



## Review

# An overview of the uses of high performance size exclusion chromatography (HPSEC) in the characterization of natural organic matter (NOM) in potable water, and ion-exchange applications



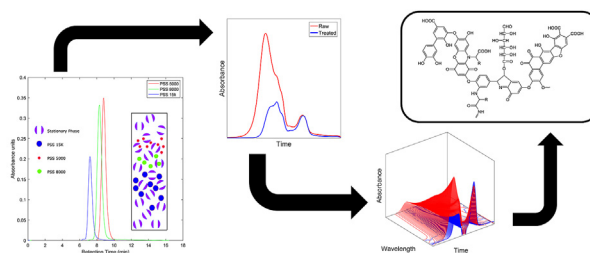
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## HIGHLIGHTS

- Summary of applications of HPSEC for NOM removal are summarized.
- Size and wavelength considerations are discussed.
- Characterization of NOM based on functionalization and acidity.
- DBP can be traced to UV absorbing moieties contained in NOM.
- Ion-exchange used for holistic removal of NOM moieties.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Natural organic matter (NOM) constitutes the terrestrial and aquatic sources of organic plant like material found in water bodies. As of recently, an ever-increasing amount of effort is being put towards developing better ways of unraveling the heterogeneous nature of NOM. This is important as NOM is responsible for a wide variety of both direct and indirect effects: ranging from aesthetic concerns related to taste and odor, to issues related to disinfection by-product formation and metal mobility. A better understanding of NOM can also provide a better appreciation for treatment design; lending a further understanding of potable water treatment impacts on specific fractions and constituents of NOM. The use of high performance size-exclusion chromatography has shown a growing promise in its various applications for NOM characterization, through the ability to partition ultraviolet absorbing moieties into ill-defined groups of humic acids, hydrolysates of humics, and low molecular weight acids. HPSEC also has the ability of simultaneously measuring absorbance in the UV–visible range (200–350 nm); further providing a spectroscopic fingerprint that is simply unavailable using surrogate measurements of NOM, such as total organic carbon (TOC), ultraviolet absorbance at 254 nm ( $UV_{254}$ ), excitation-emission matrices (EEM), and specific ultraviolet absorbance at 254 nm ( $SUVA_{254}$ ). This review mainly focuses on the use of HPSEC in the characterization of NOM in a potable water setting, with an additional focus on strong-base ion-exchangers specifically targeted for NOM constituents.

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## 1. Introduction

Natural Organic Matter (NOM) are some of the most diverse components found within natural ecosystems. From the reference point of aquatic ecosystems, NOM can be autochthonous (aquatic) or allochthonous (terrestrial) sourced. The components, and therefore the properties of NOM are source specific, which lends to the difficulty in predicting their behavior and fate within an ecosystem. NOM removal is a prime consideration for water treatment operators, being responsible for a wide array of water quality issues including taste and odour; and is responsible for reactions involving metal-chelation, redox, and the formation of disinfection by-products (Leenheer and Croué, 2003; Krasner et al., 2006; Woo et al., 2002; Boorman, 1999). Researchers and engineers are looking for new tools and methods to better describe NOM, as a better understanding will ultimately lead to a better prediction of their effects and removal.

To date, tools such as ultraviolet absorbance at 254 nm (UVA<sub>254</sub>) and total organic carbon (TOC) have acted as the most commonly used surrogate parameters for NOM (Matilainen et al., 2011). UVA<sub>254</sub> provides an estimation of the aromaticity of the sample – which is linked to reactivity – while TOC provides an estimation of the total concentration of carbon present in NOM (Hur et al., 2006). Both methods are readily available in most analytical laboratories, and UVA<sub>254</sub> has been developed in portable form factors (Beauchamp et al., 2018). The problem with these parameters is that they are one-dimensional, in that they do not encompass the heterogeneity of NOM as they only measure one variable. The use of parameters such as SUVA<sub>254</sub> – defined as UVA<sub>254</sub> divided by the TOC – has to an extent partially solved this problem, by using both variables to test for the aromaticity per unit gram of carbon (Hua et al., 2015; Sillanpää et al., 2015). Researchers however, have a wider variety of resources available at their disposal, and do not have to be limited to portability or availability. Researchers have utilized chromatographic methods to partition NOM into apparent molecular weight (AMW) fractions, and have tested various pre-treatment methods to provide a better understanding of the underlying effects of their treatment on NOM composition (Kac et al., 2017; Wang et al., 2013a, 2016a; Black and Bérubé, 2014; Świetlik et al., 2004). The use of these techniques has expanded to uses in preventing membrane fouling (Yamamura et al., 2014), tracing metal-colloid associations (Neubauer et al., 2013), and monitoring

the effects of alternative disinfectants (Wang et al., 2015).

In a potable water setting, a pressing concern is the formation of disinfection by-products (DBP) – a by-product of the disinfection process that is usually formed at the end of the conventional treatment train. These DBP are correlated to their precursor products, which in most scenarios are a function of their local NOM (Bazri et al., 2016). In order to trace the presence of certain NOM components with their corresponding DBP product, a better appreciation of the nature and scope of those components is required. The use of multi-wavelength detectors, such as photodiode arrays, provides a wealth of information regarding the functionality, reactivity, and presence of these components. Drikas et al. (2011) applied high performance size exclusion chromatography (HPSEC) in a long term investigation of water treatment operation; including tracing a relationship between AMW and hydrophobic acids (HPO) concentration. This information has been used to make predictive outcomes in NOM reactivity, that would have otherwise been unavailable. Awad et al. (2016) utilized HPSEC peak identification techniques to model the formation of trihalomethane (THM) species – a particular type of DBP. The results showed promise, but included the use of sets of arbitrary coefficients, and limited its use of information to UVA<sub>260</sub>. Greater use of the multi-spectral properties provided by HPSEC will allow for a more encompassing appreciation of NOM reactivity, and a better tool to model its outcomes on potable water systems.

## 2. Literature review

### 2.1. Natural Organic Matter characterization

Natural Organic Matter (NOM) plays an important role in the general ecological makeup of ecological and engineered systems. NOM is a ubiquitous class of complex aquatic organic molecules that are ultimately sourced from the breakdown of plant residues, lignin, cellulose and other complex macromolecules from allochthonous (terrestrial) and/or autochthonous (marine) sources (Aliverti et al., 2011; Edzwald and Assoc., 2011; Aiken et al., 1992). As a result of its ambiguous composition NOM does not have a set defined size distribution, functional group composition, acid-base functionality, or chelating potential. This makes it extremely difficult to model the behavior of NOM in regards to its impact in potable water treatment processes and distribution systems. This

chapter aims to provide an overview on the ways in which researchers have categorized NOM through physical and/or analytical means.

NOM removal is a prime consideration for water treatment operators, being responsible for a wide array of water quality issues including taste and odour; and is responsible for reactions involving metal-chelation, redox, and the formation of disinfection by-products (Leenheer and Croué, 2003; Krasner et al., 2006; Woo et al., 2002; Boorman, 1999). As a result, the removal of NOM has been extensively explored using different chemical and physical methods, including well described methods such as carbon-based adsorbents (Velten et al., 2011; Çapar and Yetiş, 2001), oxidation (Winter et al., 2016), and coagulation (Xu et al., 2016; Zhan et al., 2011); as well as some advanced forms of treatment such as membrane filtration (Yunos et al., 2014), ion-exchange (Bazri et al., 2016), electro-oxidation (EO) (Fu et al., 2007), electro-coagulation (EC) (Chellam and Sari, 2016; Mohora et al., 2012, 2014; Dubrawski et al., 2013), modified nanotechnologies (Fathizadeh et al., 2011; Chow et al., 2009), and biofiltration (Black and Bérubé, 2014; Treguer et al., 2010; Seredyńska-Sobecka et al., 2006; Hozalski et al., 1999). NOM has a significant impact on engineered potable water treatment systems by contributing to membrane fouling (Cheng et al., 2016; Wang et al., 2016a; Winter et al., 2016; Linares et al., 2011), trihalomethane formation (Bazri et al., 2016; Pifer and Fairey, 2012; Krasner et al., 2006; Kitis et al., 2002), metal mobility (Winning et al., 2017; Akaighe et al., 2013; Schmitt et al., 2003; Leenheer and Croué, 2003), aesthetic concerns (Matilainen et al., 2010), and corrosion (Elfström Broo et al., 1999). A thorough explanation on the remedies for issues concerning NOM by treatment with ion-exchange (IX) can be found in Section 5.

## 2.2. Natural Organic Matter functionalization

Researchers have used various analytical means to better describe NOM composition. Table 1 outlines the elemental, and functional composition of 1 two major components of NOM, Fulvic (FA) and Humic Acids (HA), collectively known as Humic Substances (HS). These acids are complex polyprotic acids, with the main operational definition between them being: HA are insoluble at low pH levels, while fulvic acids are soluble at all pH levels (Stevenson, 1994). Research carried out by Vance and David (1991)

**Table 1**

Common structural characteristics of humic and fulvic acids obtained using nuclear magnetic resonance (NMR)/fourier transform infrared spectroscopy (FTIR) analysis, adapted from (Sillanpää et al., 2015; Li et al., 2011; Xing et al., 2012; Matilainen et al., 2010; Her et al., 2008; Lankes et al., 2008; Mao et al., 2007a; Hay and Myneni, 2007; Kanokkantung et al., 2006; Kim and Yu, 2007; Kim et al., 2006; Leenheer and Croué, 2003; Chen et al., 2002; Wong et al., 2002; Czeżuga and Orowska, 2001; McCreary and Snoeyink, 1980).

Elemental Composition (%)	Humic Acids	Fulvic Acids
Carbon	50–60	40–50
Hydrogen	4–6	4–6
Oxygen	30–35	44–50
Nitrogen	2–4	1–3
Sulfur	1–2	0–2
Functionality (%)		
Aliphatic	74	86
Aromatic	24	16
Carboxyl	14–45	58–65
Phenol	10–38	9–19
Alcohol	13–15	11–16
Carbonyl	4–23	4–11
Methoyl	1–5	1–2

also outlines the characteristic pKa values, obtained via potentiometric titration, that distinguishes HA and FA; obtained from a northern hardwood forest floor in Central Maine, USA. Table 1 also lays out a series of other notable differences, namely humic acids have a larger proportion of aromatic groups and phenolic groups as a percentage of total level of functionality.

The proportions of these compounds in water bodies varies from source to source. Bolto et al. (2002b) found that fulvic and humic acids make up approximately 40% and 10% of the water body, respectively; while in other studies these two acids together constitute anywhere between 40 and 67% (Golea et al., 2017; Chen et al., 2016; Vik and Eikebrokk, 1988). These acidic components are important for water treatment operators and engineers alike because they are considered precursors products towards the formation of disinfection by-products (DBP) (Bond et al., 2010; Li et al., 2011; Richardson et al., 2007a; Ichihashi et al., 1999).

## 2.3. Natural Organic Matter fractionation

NOM has further been characterized through the classification of water components based on acidity and hydrophilicity (Matilainen et al., 2011; Leenheer and Croué, 2003). These have become collectively known as *NOM fractionation*, which is the separation of NOM using a combination of strong/weak resins and variations in pH. Earlier adaptations of fractionation were used as earlier as 1981 by Leenheer and Croué (2003), and further popularized in the work by Aiken et al. (1992). It is still very common practice to use a combination of XAD-8/-4 resins (Lee et al., 2004; Piccolo et al., 2002; Maurice et al., 2002) in series, as well as XAD-8/-3 (Li et al., 2017; Goslan et al., 2002; Malcolm and MacCarthy, 1992). Adaptions have been made to fractionate with disposable solid-phase extraction (SPE) cartridges, with reverse-phase sorbents (Goss et al., 2017; Williams et al., 2016; Ratpukdi and Khan, 2008; Croué, 2004) as well as C-18 sorbents (Mergen et al., 2008; Caron et al., 2014; Kim et al., 2006; Egeberg and Alberts, 2002). Table 2 lists the commonly fractionated components of NOM, as well the associated molecular weight (MW) ranges and functionality of the fractions. These fractions are important as they provide further classification into the fate and reactivity of certain components of NOM.

Similar to the proportionality of FA and HA, water bodies have different fractional compositions. Hydrophobic components are listed as the most dominate fraction in most water bodies (Wang et al., 2009; Chiang et al., 2009; Fabris et al., 2008a), citing numbers as high as 66% in the work by Golea et al. (2017). Of course, there exists deviations from the established rule (Xu et al., 2007), such as in the work carried out by Kim et al. (2006) who noted a 55–70% hydrophilic composition of the Han River located in South Korea. In addition to the information provided in Table 2, hydrophobic components tend to be resistant to microbial degradation, rich in extended conjugation (i.e., phenols, tanins, lignins), and have a relatively high molecular weight (HMW) relative to their hydrophilic counterparts (Matilainen et al., 2011; Lacorte et al., 2003). Their low biodegradability due to their insolubility makes them difficult to degrade naturally, thereby making the cellulose polymers – a mainly HPO fraction – the main contributor to color in water (Welker et al., 2015). Hydrophilics on the other hand, are readily biodegradable low molecular weight (LMW) components, including protein-like and complex polysaccharides such as hemicellulose (Matilainen et al., 2011; Pranovich et al., 2005). The functionality, size-distribution, and hydrophilicity all have an important impact on the fate of these components in engineered systems. In terms of DBP formation, the literature is very much mixed on this topic, with the dominant fraction between hydrophobic acids (HPOA) (Hu et al., 2015; Lin et al., 2014; Gough et al.,

**Table 2**

Component analysis of various water constituents based on hydrophilicity and acidity (Ratpukdi and Khan, 2008; Świetlik et al., 2004; Ritchie and Perdue, 2003; Kawasaki et al., 2011; Panyapinyopol et al., 2005; Peuravuori et al., 1997; Edzwald and Assoc., 2011; Aiken et al., 1992; Chen et al., 2003; Leenheer and Croué, 2003).

Fraction	Components	Functional Groups	Molecular Weight (Da)
Hydrophobic Base	Proteins, Amino Acids	Aromatic amines, high MW alkyl amines	100–70,000
Hydrophobic Acid	Humic and Fulvic Acids	Aromatic acids, high MW alkyl mono-, di-carboxylic acids	450–1000
Hydrophobic Neutral	Hydrocarbons	Aldehydes, high MW methyl ketones, alkyl alcohols, ethers, furans	100–70,000
Hydrophilic Base	Proteins, Amino Acids	Purines, pyrimidines, low MW alkyl amines	100–1000
Hydrophilic Acid	Fatty Acids	Hydroxyl acids, sugars, sulfonics, high MW alkyl mono-, di-carboxylic acids	250–850
Hydrophilic Neutral	Polysaccharides, aldehydes, ketones, low MW alkyl alcohols	Polysaccharides, aldehydes, ketones, low MW alkyl alcohols	120–900

2012; Han and Ma, 2012; Banchapattanasakda et al., 2011; Zhang et al., 2009; Zhao et al., 2009; Liu et al., 2009; Chen and Westerhoff, 2010; Soh et al., 2008; Wei et al., 2008; Chow et al., 2005; Leenheer and Croué, 2003; Croué et al., 2000; Singer, 1999), and hydrophilic acids (HPIA) (Musikavong et al., 2013; Goss and Gorczyca, 2012; Bond et al., 2010; Lin et al., 2010; Smith and Kamal, 2009; Panyapinyopol et al., 2005; Kitis et al., 2002; Croué et al., 1999). As outlined in Table 2, HPO are synonymous with HS (Lin et al., 2014; Lin and Xing, 2008; Sharp et al., 2005), therefore the potential for both to form THMs is well established. In the recent work of Kim et al. (2017), organic acids were cited as being responsible for a higher trihalomethane formation potential (THMfp) and haloacetic acid formation potential (HAAfp) – both known carcinogen by-products. Zhao et al. (2013) additionally cited that hydrophilic neutrals (HPIN) were a significant factor as well; at least for the water tested which was retrieved from the Songua River, a water noted by the authors as having relatively high dissolved organic carbon (DOC) ( $4.1 \text{ mg mL}^{-1}$ ) typical of Chinese waters. A similar finding was made by Chu et al. (2011) who noted HPN were traced to the formation of the DBP dichloroacetamide.

Fractionation has had a significant impact on the way in which treatment methods have been used to target NOM fractions. In order to predict the effect of NOM fractions, treatments such as ozonation have been applied to characteristically reshape the nature of NOM itself. The literature which suggests HPI groups contribute most to THM and HAA formation attribute this relation to the presence of carboxylic groups listed in Table 2 (Bond et al., 2010). Ozone has been applied to convert 50% of the proportion of NOM to 20% non-humic NOM, and have found the formation of non-humic acids, carbohydrates, lipids, and amino acids all had lower HAAfp and THMfp (Karnik et al., 2005). When Świetlik et al. (2004) applied ozone, they investigated the resulting excitation-emission matrices (EEM) and found HPIN and HPOA were the main precursors to carboxylic groups; while the HPIA fraction – similar to the findings of Zhao et al. (2013) and Chu et al. (2011) – was the main precursor for dichloroacetamide formation. Electrocoagulation has been found to specifically target aromatics, and HMW components of NOM; thereby discriminating HPO NOM that contribute most to DBP formation (Chellam and Sari, 2016; Sillanpää et al., 2015; Särkkä et al., 2015; Dubrawski et al., 2013). The work of Wang et al. (2013b) found a similar relation with HPO NOM with their implementation of enhanced coagulation. They did however note their LMW HPI NOM had a higher HAAfp, as compared to their HPO fraction which had a higher THMfp (Wang et al., 2013b). The presence of HPO can also impact other treatment units, as displayed by Cheng et al. (2016) who applied ozone to reduce the impact of the HPO fraction on irreversible membrane fouling. The HPO fraction – notably, humics and alginates – have been demonstrated as the main contributor to all forms of membrane fouling (Jermann et al., 2007). Research in membrane fouling

has noted that LMW hydrophilic neutral (HPIN) was the major component of nanofiltration (NF) fouling (Carroll et al., 2000). The discrepancy between these conclusions can be attributed to different mechanisms of membrane fouling (Howe and Clark, 2002), which is due to the initial conditions of the source water used. Research has also suggested that the removal of DOC – a predictor of THM formation – can be impacted by the HPO fraction of NOM. As investigated in the work of Mergen et al. (2008), DOC removal by ion-exchange (IX) was affected by the concentration of HPO in the 3 water sources studied. The authors noted the large MW of HPO may have impeded the adsorption mechanism of IX through steric effects. It was also suggested that HPI NOM were poorly removed by IX, as HPI components such as carbohydrates have poor electron density. This mechanism can also affect the sorption of HPO, as Egeberg et al. (2002) suggested, as the hydrophobicity of NOM is pH dependent – at least for the range of pH levels tested in their work (4.7–7.0). The impact of IX on the removal of NOM with other treatment methods is further discussed in Section 5. The following section will further illustrate the importance of monitoring and accounting for DBP formation in a wide variety of applications.

#### 2.4. Disinfection by-products formation

DBP are a known set of potentially carcinogenic, mutagenic, cytotoxic, genotoxic by-products that form as a result of water contact with a disinfectant such as chlorine (Plewa et al., 2017; How et al., 2017; Carter and Joll, 2017; Du et al., 2017; Richardson et al., 2007a). As a result, water treatment plants must abide by regulatory agencies that impose maximum acceptable concentrations (MAC) to their effluent discharges (Organization, 2017; Canada, 2017). This has led to the regulation of upwards of 20 different DBP worldwide (Richardson and Postigo, 2011), and a further classification of 600 compounds as new DBPs (Richardson and Postigo, 2015). As demonstrated in the work of Plewa et al. (2017), DBP such as THM and HAA are a small component of the overall cytotoxic effect on mammalian cells. This may be for numerous reasons, one of which is the lack of thorough toxicology and quantitative analysis have been carried out (Plewa et al., 2017; Muellner et al., 2007; Richardson et al., 2007b). Another reason is due to the lack of classification of existing total organic halogen (TOX) by-products. This problem was initially addressed by Krasner et al. (1996) who noted 50% of TOX formation was not accounted for, or regulated. This issue is very much a current one, as demonstrated in two papers carried out by Kristiana et al. (2009) and Kristiana et al. (2015); among others similar investigations (Wert and Rosario-Ortiz, 2013; Li et al., 2012; Fang et al., 2010). It is estimated in the work of Kristiana et al. (2015) that <20% of TOX by-products are accounted for, while the rest are still yet to be classified (Liu et al., 2017). The mechanisms for the formation of



DBP from their precursor products is very much a “black box” as described in the literature due to the sheer complexity of the reaction mechanism that is expected to take place (Bougeard et al., 2010; Chen and Westerhoff, 2010; Goslan et al., 2009). Mechanisms have been proposed to take place, such as in the work of Reckhow et al. (1985) and as early as 1974 in the work produced by Rook (1974) (Rook, 1977). Current investigation into modeling DBP formation with an assortment of reaction pathways had been carried out by Zhu and Zhang (2016), who were able to trace the formation of chlorinated, brominated, and iodated by-products due to chloramination disinfection with good agreement.

It has been suggested in the earlier work of Reckhow et al. (1990) that electron-rich aromatics and electron withdrawing groups help to facilitate the reaction mechanism by activation of the aromatic ring; thereby FA and HA are heavily associated with this phenomenon in the literature. Reports by Mohora et al. (2014) list the contribution from FA to be responsible for 41% of all THM formation, and 21% of all HAA formation. Investigations have been carried out which aim to find the connection between these properties and other common water characteristics and products. As an example, the work carried out by Wang et al. (2015) drew parallels between functional composition of NOM, and the formation of DBP (Singer, 1999). It was noted in the work of (Wang et al., 2015) that phenolic and hydroxyl groups, very characteristic of fulvic acids in their work and in literature, was the main contributor to the DBP dichloroacetic acid (DCAA) formation. The authors also highlighted the formation of another DBP, chloroform  $\text{CHCl}_3$ , was enhanced in the presence of aromatic, methyl, and guaiacyl lignin groups (Wang et al., 2015). This was also corroborated in the work of Kim and Yu (2007) that found a direct correlation with phenolic content of HS and THM and HAA formation. Researchers have investigated the presence of lignin phenols on DBP formation, as lignin derived NOM is responsible for the bulk of the terrestrial NOM pool (Ertel et al., 1984). In the works of Pellerin et al. (2010) and Hua et al. (2015), lignin derived NOM surrogates were heavily correlated with higher DBP yields if they were phenolic derived. In Hua et al. (2015) lignin derived NOM was shown to be responsible for higher DBP yields, but less TOX formation than humic substances. C-DBP and N-DBP formation has been associated with other precursor NOM products, including;  $\beta$ -dicarbonyl acids, amino acids,  $\alpha$ -amino acids, purines, and amines (Liu et al., 2014).

### 3. Absorbances indices

Absorbance has been extensively used as a parameter for determining various components that exist in water bodies. The simply direct correlation between the concentration of a compound and the amount of light it absorbs, lends to the reality that the study of spectroscopy is one of the foundations of analytical chemical analysis. This allows for spectroscopy to act as a practical tool in the analysis of water and wastewater, to monitor an assortment of components that can be presumed to exist. The difficulty in measuring environmental samples stems from in the inability to account for molar extinction coefficients ( $\epsilon$ ) of multiple water components, which relates to the Beer-Lambert Law,  $A = \epsilon l C$ . Where  $A$  is the absorbance;  $\epsilon$  is the molar extinction coefficient;  $C$  is the concentration; and  $l$  is the path length. Weighted linear regressive methods, or inverse least-squares methods have been used to resolve multiple peaks; however, these methods usually require the use of reference standards (Brestrich et al., 2018). The work by Francisco et al. (2016) traced the concentration of apramycin and vancomycin bioassays during kinetic studies, and utilized the inverse least-squares approach to resolve overlapping peaks in absorbance measurements. These methods require the use of initial calibrations, which therefore make them impractical when

the aim is to distinguish components in environmental samples. Environmental samples, for the most part, contain components that are unfamiliar to researchers; therefore the use surrogate compounds – each with a characteristic  $\epsilon$  – are difficult to select competently. Researchers have however developed practical approaches through the use of surrogate absorbance indices, which usually relate to a group of components collectively, or to a predictive outcome. One of the most common indices used is  $\text{UVA}_{254}$ , used to predict aromaticity of a source sample. This wavelength is selected because three unique  $\pi \rightarrow \pi^*$  electronic excitations occurs for benzene, ethylenic, benzenoid and konjugierte bands occurring at 180–210, 250–295, and 275–330 nm respectively (Her et al., 2002; Karanfil et al., 2002). The predictive nature of  $\text{UVA}_{254}$  holds true for an assortment of tested waters (See Fig. 2), regardless of the fact that benzene does not exist as the sole chromophore in environmental waters. In the case of benzene, the value for  $\epsilon$  decreases drastically (hypochromic) with increasing wavelength, with values of 65,000, 8000 and 240 at wavelengths of 180, 200, and 254 nm respectively. It has been observed in the work by Her et al. (2008) that a large shift in  $\epsilon$  occurs at 230 nm for all 4 surrogate NOM samples tested; which included FA, HA, albumin, and asparagine standards. While the reasoning wasn't clear in the work by Her et al. (2008), this may be due to bathochromic (i.e. shift to a longer wavelength) shifts in the  $\epsilon$  values between 180 and 200 nm. We readily observe this in environmental water samples, as increased levels of conjugation – due to alkene or polyaromatic substitution – leads to bathochromic shifts of these absorption bands; thereby allowing them to be more readily observed by UV spectrophotometers ( $> 200$  nm). This has led to a wide-field of research particularly interested in the use of these UV indices to observe or predict water source characteristics. A summary of these indices can be found in Table 3.

Researchers have particularly made use of these indices by monitoring these parameters following various pre-treatment methods. An example of this is the use of differential absorbance, defined as the change in UV absorbance at 272 nm, with the formation of DBP. This work was initially popularized by Korshin et al. (1996), and has since then been carried out by researchers at the University of Washington, U.S. and the University of Catalonia, Italy. The University of Washington has published a series of papers over the years, correlating this parameter with DBP formation following chlorination (Yan et al., 2016; Korshin et al., 2002, 2009; Fabbicino and Korshin, 2005; Korshin, 2002; Li and Sun, 2001). The University of Catalonia have followed suit with collaborations with the University of Washington, by testing these parameters as well others such as differential fluorescence on various source waters, such as that of the Ancipa reservoir in Sicily, Italy (Roccaro et al., 2015; Roccaro and Vagliasindi, 2009; Roccaro et al., 2008b, a). A more recent implementation of this research is in the work carried out by Beauchamp et al. (2018) in adapting onto the work by Marcoux et al. (2017). The authors noted a strong linear correlation with values for UV absorbance, taken at 254 and 272 nm, and DBP concentrations for THM4 ( $\text{CHCl}_3$ ,  $\text{CHBr}_3$ ,  $\text{CHCl}_2\text{Br}$ ,  $\text{CHClBr}_2$ ) and HAA5 ( $\text{CHCl}_2\text{COOH}$ ,  $\text{CHCl}_3\text{COOH}$ ,  $\text{CHClCOOH}$ ,  $\text{CHBrCOOH}$ ,  $\text{CHBr}_2\text{COOH}$ ) ( $0.62\text{--}0.99 R^2$ ). The original work carried out by Marcoux et al. (2017) noted limitations in their analysis, namely, limiting their case-study to one season; however, an interesting take-away was that the majority of DBP monitored (THM, HAA, haloacetonitriles (HAN), halo ketones (HK)) were formed within the treatment train, and not in the reservoir or distribution system. This was due to the 2 sites of chlorination that were implemented within the treatment train, and the long (24 h) residence time of plant operation (Marcoux et al., 2017). The work of Golea et al. (2017) is another example of a long-term investigation built upon examining UV indices, where 30 treatment works were investigated over a 30-

**Table 3**

Summary of UVA indices used to predict NOM properties.

Parameter	Ref.
A <sub>210</sub>	Nitrate concentration, microbial activity, non-conjugated carboxylic groups (Gray et al., 2008; Whitehead and Cole, 2006; Cory and McKnight, 2005; Weishaar et al., 2003; Huang et al., 2016)
A <sub>230</sub>	Largest change in $\epsilon$ Her et al. (2008)
A <sub>254</sub>	Aromatics (Phetrak et al., 2016; Gough et al., 2014; Xing et al., 2012; Edzwald and Kaminski, 2009; Ko et al., 2009; Korshin et al., 2009; Edzwald and Kaminski, 2009, 2009; Gray et al., 2008; Fabris et al., 2008b; Pernitsky and Edzwald, 2006; Cory and McKnight, 2005; Karanfil et al., 2002; Zhou et al., 2000; Traina et al., 1990)
A <sub>280</sub>	Peptides and proteins Moffatt et al. (2000)
A <sub>280</sub>	Molecular weight and percent aromaticity (Peuravuori et al., 1997; Chin et al., 1994)
A <sub>270</sub>	Colored organic matter Stolpe and Hassellöv (2007)
A <sub>220</sub>	Carboxylic and aromatics Korshin et al. (2009)
A <sub>300</sub> , A <sub>340</sub> , A <sub>410</sub>	DOC concentration Baker et al. (2008)
A <sub>254</sub> , A <sub>272</sub> , A <sub>280</sub>	DOC aromatics (Baker et al., 2008; Karanfil et al., 2003)
SUVA <sub>254</sub>	Increase in DOM MW fractions, ratio of FA to HA, DOM composition (Hur et al., 2006; Hur and Schlautman, 2004; O'Loughlin and Chin, 2001)
SUVA <sub>254</sub>	Hydrophobics and aromatics, DBPfp (Sillanpää et al., 2015; Weishaar et al., 2003; Croué et al., 1999; Edzwald and Benschoten, 1990; Chin et al., 1994; Stewart and Wetzel, 1980)
SUVA <sub>254</sub> /SUVA <sub>280</sub>	Decrease in lignin derivatives and aromatics (Lewis et al., 2012; Zimmermann, 1990)
A <sub>254</sub> /204	FA adsorption by fractionation, increasing HA content, SUVA (Kreller et al., 2013; Hur et al., 2006)
A <sub>253</sub> /203	DBP formation, phenolic content (Kim and Yu, 2007; Hyung Kim and Yu, 2005; Korshin et al., 1997)
A <sub>254</sub> /210	Relative proportion of fulvic acids to biopolymers (Korshin et al., 2009; Her et al., 2008; Li et al., 2006; Stabenau and Zika, 2004)
A <sub>250</sub> /365	Aromaticity and M <sub>n</sub> Peuravuori et al. (1997)
A <sub>280</sub> /350	Aromaticity Croué et al. (1999)
A <sub>280</sub> /350, A <sub>203</sub> /254	Activation of phenolic groups (Korshin et al., 1997, 2009; Li et al., 2006)
A <sub>254</sub> /410, A <sub>250</sub> /365	Relative size of DOM and aromaticity (Helms et al., 2008; Baker et al., 2008)
A <sub>251</sub> /256A <sub>202</sub> -205	FA adsorption by fractionation (Her et al., 2004; Korshin et al., 1997)

month period in Scotland. Investigations lead to the conclusion that UVA<sub>254</sub> was the best overall indicator in predicting DBPfp (for HAA and THM) for both untreated and treated waters, with DOC following closely behind in terms of surrogate measurement. Fractionation for HPO and HPI components was done concurrently, with the results providing little benefit to discern trends in DBPfp. This work helps to illustrate that UVA<sub>254</sub> still remains a practical tool for water treatment operators, and not all surrogate measurements have equal effectiveness or practicality. An example of this is the use of UVA<sub>300</sub> as an extensive indicator for chromophoric DOM in oceanography studies (Pitta et al., 2017; Kowalczyk et al., 2013; Stedmon et al., 2000). Absorbance parameters outside of UV, such as A<sub>254</sub>/436 has been used to trace terrestrial vs aquatic sources of NOM (Jaffé et al., 2004; Battin, 1998). With this in mind, researchers have employed the use of multiple surrogate measurements to aid in the characterization of NOM, with the use of a parameter called Specific Ultraviolet Absorbance (SUVA).

### 3.1. Specific ultraviolet absorbance and excitation-emission matrices

The difficulty in the use surrogate of parameters to model NOM behaviour is the inability to correlate multiple physical and/or electronic effects. One of such tools used is specific ultraviolet absorbance (SUVA<sub>254</sub>) - which relates the UVA<sub>254</sub> to DOC concentration - which has been used extensively to predict DBP formation (Hua et al., 2015; Hong et al., 2017). Others have found little to no correlation, meaning these parameters are not fully effective in encompassing the full complexity and reactivity of NOM (Chow et al., 2008; Fram et al., 1999). These parameters on their own have found considerable use in predicting water characteristics - including DOC which for all intents and purposes, has acted as the best surrogate indicator for NOM applied in various applications

(Rajca, 2016; Schulz et al., 2016). SUVA aims to act as an indicator for electron rich moieties, which is predicated on the presence of electron rich aromatics (Kim and Yu, 2007). UVA<sub>254</sub> is important as a preliminary water quality parameter, because it provides an initial benchmark for hydrophilicity (Egeberg et al., 2002). In terms of DBP formation, high SUVA<sub>254</sub> is associated with HAA and THM formation (Bond et al., 2014; Hong et al., 2013; Chowdhury et al., 2009; Liang and Singer, 2003; Czezuga and Orowska, 2001; Reckhow et al., 1990) while trihalonitromethanes (TRNM) - an N-DBP - form at low SUVA<sub>254</sub> values (Hong et al., 2017; Hu et al., 2010).

Others have explored excitation-emission matrices (EEM) in order to map the changing behaviour of fluorophores of NOM following pre-treatment (Wang et al., 2016a; Caron et al., 2014; Li et al., 2013; Baghoth et al., 2011; Shao et al., 2014; Alberts et al., 2002). It has been adopted to target HA and FA moieties by monitoring specific regions at  $\lambda_{ex} = 270\text{--}440\text{ nm}$ ,  $\lambda_{em} = 380\text{--}550\text{ nm}$  (Kreller et al., 2013), and trace the binding of NOM to various metals (Lu et al., 2017). It has also been adopted to monitor soluble microbial products (SMP), as well as protein products (Li et al., 2013). The work of Huguet et al. (2009) used EEM on water sources of varying salinity, and was successfully able to classify estuarine waters based on rate of humification indices and autotrophic productivity. Phetrak et al. (2016) used total fluorescent intensity for selected regions, separated into tyrosine, tryptophan-like, FA, HA, and SMP. A cumulative EEM spectra was used by Yang et al. (2008) in order to identify aromatics and HA, then correlate that with dichloroacetic acid (DCAA), chloroform, dichloroacetoneitrile (DCAN) and TOX formation. EEM continues to be a productive tool used to by researchers, as they can be coupled with separation methods such as high-performance liquid chromatography (HPLC) (Phetrak et al., 2016; Halim et al., 2013).

#### 4. Instrumental analysis of NOM

Given the complexity of NOM, it comes to reason that researchers have developed powerful tools to help characterize it. Section helped to describe many spectroscopic methods that are used, with a selection of indices listed in Table 3, in addition to the use of EEM matrices; however, the presence of chromophores only provide a small outlook into the properties of NOM. Functional groups of NOM can be better understood through the use of Fourier-transformation infrared spectroscopy (FTIR), which examines the vibrations and stretches of chemical bonds (Her et al., 2008). Nuclear Magnetic Resonance (NMR) imaging is also a useful tool at researcher's disposal, providing information on the electron environment of carbon, hydrogen, and nitrogen atoms via monitoring of  $^{13}\text{C}$ ,  $^1\text{H}$ , and  $^{15}\text{N}$  isotopes, respectively (Kim et al., 2006; Mao et al., 2007b, a). New developments have also been made in the use of chromatography methods, including size-exclusion chromatography (SEC) and flow-field fractionation (FFF) (Baalousha et al., 2011; Johann et al., 2011; Yohannes et al., 2011; Alasonati et al., 2010); as these methods provide a means of physical separation by passing samples through a separation column. This chapter provides an overview of one of the more promising column separation methods, high-performance size exclusion chromatography (HPSEC).

##### 4.1. High performance size exclusion chromatography

HPSEC is a useful separation technique that has found uses in NOM weight fractionation, and operates similar to other separation techniques such as vapor pressure osmometry (VPO) or asymmetrical flow field-flow fractionation (AF4) (Pifer and Fairey, 2012; Neubauer et al., 2013; Pelekani et al., 1999). HPSEC operates by allowing the operator to pass a source sample through the column – also known as the mobile phase – which contains a bed of porous beads in the 1–3  $\mu\text{m}$  range. As the source sample passes through the porous beads, LMW components will more readily enter the pores while HMW ones will be excluded. Provided the conditions are met, a detector will be able to identify the HMW components eluting first and the LMW components eluting later. This provides a size distribution of your target sample, provided a detector is available to measure your analyte of interest. HPSEC has become a widely used method for NOM characterization due to its size separation, speed of analysis, simple sample preparation, and the availability of HPLC systems already present in various research laboratories. HPSEC has also shown to be more effective than FFF, due to its ability to cover a wider distribution of MW (Stolpe and Hassellöv, 2007; Dahlqvist et al., 2007). The use of HPSEC coupled with UVA (Li et al., 2017; Wang et al., 2016a; Lai et al., 2015; Kawasaki et al., 2011; Matilainen et al., 2006) and/or TOC detection (Huber et al., 2011; Kawasaki et al., 2011; Lankes et al., 2009; Warton et al., 2008; Lee et al., 2004; Alberts et al., 2002) has shown promise in further NOM characterization. Researchers have used HPSEC-UVA to analyse the effect of pre-treatment applications for the purpose of comparison between water quality following membrane filtration (Domínguez Chabalíná et al., 2013), coagulation (Lai et al., 2015; Xing et al., 2012; Korshin et al., 2009; Chow et al., 2008; Fabris et al., 2008b), ozonation (Wei et al., 2016), ion-exchange (IX) (Allpike et al., 2005) and in analysing other general NOM characteristics (Yan et al., 2012; Li et al., 2006, 2012; Her et al., 2008). HPSEC research has more recently been expanded through the use of more advanced detection methods such as: photodiode arrays (Yan et al., 2012; Sarathy and Mohseni, 2007; Huang et al., 2016), mass spectrometry (Li et al., 2017; Laborda et al., 2009; Bolea et al., 2006; Jackson et al., 2005; Kaschl et al., 2002), and fluorescence detection (Liu et al., 2009; Park, 2009; Baker et al.,

2008; Allpike et al., 2005; Her et al., 2003; Alberts et al., 2002; Baker, 2001).

An example of the application of HPSEC in the work carried out by Lai et al. (2015) who examined the effects of coagulation on pre-treatment control for an UF membrane. By using peak-identification techniques the authors were able to identify that HMW components were readily removed first, followed by humic substances, followed with LMW acids (Lai et al., 2015). This was to be expected as coagulation does preferentially remove HMW components (Fabris et al., 2008b; Haberkamp et al., 2007; Sharp et al., 2006), which did, in practice, correlate to improved normalized flux. However, a better understanding of NOM can help supplement the conclusions researchers and practitioners can make; as components such as hydrophilic NOM has been tied to membrane fouling (Liu et al., 2017). When HPLC was used to characterize two surface waters as performed by Alberts et al. (2002), SEC was used to characterize HS from the source water. HS was found to heavily correlate to UVA<sub>254</sub> and characteristic fluorescence peaks at  $\lambda_{\text{ex}}$  and  $\lambda_{\text{em}}$  of 225 and 425 respectively (Alberts et al., 2002). The effects of heated aluminium oxide particles (AOP) on NOM-induced fouling was investigated using HPSEC in the work of Wang et al. (2016b). This work was of particular interest as AOP were found to remove aromatic groups of NOM, and over a wide-distribution of MW ranges. This allows HPSEC to act as an investigator into the nature of NOM components, while additionally serving to provide information on the size distribution of these components; thereby providing a practical way of removing components that would have otherwise contributed to membrane fouling. HPSEC has also been expanded in the analysis of IX treatments, and other processes which isolate species based on charge affinity as oppose to physical separation (Allpike et al., 2005). A type of IX resin, known as MIEIX (magnetic ion-exchange) have been implemented for the purpose of organics removal due to its affinity for negatively charged NOM, therefore facilitating its removal in various water bodies (Kaewsuk and Seo, 2011; Kabsch-Korbutowicz et al., 2008; Mergen et al., 2008; Humbert et al., 2005; Singer and Bilyk, 2002). The work of Kaewsuk and Seo (2011) utilized MIEIX technology by looking at the major components of NOM treated using MIEIX in examining fouling potential on NF systems (Kaewsuk and Seo, 2011). Research using HPSEC led to the discovery that MIEIX (and IX in general) is not effective in removing hydrophobic groups in NOM, which also have high corresponding values for SUVA. It was noted that both polyhydroxyl aromatics and lignin were not retained via adsorption, and this led to induced flux decline (Kaewsuk and Seo, 2011). An earlier study carried out by Fearing et al. (2004) was aware of this limitation, and noted a 70% removal of chromophoric groups (measured as UVA<sub>254</sub>) and 60% removal of DOC following MIEIX; but following a minimal dose of ferric coagulant (5  $\text{mg L}^{-1}$ ) values improved to 75% and 100% for the removal of DOC and UVA<sub>254</sub> respectively. Investigators also looked at TTHMf, which improved from an initial value of 200  $\mu\text{g L}^{-1}$  following MIEIX; to 71  $\mu\text{g L}^{-1}$  following coagulation; followed by 24  $\mu\text{g L}^{-1}$  following coagulation and MIEIX in combination (Fearing et al., 2004). This outlines an interesting application of SEC, that is, treatments can be tested in tandem in order to provide additional information on the resulting water quality parameters affected. A more wide spread implementation of SEC was carried out in the work of Kim et al. (2017), which traced various fractions of NOM and the effect of ozone and bank filtration on THM formation. The removal of organic acids – associated with humic acids and the HPO, as well as transphilic (TPI) fraction, were noted to follow in a lower HAAfp per DOC carbon. Another interesting take-away from their work was that the use of a weak anion-exchange resin to remove weak acids, noted by the authors as the HPO fraction, introduced a significant reduction in THMf, HAAfp and SUVA (Kim et al., 2017).

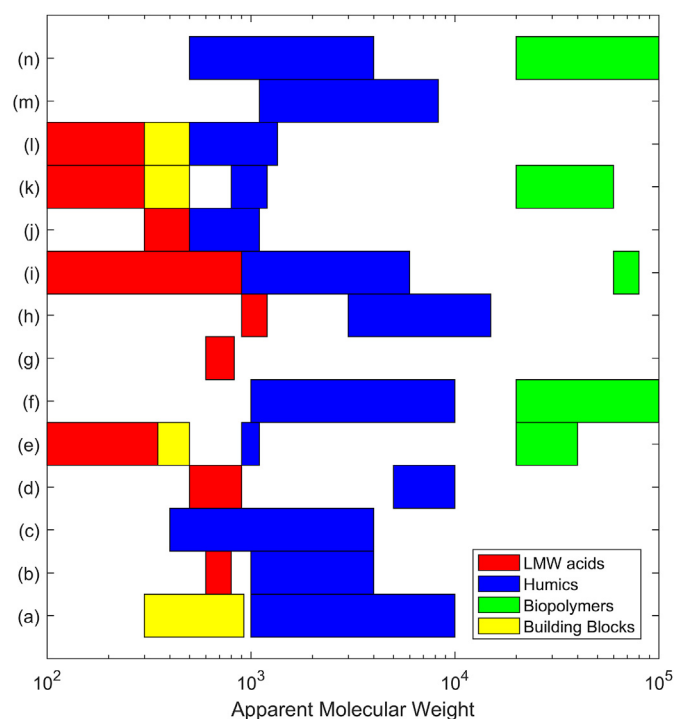


In the work of Xing et al. (2012), HPSEC was used to test the effectiveness of coagulation treatments on the elimination of chromatogram peaks compared using peak fitting. The authors found that the elimination of select peaks were tied to the means of treatment, including: conventional coagulation, enhanced coagulation; or were recalcitrant (Xing et al., 2012). The main limitation to this work is that the waters tested, which included a series of Chinese waters, all included low SUVA values, therefore the limitation of their analysis was specific to only these water types. A newer application of HPSEC was carried out recently in the work of Chon et al. (2017), that can potential establish a new field of research in NOM characterization. The researchers provided a means to reduce the heterogeneity of NOM using HPSEC, while allowing further characterization after separation. Many HPSEC applications are destructive methods, requiring a phosphate mobile phase to separate properly, while the work of Chon et al. (2017) was able to do so with de-ionized (DI) water and with a 97% retrieval rate by carbon mass. The work was also able to relate the size of NOM with the binding affinity to form NOM complexes; with polyhydroxyl aromatics, with medium MW and high hydrophobic character, having the greatest propensity to form NOM-metal complexes (Chon et al., 2017). This conclusion could have helped to explain more about the fate of metallic species in contact with NOM, as was investigated in the work of Li et al. (2017). The work attempted to demonstrate the impact of NOM on the sorption of As(III) and As(V) on a modified granular natural siderite. Using HPSEC, the researchers were able to demonstrate that As(V) complexed with HMW components of NOM – more specifically, the HA fraction – were thereby removed via ternary complex formation via adsorption onto the sorbent. HA and FA were found to have different impacts on complex formation, which is to be expected as they fundamentally have different structures as demonstrated in Table 1. This conclusion was based off of a series of kinetic isotherms and HPSEC chromatograms; while a more thorough analysis using pyrolysis-gas chromatography-mass spectrometer (Py-GC-MS) or FTIR could have been carried out if samples were preserved following HPSEC analysis. The series of work summarized previously provides an overview of the main uses of HPSEC in a potable water setting – the benefits of which are two-fold: (i) HPSEC can trace the elimination of select MW ranges to a treatment method, in which various treatment methods have preferential removal for certain ranges due to steric effects; and (ii) combining HPSEC with fractionation and/or absorbance detection can show removal of select NOM fractions, and correlate those with an predictive outcome such as DBP formation. For these reasons, HPSEC can act as a practical tool for researchers to further unravel the heterogeneity of NOM.

#### 4.2. Apparent molecular weight determination

One of the advantages of HPSEC is the ability to trace removal of water components by apparent molecular weight (AMW). The determination is very much *apparent* as the separation by weight is specific to the SEC column conditions and apparatus for which it is measured, and it is not meant to be applicable to the results of others. This however does not mean that we cannot draw useful conclusions from the results of HPSEC. For example, researchers in a wide variety of disciplines have classified the main components of water bodies into AMW ranges. Since researchers have utilized different column conditions, for the aforementioned reasons these ranges have some variance. Fig. 1 lists some of the main designations given to AMW ranges for biopolymers, building blocks (hydrolysates of humics), LMW acids/neutrals, and humics (Sillanpää et al., 2015).

Variance exists between groups and within definitions due to



**Fig. 1.** Summary of AMW ranges used by researchers investigating potable/wastewater treatment using HPSEC, (a) (Lewis et al., 2011; Carvalho et al., 2008), (b) (Phetrak et al., 2016; Humbert et al., 2005), (c) (Phetrak et al., 2014), (d) (Matilainen et al., 2002; Amy et al., 1987), (e) (Hu et al., 2014; Huber et al., 2011), (f) (Kaewsuk and Seo, 2011; Shon et al., 2006; Her et al., 2002), (g) (Chin et al., 1994; Aiken et al., 1992; Aiken and Malcolm, 1987), (h) (Beckett et al., 1987), (i) (Lai et al., 2015), (j) (Lewis et al., 2012; Ko et al., 2009; Chamorro et al., 2005), (k) (Hidayah et al., 2016), (l) (Black and Bérubé, 2014), (m) (Matilainen et al., 2011; Chen et al., 2003), (n) (Li et al., 2012).

the nature of the application. For example, in the work of Liu et al. (2010), a distinction is made between LMW acids and LMW neutrals; while in other studies these compounds are grouped together. It is also the case that the HPSEC chromatograms produced in Liu et al. (2010) did not include MW standards, and therefore relied on attributing the retention time of peaks to the work of others (Rosenberger et al., 2006; Huber and Frimmel, 1996). Other work such as that carried out by Li et al. (2013) included EEM matrices to assign peak to humics and humic-like components in reverse-phase HPLC. As noted by the authors, reverse-phase HPLC separates based on hydrophilic as opposed to size; thereby treating hydrophobic humics differently depending on the mobile phase used (Li et al., 2013). This of course can have an impact on the AMW determined for your sample if the relative elution of peaks varies from one condition to the next. It was also mentioned prior that FFF and HPSEC are two methodologies applied in this regard, and it has been shown in literature that the AMW determined between these two methods varies when applied on the same source water (Neubauer et al., 2013; Jackson et al., 2005). However, consistent with the bulk of literature outlined in Fig. 1, HMW components consist of polysaccharides and proteins (biopolymers), while humics and aromatics are concentrated in the LMW range of the distribution (Liu et al., 2014; Kaewsuk and Seo, 2011; Shon et al., 2006; Her et al., 2002). The work of Lewis et al. (2011) outlined HMW components as those that are greater than 1 k Da, most of which are derivations of lignin-like material. This is in line with investigations using HPSEC which note the absence of HMW components in well waters, which are most likely due to an absence of lignin and lignin-like derivatives (Szabo and Tuhkanen, 2010). Lignin materials have been heavily tied to fouling in membrane



filtration units, so HMW components identified by HPSEC can be used to monitor this effect (Al-Halbouni et al., 2008). As these MW distributions constitute complex groups of organic constituents, researchers have developed strategies to target them. The impact on these AMW distributions by conventional and advanced treatment methods will be explored in this section.

The effect of conventional potable treatment methods on the AMW distribution is well established in the literature. HMW components in water are readily removed in conventional treatment plants, defined as those having coagulation coupled with clarification (Drikas et al., 2011; R. D. Mergen et al., 2009; Chen et al., 2003; Matilainen et al., 2002). This is due, in part, to the nature of floc formation in coagulation treatment, with HMW components settling better than LMW components. When activated carbon (AC) was used to address this shortfall in the work of Matilainen et al. (2002), it did not readily remove the LMW fraction. In theory, LMW components would have additional surface area available for sorption within the porous structure of AC (McCreary and Snoeyink, 1980); however, LMW components are known to have high charge density, thereby making it more difficult for them to overcome the zeta potential of the sorbent surface (Collins et al., 1986). It has however been concluded by others that HMW components, in fact, with a larger aromatic character, have the highest charge density (Shuang et al., 2014; Leenheer and Croué, 2003). The discrepancy may be due to the use of terminology, as aromatics rich in electron density do not necessarily have a high charge density, which is more of a product of functionalization. It was noted in the work of Kreller et al. (2013) that aromatics are preferentially removed by adsorption on  $\alpha$ -aluminum oxide ( $\alpha$ -Al<sub>2</sub>O<sub>3</sub>), with greatest removals of components  $> 1$  k Da. It was also confirmed that over several treated bed-volumes, 5–10 k Da displaced the LMW components – leading to the conclusion that LMW components do take precedence in sorption applications, at least for the Suwannee river fulvic acid (SRFA) standard tested (Kreller et al., 2013). This conclusion had been echoed by others (Kreller et al., 2013); as well as predicted in a model developed in the work of Vandenbruwane et al. (2007) who modelled the kinetics of HMW and LMW components of NOM.

The exact MW range targeted by coagulation is up for debate, but waters tested with alum have been shown to leave AMW components in the range of 150 – 3 k Da (Lewis et al., 2011; Chow et al., 2008); while the work of Zheng et al. (2016) noted the bulk removal of components between 1–3 k Da. In the work of Lewis et al. (2012), coagulation was found to target components within 3–8 k Da, with a noticeably peak at approximately 1700 Da that was found to belong to humification by-products that contribute to color development. Oxidation using UV has also been examined using HPSEC, with a similar preference for HMW components of NOM (Black and Bérubé, 2014). Oxidation also has the added effect of increasing the portion of carboxylic acidity in the treated water (Toor and Mohseni, 2007), thereby changing the hydrophilic nature of the water distribution. This is important, as not only is MW a consideration for identifying a treatment specific for the source water, but so is choosing a treatment that accounts for the hydrophilic nature of the water. Investigation into MW fractions using IR spectroscopy was carried out by Piccolo et al. (1992), who observed HMW components are less hydrophilic than LMW fractions; thereby concluding that HMW components may aggregate due to their HPO nature (Piccolo et al., 2002). Other accounts of this phenomena have been confirmed by Xing et al. (2012) and Wang et al. (2009), who noted LMW components were hydrophilic in nature – and were identified as so by their low SUVA. It was established in Section 2.3 that the formation of DBP can follow from identification of hydrophilicity, which leads to a more focused area of research for this purpose. As mixed as this relation may be, the

additional information provided by HPSEC aims to close some of the gaps in the literature.

Investigations lead by Liu et al. (2014) noted that wastewater organic biodegradation by-products – known as soluble microbial products (SMP) – were responsible for the bulk of NOM in potable water treatment. The LMW components ( $< 1$  k Da) were responsible for 85% of the organic carbon pool and 65% of all DBP precursors (Liu et al., 2014) – and have been known to dominate municipal wastewater effluents (Szabo and Tuhkanen, 2010). This focus on LMW components was also identified in a series of work by Hua and Reckhow (2008) in a series of papers published in 2007 and 2015; as well as by others (Aryal et al., 2015). This series of work is mainly focused on the impact of chlorination conditions (more widely, disinfection in general) on the formation of DBP. The earlier work identified LMW compounds ( $< 500$  Da) with the greatest formation of HAA, THMs and other identified DBP (Hua and Reckhow, 2007). While HPO NOM have been heavily tied to the formation of known DBPs, the work identified HPO as being responsible for 80% of all unknown DBP (associated as total TOX). This follows from the statement made earlier that very few DBP are classified and regulated to begin with. In the later work of Hua et al. (2015) similar findings were made, with the addition of relationships between SUVA values and the formation of select DBPs. The findings of these publications were consistent when tested on a series of Chinese waters in the work of Lin et al. (2014). Researchers identified NOM with MW  $< 1$  k Da as being the prime contributors to carbonaceous DBP (C-DBP) and THMs. The work of Hua et al. (2018) identified peaks using HPSEC as those belonging to HS with the formation of C-DBP; however, the exact AMW range was less undertermined. When the investigation was expanded to include nitrogenous-DBP (N-DBP), the findings were mixed and inconclusive. The only other significant finding was that hydrophilics were still responsible for the bulk of iodated-DBP (I-DBP) formation, with no trend as a function of MW. Given what these selected studies tell us – the formation of DBP with different halogens, with nitrogen, and with alternative disinfectants is very much a multi-faceted environment, with no one mechanism expected to be solely responsible. With this in mind, treatments that can target the bulk MW distribution of NOM, or at the very least the HPI portion, would find practical use in preventing DBP formation.

IX is one of the treatment strategies that comes to mind that have been known to target a wide variety of NOM components. A good example of this is the work carried out by Allpike et al. (2005) who investigated enhanced coagulation in tandem with MIEX treatment. HPSEC performed indicated that enhanced coagulation preferentially removed HMW components of aromaticity and humic character, while MIEX was able to remove a more evenly distribution of NOM with less discrimination. This was determined on a MW basis and confirmed using EEM matrices, but in other work this has been confirmed using absorbance detectors (Drikas et al., 2011; R. D. Mergen et al., 2009). A further investigation by Phetrak et al. (2014) confirmed that the MW cutoff effectiveness for IX was in the region of 1.6 k Da, with aromatic DOM less than 1 k Da still remaining. The removal of components  $> 1$  k Da translated to removal rates for THMfp and HAAfp in the region of 52–77% and 47–89%, respectively (Phetrak et al., 2014). This observation suggests that humics do in fact exist in the 1.5 k Da range as suggested in the bulk of literature summarized in Fig. 1. Other accounts for IX put the effectiveness of MIEX at 1 k Da due to a combination of effects, including: entropy assisted sorption due to hydrophobic moieties, and size exclusionary effects (Tan and Kilduff, 2007; Croué et al., 1999). When MIEX was operated in a fluidized bed, the work of Kaewsuk and Seo (2011) put the MW cut-off of IX closer to 500 Da. It is uncertain whether or not this lower approximation is due to the nature of the MIEX resin itself, or whether it is due to the

conditions of the source water used; as other investigations by Humbert et al. (2008) came to a similar conclusion when using MIEX. In a thorough analysis carried out by Phetrak et al. (2016), MIEX was found to be responsible for the removal of MW components greater than 1 kDa, equated as the HPO and HPI components of NOM, with accompanying removal rates of 89–92% and 86–90% for humic-like and fluvic-like components respectively. This also led to the observation that the HPI non-acid < 700 Da was the remaining fraction that was not effectively removed (Phetrak et al., 2016; Humbert et al., 2005), which is a finding confirmed by other similar accounts (Phetrak et al., 2012; Drikas et al., 2011).

## 5. Ion-exchange resins and applications

Magnetic Ion-Exchange (MIEX) are a subset of ion-exchange resins that utilize a magnetic property which allows them to act as weak magnets, promoting fast aggregation and rapid settling (Boyer, 2015; Humbert et al., 2012). This technology was initially developed Orica Watercare, and has since been adapted for a wide assortment of applications including: heavy metal removal (Kiefer et al., 2007; Hans et al., 2016; Vagliasindi and Benjamin, 1998), wastewater effluent (Nguyen et al., 2011), nitrate reduction (Mendow et al., 2017); including the removal of phosphate (Ding et al., 2012), mercury (Neagu et al., 2007), pharmaceuticals (Wang et al., 2017, 2016a; Hu et al., 2014; Jiang et al., 2015; Lu et al., 2016), cyanobacterial toxins (Dixit et al., 2017), and NOM removal (Dixit et al., 2017; Neale and Schäfer, 2009). IX research all have a few common aspects in their implementation, the reasons are two-fold, which is that they (1) manipulate the physical and electronic properties of the exchange site to match the application; and (2) modify the backbone/counter-ion to change the selectivity of the resin towards different species. This chapter hopes to summarize the existing literature on the newest and most recent advancements of the implementation of the IX technology, with a particular emphasis on the industry-leading technology MIEX.

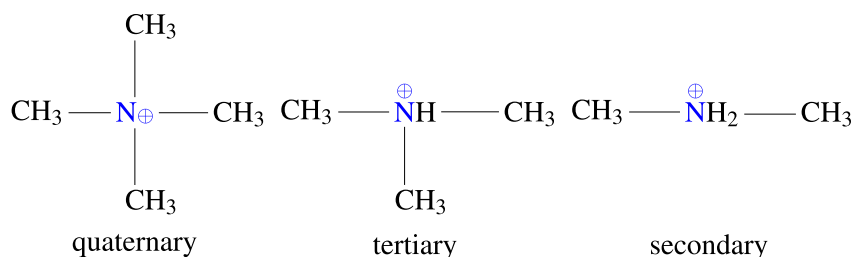
### 5.1. Resin properties

Manipulation of the resin properties has become a field of research in water technology in of itself. Bolto et al. (2002a) investigated a wide selection of IX resins and the relative effects of crosslinking and water content on effective NOM removal. As a general rule, low degrees of crosslinking and high-water contents improved resin performance (You et al., 2017; Bolto et al., 2002a). This is due to the fact that decreased cross-linking leads to an increase in the fraction of total porosity contributed by mesopores (You et al., 2017). This conclusion has been echoed by others, such as in the work by Zhang and Huang (2017) who found an increase in pore volume and mesoporosity followed a decrease in microporosity volume. The reason for this phenomenon is that crosslinking opposes osmotic forces caused by the exchange with ions with large hydrated radii; thereby facilitating the exchange of components with smaller radii that are more tightly bound to the resin. (Crittenden et al., 2012). This allows for an indirect mechanism by which the selectivity of compounds can be affected simply through steric and physical effects. The degree of crosslinking has also showed to have an effect on the acidity of IX groups in ethoxylated polyethyleneimines (Chanda and Modak, 2004; Chanda et al., 2004). The presence of acidic and basic groups in these tertiary amine groups were noted in Chanda and Modak (2004) to facilitate the diffusion path for adsorption. The structure of the backbone group has also been identified as a consideration when determining application – the two most common forms include polystyrene or polyacrylic. Styrene forms have been identified as having preferential removal for aromatic NOM versus their counterparts

(Phetrak et al., 2014; Humbert et al., 2005; Gustafson and Lirio, 1968); and has even shown preferential removal for brominated groups over other halogens (Phetrak et al., 2014). A quick application that comes to mind would a treatment system that has difficulty meeting effluent goals for brominated DBPs. Counter ions are another consideration, as their removal from the adsorbent much be thermodynamically favorable to exchange with the adsorbate of interest. Walker and Boyer (2011) noted bicarbonate counter-ion resins have a greater bromide removal than chloride form resins. Counter-ions have limited application, as the removal rates for DOC, UVA<sub>254</sub>, and sulfate were identical for both counter-ions tested. As noted earlier, the degree of substitution on the IX resin is what provides the greatest degree of selectivity, and is one of the main considerations when deciding which resin is appropriate for which application. Fig. 2 provides the series of strong-base ammonium groups and their degree of functionalization. This represents the nature of the adsorbent, and not include consideration for the backbone, counter-ion, or cross-linking effects mentioned earlier.

With an increase in substitution the amine group becomes more strongly basic, and thus can dissociate in acidic as well as alkaline environments. Their strong basicity allows them to exchange with the weakest of acids (i.e. H<sub>2</sub>CO<sub>3</sub>, H<sub>4</sub>SiO<sub>4</sub>), as well as inorganic and neutral salts (NaCl, HCl). This is true in practice, as substitution of hydrogens on quaternary amines has been shown to coordinate with the selectivity of sulfate and chlorine, by influencing the basicity of the resin (Hilal et al., 2015; Zhou et al., 2015; Sarkar and SenGupta, 2008; Boari et al., 1974). This can be useful in applications where chloride, or other water components (NaCl, MgCl<sub>2</sub>), are a substantial part of the water table, such as in seawater desalination (Chanda and Modak, 2004). Primary amines have been shown to be effective in the removal of water carbonic acid CO<sub>2</sub> (Parvazinia et al., 2018); tertiary amines in the removal of water salinity (Chanda and Modak, 2004); and quaternary amines in the removal of perchlorate (Darracq et al., 2014). As further demonstrated by Darracq et al. (2014) - resins with varying densities, polymer structures, capacities, and functional groups all have different specificity for the adsorbent of interest relative to other water ions.

Strong base resins are typically employed to remove even the weakest acids in NOM, as many of these acid groups are responsible for various water quality woes as outlined in Sec. 2.3. In the work carried out by Bolto et al. (2002a) the authors noted a comparatively even removal of hydrophilic NOM groups between strong and weak base resins, while strong base resins have more total sites available at neutral pHs. A similar conclusion was given in the work by Cornelissen et al. (2008), where the best performing resins were ones with the highest water contents. It was also suggested in the work that at neutral pHs physical adsorption comes into play (Croué et al., 1999), and hydrogen bonding of the acidic species to the unprotonated amino groups in the resin backbone was suggested as the mechanism (Bolto et al., 2002a). In the work by Cornelissen et al. (2008), a decrease in resin size was found to improve resin performance, although the driving mechanism for which was inconclusive. A larger surface area (i.e. higher capacity) was found to be the worst performing resins tested by Cornelissen et al. (2008); and Boyer (2015) noted MIEX resins - with smaller particle sizes (4–5 fold smaller) and smaller capacities (3 fold smaller) than SBA resins - were the best performing for NOM removal. The reason for which may be attributed to the selectivity of medium AMW NOM over competing ions such as carbonates, nitrates, bromides, and chlorides; while general SBA resins are more effective for inorganic ions such as bromide (Boyer and Singer, 2008).



**Fig. 2.** Ammonium group typically found in strong and weak anionic exchange resins. Increased methyl substitution increases the basicity of the ion.

## 5.2. IX applications

A wide spread field of research has been conducted on the implementation of MIEX to supersede, or to accommodate existing water treatment trains. This means improving treatments such as coagulation through enhanced optimization with MIEX (Xu et al., 2016; Humbert et al., 2005; Morran et al., 2004) and in the implementation of pilot scale operations (Boyer, 2015; Singer et al., 2009, 2007). All these studies have a common goal in mind: improving the removal rate of NOM – measured as DOC – and identify the underlying mechanisms for NOM removal. This also includes accommodating for the associated removal of water constituents which are removed in competition with NOM, such as alkalinity, chloride and bromide (Ding et al., 2012; Hsu and Singer, 2010); and even the presence of components which may enhance removal of NOM such as calcium (Arias-Paic et al., 2016). The work performed by Xu et al. (2016) found that a moderate MIEX dose (undefined) was sufficient to improve the removal of humic acids (measured as DOC) and sulfamethazole, and reduce the chemical consumption of aluminum sulfate ( $\text{Al}_2(\text{SO}_4)_3$ ). MIEX was noted as being an effective in combination with  $\text{Al}_2(\text{SO}_4)_3$  for the removal of HMW NOM components, and an effective combination for the reduction in chemical demand (Xu et al., 2016). When anionic exchange is combined with cation exchange in a mixed vessel, an effective removal of NOM and hardness can both be achieved as demonstrated in the work of Comstock and Boyer (2014). In a fluidized bed operation, Cornelissen et al. (2009) utilized anionic and cationic resins for NOM and calcium/magnesium removal respectively. In this configuration, they were able to reduce the formation of calcium-NOM complexes by targeting both components separately. It was also noted that removing one component, in removing calcium using cationic exchange, actually promoted irreversible fouling on an UF membrane (Cornelissen et al., 2009). Therefore, ion-exchange is very much a multi-faceted treatment that requires proper implementation necessary for its application.

In terms of THM formation, when put under full-scale conditions, MIEX did not show improvement over coagulation in the work carried out by Bond et al. (2010). The work tested 8 surrogate groups of NOM, and based this conclusion on relative removals of groups based on hydrophilicity prepared using octanol-water coefficients (Bond et al., 2010). When source water from nine different water utilities was compared to coagulation with alum, MIEX showed improvement for all parameters including TOC,  $\text{UVA}_{254}$ , and both HAA and THM formation (Singer and Bilyk, 2002). The enhanced performance of MIEX was also described by Kingsbury and Singer (2013), but was compared to a semi-batch treatment with ozone (Kingsbury and Singer, 2013). This conclusion was further reinforced two years later in the work performed by Fearing et al. (2004), who tested a unique water source in Halifax, England over a one-year period. The authors noted the advantage of a serial operation of a MIEX/Coagulation train, with the coagulation process alone targeting the hydrophilic groups that contributed most

to trihalomethane formation potential (TTHMfp) (Fearing et al., 2004). The contrast with the conclusions of Bond et al. (2010) can be attributed to the methodology by which the group established their baseline DBPfp. The performance of MIEX was based off of the relative removal of NOM surrogates which each had their associated contributions to DBPfp; the compound resorcinol, a hydrophobic neutral, had only a 9% removal by MIEX but had the highest DBPfp ( $1588 \text{ mg mgC}^{-1}$ ). The removal of tannic acid, a transphilic acid, had the best rate of removal (56–92%) but a very low DBPfp ( $5 \text{ mg mgC}^{-1}$  for chloroform, and  $3 \text{ mg mgC}^{-1}$  for DCAA). This lack of agreement with other literature may be due to the inability for a surrogate parameter to be assumed as represented of a larger group, and the lack of consensus on the most reactive form of NOM (Hu et al., 2014; Zhao et al., 2013; Wang et al., 2013b; Lin et al., 2010; Kitis et al., 2002). The fact that hydrophilic groups are more readily removed by MIEX is not unfamiliar in the literature (Phetrak et al., 2016; Mergen et al., 2008) but has been at odds with others (Drikas et al., 2011). The work of Phetrak et al. (2016) noted hydrophobic acids were the dominate fraction in their surface waters, and have the largest contribution to THMfp; however, the hydrophilic acids had the highest specific THMfp (Phetrak et al., 2016). The work also used surrogates for NOM which noted that the removal of hydrophilic groups was mainly driven by IX mechanisms, while hydrophobic groups can be partially removed through physical sorption in the MIEX porous structure (Phetrak et al., 2016). The inner porous structure has size exclusionary effects as the rate of free liquid diffusivity in the pores is proportional to the inverse of MW (Bazri et al., 2016; Egeberg and Alberts, 2002). This interaction has shown to be important in applications where the inner porous structure of IX is available for exchange with for pesticides such as atrazine and isoproturon, while the removal of HMW NOM readies the availability of the sites (Humbert et al., 2008). Hu et al. (2014), performed a similar analysis of the sorption of atrazine and caffeine in the inner porosity of an activated carbon resin, but utilized a MIEX resin as the pre-treatment. Similar to the conclusions of Humbert et al. (2008), MIEX removed the bulk of the HMW humic substances thereby facilitating the diffusion of LMW pesticides (Hu et al., 2014).

The long-term performance and mitigation of membrane fouling is of prime consideration for those seeking to manage a variety of source waters. Therefore, the ability for MIEX to target components of NOM that contribute to irreversible membrane fouling is paramount. The effectiveness of MIEX to remove color and DOC has been shown to operate effectively over a series of resin doses and a range of UF membranes with different MW cut-offs (Kabsch-Korbutowicz et al., 2008). When MIEX performance was analyzed over a long-period (August 2005–June 2007) – as was carried out by Drikas et al. (2011); Huang et al. (2016); Fabris et al. (2007) – DOC and  $\text{SUVA}_{254}$  was consistently removed over a variety of source water compositions. The work also showed better effectiveness in MIEX combined with NF or coagulation. The work carried out by Jutaporn et al. (2016) attempted to correlate the



improvement in MF membrane flux following MIEX, by monitoring parameters of SUVA, UVA<sub>254</sub> and DOC. While none of these parameters were directly tied to normalized membrane flux over time, MIEX pre-treatment did reduce membrane fouling potential in four natural and two synthetic waters with a wide range in composition (DOC: 1.8–11 mg L<sup>-1</sup>, UVA<sub>254</sub>: 0.056–0.208 cm<sup>-1</sup>). Humbert et al. (2012) used an IX/NF coupled operation following coagulation in a high DOC source water (DOC: 7.5–9.8 mg L<sup>-1</sup>, UVA<sub>254</sub>: 0.2–0.35 cm<sup>-1</sup>), and found a reversibility in fouling as a consequence of NOM removal (final DOC residual of 1.6 mg L<sup>-1</sup>). In the work of Cornelissen et al. (2009), the removal of NOM in addition to calcium is an effective means to reduce irreversible fouling due to the elimination of calcium-NOM complexes (Cornelissen et al., 2009). Biopolymers have been linked as key contributors to fouling, therefore investigation into their removal has been carried out (Kimura et al., 2014; Tian et al., 2013). Kimura and Oki (2017) found that MIEX combined with coagulation, in that order, performed better than MIEX/PAC, and even coagulation followed by MIEX, for normalized flux decline in MF and UF membranes. The authors also noted a better relationship between the concentration of biopolymers and fouling potential, than with DOC and fouling potential (Kimura and Oki, 2017).

The presence of biopolymers – involving LMW carbohydrates and proteins in water – are considered hydrophilic constituents, and which have also shown to promote irreversible fouling when compared to fouling caused by solely hydrophobic groups (Yamamura et al., 2014; Kaewsuk and Seo, 2011). This has been proven in combined MIEX-NF systems which show the main components in membrane permeate are carboxylic, phenolic, and fatty acids (Kaewsuk and Seo, 2011). MIEX was noted to remove the majority of phenolic components and aliphatic hydrocarbons, which are components with relatively low SUVA values. A similar conclusion was found by Huang et al. (2012) where fouling potential did not follow from DOC removal by MIEX, but the author noted this was most likely due to the composition of the membrane itself (Huang et al., 2012). The same work by Yamamura et al. (2014) noted the effect due to the membrane was hydrogen bonding between the hydroxyl groups of the biopolymers and the fluorine groups of the polyvinylidene fluoride membrane (Yamamura et al., 2014). Other researchers have examined the size composition of the water itself rather than using any surrogate form, and have found promising results. Fabris et al. (2007) looked at the effects of high molecular weight (< 50,000 Da) peaks corresponding to colloidal fractions on MF membrane fouling. HPSEC analysis found that the HMW component contributed most to MF fouling, and similar to the conclusions of Huang et al. (2012) and Kimura and Oki (2017), DOC was not indicative of any trend in fouling. When the averaged AMW for DOC was removed using a combination of biological activated carbon combined with MIEX, NF membrane saw an increase steady flux over time (Aryal et al., 2015). A more recent study by Gibert et al. (2017) reinforced this notion, where the LMW fractions of NOM (corresponding to humic and fulvic substances) where more readily removed by MIEX (DOC removal of 32–46%), as confirmed by a combination of HPSEC and Excitation-Emission Matrices (EEM) (Gibert et al., 2017). NF was found to have a preferential removal of HMW components (5% removal of LMW components) – due to size exclusion – which would indicate an ability to foul more readily when the concentration of the HMW components are higher.

## 6. Summary

This review has hope to expand on the current uses and applications of HPSEC in a potable water setting, with additional investigation on the use of strong base ion-exchange resins for

NOM control.

The literature has revealed that the coupling of our current understanding of NOM characterization – i.e. NOM fractionation and functionalization – has implications on the pre-treatment in NOM removal. This is more so the case in preventing the formation of carcinogenic disinfection by-products (DBP); which by most work can be traced to the presence of humic substances – defined as the hydrophobic fraction of NOM. The disambiguity in the most reactive fraction to form DBPs lends credence to the notion that a better tool is required for NOM characterization.

Partitioning on the basis of apparent molecular weight (AMW) can identify specific regions of ultraviolet moieties that are targeted by conventional treatment methods. This partitioning has indirect relationships to other factors (i.e. high molecular weight components are less hydrophilic), while also having direct implications on the ability to trace the removal following a treatment such as coagulation. Research has also identified low AMW components as being a large contributor to known DBP, as well as being tied with unknown DBP formation – which is a significant portion of the total organic halogen formation pool.

The focus on ion-exchange (IX) helps to highlight it as a known effective treatment specifically targeted towards the removal of the carboxylates and phenolates of NOM. The amount of potable water treated from all sources via ion-exchange in Canada has almost increase 4-fold from 2013 to 2015 when the most recent surveys were recorded. This has occurred as IX resins became more commercially available apart from the popular MIEX variant; and as new resins received their NSF/ANSI certification for use in human consumption.

Search results of peer-reviewed papers covering the topic of “NOM” and “HPSEC” has almost doubled in the past decade; and more than doubled in applications spanning many disciplines. This review does not even include the numerous applications of HPSEC in a wastewater setting, such as monitoring the presence of extracellular polymeric substances. The scope of this work is very much at its early stages, with a more commercial application for HPSEC as a design tool very much in the horizon.

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