad University, Mahshahr, Iran).

### Conflict of Interest

The authors declared that they have no conflicts of interest to this work.

### Funding

The authors gratefully acknowledge the Islamic Azad University of Kerman for financial supports.

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#### HOW TO CITE THIS ARTICLE

Mohsen Vaziri, Seyed Mostafa Tabatabaee Ghomsheh, Alireza Azimi, Masoomeh Mirzaei, Haloacetonitriles (HANs) Removal Assessment from Aqueous Solution by Hybrid Adsorption/Membrane Filtration Process, Chem. Methodol., 2021, 5(1) 41-49

DOI: [10.22034/chemm.2021.118773](https://doi.org/10.22034/chemm.2021.118773)

URL: [http://www.chemmethod.com/article\\_118773.html](http://www.chemmethod.com/article_118773.html)