

## **Ion Exchange Processes: A Potential Approach for the Removal of Natural Organic Matter from Water**

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### **Abstract**

Natural organic matter (NOM) is among the most common pollutant in underground and surface waters. It comprises of humic substances which contains anionic macromolecules such as aliphatic and aromatic compounds of a wide range of molecular weights along with carboxylic, phenolic functional groups. Although the concentration of NOM in potable water usually lies in the range of 1-10 ppm. Conventional treatment technologies are facing challenge in removing NOM effectively. The main issues are concentrated to low efficiency, membrane fouling, and harmful by-product formation. Ion-exchangers can be considered as an efficient and economic pretreatment technology for the removal of NOM. It not only consumes less time for pretreatment but also resist formation of trihalomethanes (THMs), an unwanted harmful by-product. This article provides a comprehensive review of ion exchange processes for the removal of NOM.

**Key words** : Climate change; Global warming; Sanitation; Ion-exchange; Natural organic matter (NOM)

### **1. Introduction:**

Natural organic matter (NOM) is a complex mixture which mainly includes organic compounds such as polysaccharides, humic acids, fulvic acids and other small organic compounds that can be present in dissolved and particulate forms in underground and surface waters [1]. Figure 1 illustrate the brief classification of natural organic matter. NOM can be divided into two categories i.e. microbially and terrestrially. Microbially derived NOM are generated by microorganisms and can be found in streams, lake, wells and reservoirs [2]. Intense color, high carbon to nitrogen ratio (around 100/1) represents terrestrially derived NOM. They mostly comprise organic portion of plants and can be found in

rivers and canals [2]. In addition, there is one characterization of NOM, colloidal natural organic matter. Those mostly includes relatively polar amino sugars and can be responsible for fouling. The main reason for their high potential regarding fouling is because of their neutral nature [3]. Complete characterization of NOM is usually very difficult mainly due to its complexity variability and typical chemical structure. Focussing on some of basic chemistry of NOM is generally more useful in understanding the mechanism of NOM removal.

Sources of potable water typically consist of 1-10 mg/l of NOM and till now only 10-30% has been investigated and identified [4]. There are numerous reasons why it poses challenges for water treatment industries. The foremost issue is the formation of carcinogenic chlorinated hydrocarbons during the disinfection process with chlorine [5,6]. In the last decade there are a number of reports highlighting

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the formation of new aromatic halogenated disinfection by-products (DBPs), approximately 600-700 DBPs can be formed [7-9]. Haloacetic acids (HAAs), as an example are considered harmful for human and responsible for toxicological effects on both terrestrial and aquatic animals. Trihalomethanes (THMs) are another example of DBPs, which has been identified as carcinogens for humans [10]. NOM can also act as electron transfer agents through oxidation of dissolved iron and manganese which transform these metals from soluble to insoluble state, catalysing the corrosion process [11]. NOMs are also responsible for fouling of membranes and can adversely affect the efficiency of many adsorbents like activated carbon [12-15]. The removal of NOM mainly from potable water is highly desirable due to aesthetic concerns such as stinky odour, unacceptable taste and color [16].

Numerous conventional treatment methods were already applied for removal of NOM depending on their physiochemical properties [17,18]. Among

them the most common are coagulation, membrane filtration, biological processes, advanced oxidation and ion exchangers [19-21]. Researchers found that the ion exchange process holds a great potential for the elimination of NOM from both drinking water and industrial waste water. In last decade numerous work has been done on ion exchange treatment method over NOM. Bench or pilot scale column test have been studied to analyse the treatment efficiency of NOM, operating conditions and capacity of an ion exchanger resins. Recently, many varieties of ion exchanger are developed such as fluidized ion exchanger (FIX) [22], suspended ion exchange (SIX) [23], Magnetic ion exchange (MIEX) [24] and ion exchangers can also be coupled with other treatment technologies to further enhance NOM removal efficiency.

In this review, the main objective was to highlight the recent work done on ion exchangers regarding NOM removal efficiency from drinking water.

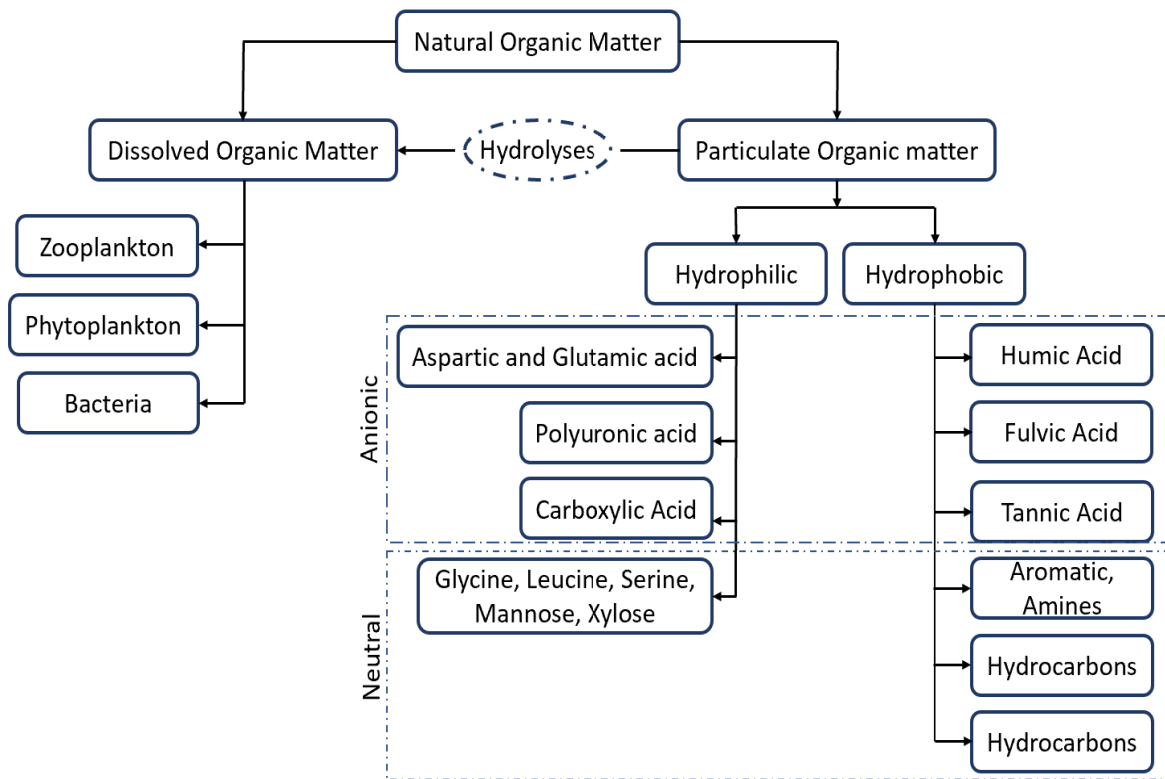


Fig. 1: Classification of Natural Organic Matter (NOM) (Adapted from [16,25]).

## 2. Ion Exchange Process

According to [26,27], ion exchange is a reversible process in which the exchange of ions takes place between solid and liquid phases. The solid phase is insoluble in liquid and holds exchangeable ions and hence called ion exchanger. In addition, those are very stable and does not possess structural changes during reactions [28]. Ion exchangers are broadly classified as cation exchangers and anion exchangers. Some of the main characteristics which determine the quality of ion exchangers are physiochemical stability, ion exchange capacity, hydrophilicity, effective surface area, economic value and particle size [26].

Nowadays, a wide range of ion exchange resins are available depending on the type of application. Synthetic resins are more prominent than natural resins and are used widely in waster water and other commercial applications. A brief summary of different ion exchangers for removal of NOM are depicted in Table 1. Numerous types of ion exchange resins available can be categorized as Strong-acid cation resins, Strong-base anion resins, Weak-acid cation resins, Weak-base anion resins, and Metal-selective chelating resins [29].

Strong acid cation resins mostly consisted of sulfonic acid functional groups or other strong acid functional groups. Strong base anion resins may contain functional groups of ethanol or multiple methyl groups. They also hold different characteristics, such as better stability in the case of methyl groups but higher efficiency in the case of ethanol groups [27].

Weak acid cation resins contain carboxylic functional groups whereas Phenol formaldehyde with groups of amines attached with it represents weak base anion resins. Metal selective chelating resins have greater potential against heavy metals. Ethylenediaminetetraacetic acid (EDTA) compounds are usually attached to these resins as these are very similar to weak acid cation resins [27]

## 3. Recent Development of Ion Exchangers

### 3.1. Fluidized Type Ion Exchange

Fluidized ion exchanger (FIX) can effectively be used for the removal of NOM [22]. It can also be employed to enhance the performance of downstream treatment processes. The main principle behind FIX is that the ion exchange resins settled faster than suspended solids present in the treated water. In other words, sedimentation rate of resins is much better than suspended solids. They are also equipped with specially designed vessels in which water flows in an upward direction. Hence, when the water to be treated is pumped up-flow manner at a specific velocity, the suspended solids contaminants present in water are removed easily. Few researchers tested this technology as a pretreatment method followed by mostly nanofiltration and ultrafiltration [22,30]. By employing this technology prior to the aforementioned filtrations, can significantly reduce membrane fouling. According to an experiment conducted by Cornlissen et al., (2010) [30], FIX satisfactory removed hydrophobic organic carbons (HOC) with more than 80% efficiency and humic substances with more than 90% of removal efficiency. In addition, iron which usually presents as complex compounds with humic substances was also removed with the remarkable efficiency of 71% from feed water. Contradicting to other reports, membrane fouling occurred especially biofouling despite FIX pretreatment for NOM removal. Another study conducted by [31] found a very low molecular weight NOM with functional groups of amine, carboxyl and aliphatic hydrocarbon after treatment of feed water of Nakdong River (South Korea). They associate FIX with ultrafiltration process and found a significant reduction in membrane fouling. There was a significant reduction in compounds with low aliphatic groups and phenolic compounds whereas reduction efficiency for carboxylic compounds was much lower. From the previous studies, it can be concluded that more focussed research and deep understanding of mechanism are still required to understand FIX

treatment effect for NOM removal on membrane fouling.

### 3.2. Magnetic Ion Exchange Process

Australian Water Quality Center (AWQC) along with Orica Australia Ltd. developed a magnetic ion exchange resin (MIEX). They used this system in a slurry reactor which was followed by a coagulation process [32]. The most innovative part of MIEX is that these resins are strong basic anion exchange resin with magnetic iron oxide in its core and polyacrylic as an outer matrix in the form of chlorine [33,34]. Separation and recycling after usage is comparatively easy due to the magnetic nature of resins [35]. MIEX also leads to better ion exchange kinetics due to the smaller size of resins particles which are 2-5 times smaller than conventional commercially available resins. This not only provides more contact surface but also reduces the contact time required for treatment. There are some advantages as well as disadvantage of MIEX. Advantage includes its stability for long time usage and no pretreatment is required which makes it a suitable option for potable water treatment [36]. On another side, if the waste water contains phosphate groups in it then MIEX can be an expensive option. Porous MIEX has the tendency to adsorb Phosphates which then resulted in the formation of biofilms that ultimately responsible for resin binding effect [23].

Many authors are now paying attention to a variety of possible applications of MIEX. Ates and Incetan, (2013) [37] conducted a study in which raw water from Camlidere and Kesikkopru were treated. The contaminations in raw waters tested were dissolved organic carbon (DOC), sulfate ( $\text{SO}_4^{2-}$ ) and bicarbonates. The resulting treatment trends in terms of removal affinity observed from MIEX were  $\text{SO}_4^{2-} > \text{DOC} > \text{Bicarbonates}$  for Camlidere water and  $\text{DOC} > \text{SO}_4^{2-} > \text{bicarbonates}$  for Kesikkopru water. A comparative study was also performed by [38], in which a performance comparison was made between MIEX bicarbonate-form resin and MIEX chloride-form resin. The performance was similar for both types of

resins regarding DOC,  $\text{SO}_4^{2-}$ , UV absorbance. The only difference was that the removal efficiency of MIEX bicarbonate-form for bromide was better than MIEX chloride-form. Moreover, for both type of resins, the removal efficiencies decrease with a number of regeneration cycles. Performance of four different types of resins i.e. MIEX, DOWEX-MSA, IRA-938 and DOWEX-11 were also studied for the simultaneous removal of NOM along with sulfates, nitrates and some pesticides [39].

Karpinska et al., (2013) [40] took a raw water sample from a water treatment plant on Douro River, having considerably high DOC in it. They revealed that on treatment with MIEX of an optimal resin dose of  $15 \text{ mL L}^{-1}$ , the DOC removal efficiency exceeds 90% within 10 min of contact time. Another study based on drinking water was conducted by Kitis et al., (2007) [36]. Water samples from different potable water treatment plants were collected in Istanbul. Results revealed that with optimal contact time and resin dose complete removal of DOC can be achieved. A study also suggested that a combination of MIEX and alum for the removal of DOC from water was more effective than using alum alone for the treatment process. It also suggested that a broader range of Total organic carbon (TOC), UV absorbance and DBPs including THMs can be lowered if MIEX and alum were used in proper combination [41].

### 3.3 Suspended Type Ion Exchange

Conventionally packed bed columns have been used for ion exchange process, but often encountered operational challenges such as biofilm formation and clogging under more viscous and turbid water [42,43]. These issues have been minimized in the newly developed ion exchange process called suspended ion exchange (SIX). This innovative process was developed by PWN Technologies in the Netherlands [23] for the removal of organic carbon from surface water. One major benefit of using SIX is that this process can use most commercially available resins. SIX is also a single pass plug flow

system, hence, the probability of fouling decreases with more stable and greater adsorption kinetics. The performances are greatly dependent on the type of resin selected for the operation. With different resin type and NOM concentration in treated, the resin concentration can vary from 4-20 ml resin/l [23]. These resins were allowed to pass through plug flow reactor after which they were separated and collected through lamella settler and sent for regeneration. The allowed contact time was only 10-30 seconds. Another advantage of SIX is the short contact time that minimizes resin blinding effect which is biofilm formation on the resin surface.

There are not many studies available on SIX treatment process. The first water treatment plant based on SIX processes (WTP, Andijk) with a capacity of  $5500 \text{ m}^3\text{h}^{-1}$  was operated in 2014 [23]. All commercially available resins can be employed in this plant, but WTP Andijk preferred acrylic gelular anion resin which is strongly basic in nature. Numerous factors were involved like sedimentation properties, economic feasibility, adsorption and desorption isotherm in the selection of this resin. For high quality effluent and better NOM removal efficiency the resin loading and contact time were found to be 12-15 ml and 20-30 min respectively. Another study conducted by [44] utilized SIX with in-line coagulation followed by ceramic membrane

filtration. Authors found that SIX potentially removes low molecular weight fraction and while high molecular weight fractions were significantly removed through coagulation.

### 3.4. Other Treatment Technologies

Numerous authors studied ion exchangers in combination with other treatment technologies. Some of the examples include the MIEX process along with ultra and nano-filtration [44], alum for DOC removal [41] and coagulation [32,35]. Moreover, when the FIX was used in combination with nanofiltration membrane, it resulted in higher biomass densities (400%) and 20% lesser iron deposition on membrane [30]. The impact of MIEX as a pretreatment was investigated with ozonation in achieving disinfection goals while restricting bromate and chlorinated DBPs formation. Three raw water samples from the San Francisco Bay Delta were collected which contains a different concentration of bromide and TOC. The results clearly show the difference MIEX made in the process. 41-68% of TOC was removed by MIEX alone from raw water compared to 12-44% for alum. Bromide concentration was also reduced by 20-50% by MIEX [45]. This also significantly reduced the amount of ozone for the treatment process of all water samples hence, can make overall treatment more economic.

**Table 1:** A brief collection of experimental work with various ion exchangers for NOM treatment.

Ion Exchange Process	Physiochemical Properties of Water Samples	Treated Water Composition/ Efficiency
Suspended Ion Exchange (SIX) Process [43]	Water samples were taken from Suwannee River, NOM = 8.33 mg/l Fulvic Acid = 8.85 mg/l Humic Acid = 7.40 mg/l	TOC in NOM, fulvic acid and humic acid was reduced by >90%.
Suspended Ion Exchange Process [44]	Synthetic waters were produced for each test. <b>Test 1:</b> DOC = 1.1 mg/l SUVA = 4.2 L/(mg.m) <b>Test 2:</b> DOC = 4.0 mg/l SUVA = 3.7 L/(mg.m) <b>Test 3:</b> DOC = 6 mg/l SUVA = 5.7	<b>Test 1:</b> DOC = 58% SUVA = 68% <b>Test 2:</b> DOC = 62% SUVA = 60% <b>Test 3:</b> DOC = 38 % SUVA = 42 %

Ion Exchange Process	Physiochemical Properties of Water Samples	Treated Water Composition/ Efficiency
Magnetic Ion Exchange Process (MIEX) [45]	Three water sample collected from San Francisco Bay Delta. <b>North Bay Aqueduct (NBA):</b> TOC = 3.7 mg/l UV254 (cm <sup>-1</sup> ) = 0.113 <b>South Bay Aqueduct (SBA):</b> TOC = 2.4 mg/l UV254 (cm <sup>-1</sup> ) = 0.071 <b>Lake Campbell Water:</b> TOC = 8.5 mg/l UV254 (cm <sup>-1</sup> ) = 0.256	<b>North Bay Aqueduct (NBA):</b> <b>MIEX-Cl</b> TOC = 2.2 mg/l UV254 (cm <sup>-1</sup> ) = 0.058 <b>MIEX-HCO<sub>3</sub></b> TOC = 2.2 mg/l UV254 (cm <sup>-1</sup> ) = 0.053 <b>South Bay Aqueduct (SBA):</b> <b>MIEX-Cl</b> TOC = 1.1 mg/l UV254 (cm <sup>-1</sup> ) = 0.024 <b>MIEX-HCO<sub>3</sub></b> TOC = 1.2 mg/l UV254 (cm <sup>-1</sup> ) = 0.021 <b>Lake Campbell Water:</b> <b>MIEX-Cl</b> (No data available) <b>MIEX-HCO<sub>3</sub></b> TOC = 2.8 mg/l UV254 (cm <sup>-1</sup> ) = 0.033
Magnetic Ion Exchange Process (MIEX) [46]	Three type of sample waters were analysed. <b>Barcombe:</b> DOC = 9.6 mg/l UV254 = 16.5 m <sup>-1</sup> Turbidity = 13.1 NTU <b>Draycote:</b> DOC = 10.7 mg/l UV254 = 13.9 m <sup>-1</sup> Turbidity = 1.4 NTU <b>Albert:</b> DOC = 9.4 mg/l UV254 = 60.1 m <sup>-1</sup> Turbidity = 1.8 NTU	DOC removal efficiency for- Barcombe = 56% Draycote = 33% Albert = 25%
Magnetic Ion Exchange Process (MIEX) [36]	Five potable water samples were analysed: <b>Elmali:</b> DOC = 4.3 mg/l SUVA <sub>254</sub> (l/mg DOC-m) = 5.11 <b>B.Ceknece:</b> DOC = 3.1 mg/l SUVA <sub>254</sub> (l/mg DOC-m) = 2.71 Turbidity = 1.4 <b>Omerli:</b> DOC = 2.6 mg/l SUVA <sub>254</sub> (l/mg DOC-m) = 3.92 Turbidity = 4.2 <b>Ikıtelı:</b> DOC = 3.1 mg/l SUVA <sub>254</sub> (l/mg DOC-m) = 3.45 Turbidity = 1.0	<b>Elmali:</b> DOC = 1.2 mg/l SUVA <sub>254</sub> (l/mg DOC-m) = 1.9 <b>B.Ceknece:</b> DOC = 1.3 mg/l UV254 (cm <sup>-1</sup> ) = 1.1 <b>Omerli:</b> DOC = 1.2 mg/l SUVA <sub>254</sub> (l/mg DOC-m) = 1.0 <b>Ikıtelı:</b> DOC = 0.8 mg/l SUVA <sub>254</sub> (l/mg DOC-m) = 1.3 <b>Kagıthane:</b> DOC = 1.1 mg/l SUVA <sub>254</sub> (l/mg DOC-m) = 1.4

Ion Exchange Process	Physiochemical Properties of Water Samples	Treated Water Composition/ Efficiency
	<b>Kagithane:</b> DOC = 2.8 mg/l SUVA <sub>254</sub> (l/mg DOC-m) = 3.86 Turbidity (NTU) = 21.5	
Combined Magnetic Ion Exchange and Cation Exchange [47]	Four water samples were taken from United States, <b>Cedar Key:</b> DOC (mg/l) = 6.1 SUVA <sub>254</sub> (l/mg.m) = 3.7 UVA <sub>254</sub> (cm <sup>-1</sup> ) = 0.224 <b>Yankeetown:</b> DOC (mg/l) = 2.9 SUVA <sub>254</sub> (l/mg.m) = 2.8 UVA <sub>254</sub> (cm <sup>-1</sup> ) = 0.081 <b>Palm Spring:</b> DOC (mg/l) = 9.5 SUVA <sub>254</sub> (l/mg.m) = 3.8 UVA <sub>254</sub> (cm <sup>-1</sup> ) = 0.370 <b>North Miami Beach (Nanofiltration Concentrate):</b> DOC (mg/l) = 33.2 SUVA <sub>254</sub> (l/mg.m) = 3.2 UVA <sub>254</sub> (cm <sup>-1</sup> ) = 1.04	<b>Cedar Key:</b> DOC = 67% UVA <sub>254</sub> = 64-90% <b>Yankeetown:</b> DOC = 77% UVA <sub>254</sub> = 64-90% <b>Palm Spring:</b> DOC = 85% UVA <sub>254</sub> = 64-90% <b>North Miami Beach:</b> DOC = 77% UVA <sub>254</sub> = 64-90%
MIEX; DOWEX-MSA; DOWEX-11 and IRA-938 [39]	Samples were from Villejean/Rennes drinking water treatment plant. DOC (mg/l) = 2.1-4.8 UV <sub>254</sub> (cm <sup>-1</sup> ) = 0.021-0.1 SUVA (mgCC L <sup>-1</sup> ) = 1-2	DOC concentration of treated water by all types of resins were found to be in the range of 1-2 mg/l.
MIEX combined with Nanofiltration (NE70 and NE90) [31]	Water samples were taken from a water treatment plant located in Changwon city, Korea. DOC (mg/l) = 2.5 UVA (cm <sup>-1</sup> ) = 2.56 SUVA (L/mg.m) = 0.021 Turbidity = 10 NTU	DOC (mg/l) = 1.28 SUVA (L/mg.m) = 0.003 UVA (cm <sup>-1</sup> ) = 0.001

#### 4. Regeneration of Resins

To make ion exchange process economically feasible for the treatment of NOM from ground and surface water, it is of utmost desire to regenerate the used resins. Not many studies focussed on ion exchange treatment along with regeneration of resins used. Experiments were performed for the regeneration of MIEX resin through sodium carbonate

when the resins were saturated with sulfate. MIEX resin in chlorine form was tested. This was first saturated with sodium sulfate solution (10x), the resulting resin formed was MIEX-SO<sub>4</sub>. This was further saturated with sodium bicarbonate solution (10x) to convert it into MIEX-SO<sub>4</sub>-HCO<sub>3</sub>. Both MIEX-SO<sub>4</sub> and MIEX-SO<sub>4</sub>-HCO<sub>3</sub> showed similar performance for first few regeneration cycles representing that sodium bicarbonate can potentially regenerate exhaus-

ted anion exchange resins [48]. In another study, performances of regenerated resins were analysed based on the regeneration procedure. Two different regeneration methods were employed i.e. acid/base regeneration and brine regeneration [24]. MIEX-Na resin was regenerated with both acid/base and brine solution and tested on the removal of DOC and UV<sub>254</sub>. After three regeneration cycles, the DOC and UV<sub>254</sub> removal efficiencies increased by 8-16%. Similarly, on hardness test, a dramatic behaviour was observed. The hardness removal efficiency dropped from 66% to 52% when treated after regeneration with brine solution and from 52% to <10% with acid/base solution. The reason behind this awkward behaviour was provided as carboxylic acid functional groups on resins have a better affinity towards the hydrogen, sodium and calcium.

In Andijk, Lewatit VPOC 1017 resin was regenerated during large scale SIX process. The regeneration was done with chlorine ion and the average salt required for regeneration was 0.05-0.2 kg.m<sup>3</sup>. In addition, no significant difference was observed in the DOC removal efficiency before and after regeneration [23]. Another recent example of using brine solution for regeneration provided by [49]. Regeneration of Nano-Resin attached to the membrane surface was achieved with 2.0 M brine solution. In this case as well no significant deterioration in removal efficiencies was observed even after a considerably high number of regeneration cycles.

## 5. Conclusion

In the current review study, most of the recent studies on the natural organic matter (NOM) removal was discussed. In the majority of the work, ion exchangers were used as pretreatment unit and hence, the quality and composition on contaminants present in wastewater play an important role in the selection of resin type. As NOM have the tendency to further produce disinfection by-products (DBPs), ion exchangers can be a best possible option for their removal.

One of the major challenge water treatment

industries are facing is NOM. If NOM can be removed in earlier stages of surface water treatment, the performance and efficiency of following treatment units can be increased to many folds. Magnetic ion exchange resins (MIEX) can be used in a slurry mode which widens the range of its possible applications (for example in different design reactors) with simple separation and regeneration techniques. Furthermore, the high stability with no pretreatment makes MIEX a potential option for numerous industrial applications.

Regeneration, an important aspect in ion exchange process without which it will be very challenging to make the process economically viable. Despite its significance, very few detailed studies were performed. It is highly desirable to conduct more research on a variety of regeneration techniques. This will not only converge the gap for regeneration studies but will also enhance the possibility of developing novel resins for NOM removal from all kinds of waters.

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