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MIEX®DOC process to remove disinfection by-product precursors

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ABSTRACT

In this study, the MIEX®DOC process was applied to test the removal potential of halogenated organic water disinfection by-products (trihalomethanes, haloacetic acids, haloacetonitriles, haloketones, chloropicrin and chloral hydrate) precursors. The experiments were conducted using model and surface water samples. The samples of untreated water and the MIEX®DOC process effluent were chlorinated with sodium hypochlorite which resulted in residual free chlorine of 3–5 mg L^{-1} after 24 h. After this time, the water was analysed to check the presence of chlorination by-products using the gas chromatography method. Subsequently, the formation potential was calculated for each group of halogenated organic compounds. The efficiency of the MIEX®DOC process in removing the disinfection by-product precursors was dependent on the type and quality of raw water. The MIEX®DOC process showed the highest removal efficiency for trihalomethanes (68%–90%), haloacetic acids (76%–82%) and chloral hydrate (62%–88%) precursors. Lower removal efficiencies were observed for the precursors of haloacetonitriles (42%–72%), haloketones (26%–73%) and chloropicrin (9%–74%).

Keywords: MIEX®DOC process; Water treatment; Formation potential; Disinfection by-products

1. Introduction

By-products of water disinfection are formed as a result of chlorine reaction with natural organic matter (NOM) and other halides present in water during its disinfection. During water chlorination various halogen organic compounds are created and this process has been proven harmful to human health. The removal of NOM prior to disinfection can significantly minimise the formation of disinfection by-products. NOM is most often removed in the process of coagulation and adsorption on active carbons; however, a very promising alternative to these conventional processes is the application of the magnetic ion exchange resin MIEX® [1]. The characteristic feature of the MIEX® resin is a magnetic element embedded in the polymer structure, which enables the formation of large and heavy agglomerates, that sediment efficiently [2]. Open flow-through tanks used in the process facilitate recovery and regeneration of the resin while fine granulation of the resin (ca. 150 μm) enables work in suspension [3,2]. The MIEX®DOC process has proven to efficiently decrease the dissolved organic carbon (DOC) concentration, colour, and absorbance (UV 254 nm) [4,5]. Also, the exchange of anions takes place [4,6]. The MIEX®DOC process can be easily combined with other individual processes. When combined with the coagulation process, it results in lower coagulant's dose. In case of combining with the membrane filtration process, the adverse phenomenon of microfiltration and ultrafiltration membrane blocking can be prevented [7] and the permeate is of much higher quality [8].

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NOM stands for a mixture of many hydrophilic and hydrophobic compounds of aliphatic and aromatic structure with different functional groups attached [9] and therefore in the MIEX®DOC process the NOM compounds are removed selectively [5,10,11]. In the MIEX®DOC process, the compounds with lower molecular masses are more easily removed [5,12]. The MIEX® resin does not remove organic particles larger than 5,000 Da [12], but is very efficient in removing organic particles with molecular mass between 500 and 1,500 Da [12]. The MIEX® resin is capable of exchanging anions, so in the MIEX®DOC process anionic NOM fractions are removed, for example, hydrophobic and hydrophilic acids [5,12–14,16]. This resin, however, is also capable of exchanging neutral hydrophilic compounds [5,11,12]. According to Fearing et al. (2004) [13], the MIEX® filter is the most efficient in removing hydrophobic NOM fractions, slightly less effective in neutral hydrophilic compounds removal, while acid hydrophilic fractions are removed the least efficiently. However, high-molecular hydrophobic acids can quickly saturate the filter or block the access to the filter for other compounds. For that reason, the efficiency of removing individual NOM fractions also depends on the content of raw water. In water with small content of high-molecular weight hydrophobic fractions, the removal of acid hydrophilic fractions can be more effective [11]. In acid hydrophobic fractions, which include humic and fulvic acids, the preference of the MIEX®DOC process in their removal from water was also reported. According to Fearing et al. [13], the ion exchange process in the MIEX® resin remove fulvic acid fractions more effectively than humic acid fractions. Acid hydrophilic fractions can also be selectively removed. As the research conducted by Bond et al. [11] showed, aspartic acid is more efficiently removed than glutamic acid on the MIEX® resin.

Typical NOM characterisation through the measurement of DOC, UV_{254} absorbance or colour is not sufficient to determine the applicability of the MIEX®DOC process to reduce precursors concentration in the formation of halogen disinfection by-products. For this purpose, the formation potential of chlorination by-products should be determined. Its aim is to specify the reactivity of NOM included in water in relation to the formation of organic halogen disinfection products. Such studies have been carried out by many authors [11,13,15–18]; however, the research has reported on the formation potential of trihalomethanes (THM) and haloacetic acids (HAA), which are most common water chlorination by-products. The studies with regard to the formation potential of other by-products are scarce. So far, only one study has been published on the impact of the MIEX®DOC process on the reduction of formation potential of organic by-products that include nitrogen in their composition [17], namely halonitromethanes. The removal of precursors of other by-products in MIEX®DOC process has not yet been published.

The aim of this paper is to determine the applicability of the MIEX®DOC process to remove precursors to the formation of a range of halogen organic water disinfection by-products. The research was conducted in model and surface water samples. The disinfection by-products formation potential was determined for THM, HAA, haloacetonitriles (HAN), haloketones (HK), chloral hydrate (CH) and chloropicrin (CP).

2. Methods

2.1. Water samples

The research was conducted on the following water samples:

- model water containing humic acids (Sigma-Aldrich, Germany) dissolved in deionized water; denoted here as “HA”;
- model water containing fulvic acids (Beijing Multigrass Formulation Co. Ltd) dissolved in deionized water; denoted here as “FA”;
- model water containing fulvic and humic acids added to deionized water as well as Br^- and F^- anions (added to water in the form of KBr and NaF salts made by Avantor Performance Materials Poland S.A.); denoted here as “HA + FA”;
- surface water collected from the water reservoir at Kozlowa Gora water treatment facility, Silesian Voivodeship; denoted here as “KG”.

2.2. MIEX®DOC Process

All tested waters were treated in the ion exchange MIEX®DOC process. In the ion exchange process the macroporous anion-exchange MIEX® resin was used (Orica Watercare). 10 ml L^{-1} resin was added to a 20-L container with raw water to form a suspension and it was regenerated with the 10% NaCl solution. The MIEX® resin was in contact with treated water for 30 min. After this time, water was decanted and samples were taken for further analyses.

2.3. Formation potential of chlorination by-products

In order to study the formation potential of individual disinfection by-products, a 24-h chlorination test was used. Water samples were chlorinated with the use of sodium hypochlorite in a dose sufficient to achieve 3–5 mg L^{-1} of residual free chlorine after 24 h. pH of all samples were corrected to 7 with the use of sulphuric acid, sodium hydroxide or phosphate buffer, depending on the initial pH reading. Chlorinated samples were incubated in the temperature 25°C ± 2°C in dark glass bottles with the seal covered with the PTFE material.

After 24 h of incubation, the samples were dechlorinated and concentrations of the following compounds were determined:

- THM group: trichloromethane, bromodichloromethane, dibromochloromethane, tribromomethane;
- HAA group: monochloroacetic acid, monobromoacetic acid, dichloroacetic acid, trichloroacetic acid, bromochloroacetic acid, dibromoacetic acid;
- HAN group: trichloroacetonitrile, dichloroacetonitrile, bromochloroacetonitrile, dibromoacetonitrile;
- HK group: 1,1 – dichloropropanone, 1,1,1 – trichloropropanone; CH;
- CP.

2.4. Analytical methods

After 24 h, the formed disinfection by-products were analysed by means of the GC-MS method using gas chromatograph Trace Ultra DSQII by Thermo Scientific. Helium was used as a carrier gas. Compounds separation was performed on capillary column Rxi^{TM}-5ms made by Restek company (film thickness 0.5 µm; column length 30 m; internal column diameter 0.25 mm). Analysed volatile compounds (THM, HAN, HK, CH and CP) were extracted using MTBE (methyl-tert-butyl-ether) and detected by means of the GC-MS method. The following temperature phased sequence was used: 35°C (9.5 min) to 200°C (0 min), temperature accretion rate 40°C min^{-1}. Individual compounds detection limits were ±0.01 µg L^{-1}.

HAA concentration was determined with the use of acid esterification and the GC-MS method [17]. The column of chromatograph was heated from 40°C (0 min) to 100°C (5 min) with temperature build-up 40°C min^{-1}, then to 200°C (0 min) with the accretion rate 8°C min^{-1}. Detection limit was 0.5 µg L^{-1} for MCAA and MBAA and 0.01 µg L^{-1} for other HAA.

Free chlorine concentration was determined with the colorimetric method utilising DPD (N,N-diethyl-p-phenylenediamine) according to the PN-ISO 7393-2 norm. The content of free chlorine was measured on Aurius 2021 UV-VIS spectrophotometer made by Cecil Instruments. Detection limit of this method was 0.03 mg L^{-1}.

DOC was determined on HiPerTOC (Thermo Scientific) organic carbon analyser. UV absorbance with the wave length 254 nm (UV$_{254}$) was measured on Aurius 2021 UV-VIS spectrophotometer (Cecil Instruments).

Concentration of dissolved organic nitrogen (DON) was calculated as a difference between total nitrogen and the sum of all inorganic nitrogen forms (NH_4-N, NO_2-N, NO_3-N). Nitrogen compounds were determined photometrically with the use of Nanocolor tests on Aurius 2021 UV-VIS spectrophotometer (Cecil Instruments). Detection limits for nitrogen compounds were: total nitrogen – 0.1 mg L^{-1}; NH_4-N – 0.01 mg L^{-1}; NO_2-N – 0.002 mg L^{-1}; NO_3-N – 0.02 mg L^{-1}.

Bromide concentration was determined by the spectrophotometric method with chloramine-T as an oxidizing agent and phenol red as an indicator. Bromides content was measured on Aurius 2021 UV-VIS spectrophotometer made by Cecil Instruments. Detection limit of this method was 0.1 mg L^{-1}.

3. Results and discussion

3.1. *Organic matter removal*

In Table 1, the results of raw water samples quality are presented. DOC contents in all samples of model raw water was at the same level and ranged from 9.55 mg L^{-1} for the FA sample to 9.68 mg L^{-1} for the HA sample. DOC concentration in surface water samples was slightly lower and amounted to 8.68 mg L^{-1}. Considering the value of SUVA$_{254}$ parameter, the HA model water sample had the highest hydrophobicity (5.837 L mg^{-1} m^{-1}), while the KG surface water sample had the lowest (1.786 L mg^{-1} m^{-1}). The KG water sample also had the lowest content of bromides (0.1 mg L^{-1}) while the HA + FA model water sample the highest (0.3 mg L^{-1}). It resulted from the addition of inorganic salts including bromides. Based on the fact that during chlorination nitrogen-containing by-products are also formed (haloacetonitriles, halonitromethanes), the DON content was determined. The values of these parameters for individual water samples are included in Table 1.

Fig. 1 shows the efficiency of NOM removal in the MIEX®DOC process. The highest efficiency of DOC removal in the MIEX®DOC process (92%) was achieved for HA and FA model samples, which include pure solutions of humic and fulvic acids. The lowest result of DOC removal (56%) was achieved for the HA + FA water sample, in which inorganic bromide and fluoride salts were additionally dissolved. The efficiency of the MIEX®DOC process depends then on the competitiveness between individual ions in the treated water [19]. However, with lower efficiency of DOC removal in the HA + FA sample, the absorbance value UV$_{254}$ decreased (88%) but is still comparable with its removal efficiency in the MIEX®DOC process in model water samples, that is, HA (85%) and FA (92%). In surface water sample with 62% efficiency of DOC removal, the UV$_{254}$ parameter has decreased by 85%. Organic nitrogen from model water samples was removed in only 9%, while KG surface water samples in 17%. Previously reported DON removal efficiency in the MIEX®DOC process are slightly higher and reach 37%–54% [17]; however, as Boyer et al. (2008) report, there is no solid pattern in DON removal in the MIEX®DOC process [20].

Table 1
Raw water quality

Name of the sample	DOC (mg L^{-1})	DON 9mg L^{-1})	SUVA$_{254}$ (L mg^{-1} m^{-1})	Br^{-} (mg L^{-1})
HA	9.68	1.08	5.837	0.2
FA	9.55	0.92	3.204	0.3
HA + FA	9.64	0.57	4.232	0.3
KG	8.68	0.90	1.786	0.1

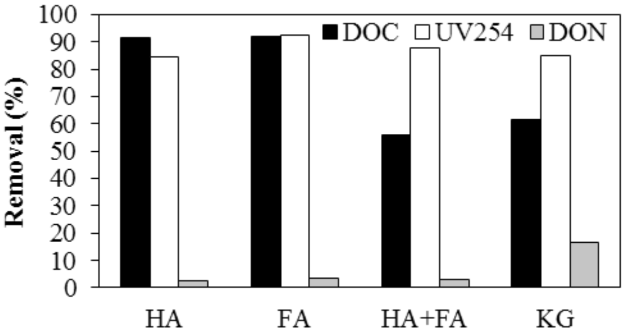

Fig. 1. Natural organic matter removal in MIEX®DOC process.

3.2. *Trihalomethanes formation potential*

The results of analysis on trihalomethanes formation potential (THM-FP) are presented in Fig. 2. THM-FP is a sum of formation potentials of trihalomethane, bromodichloromethane, dibromochloromethane and tribromomethane.

The observed THM-FP values for raw waters oscillated from 203.02 µg L^{-1} to 500.67 µg L^{-1}. The highest THM-FP has been achieved for the HA model water sample, and the lowest for the FA model water sample. The KG surface water sample had higher THM-FP than the FA sample despite lower SUVA$_{254}$ (Table 1). After the MIEX®DOC process THM-FP values were from 19.91 µg L^{-1} for the FA water to 159.60 µg L^{-1} for the purified HA model water, which corresponded to the THM formation potential of 90% and 68%, respectively.

The THM-FP of the combined HA and FA model water was decreased by 80% in the MIEX® resin. Similarly, treated KG water has reduced the THM-FP by 86%.

Specific precursors of THM are organic aromatic compounds, which have high hydrophobicity and contain carboxylic groups in their structures. The highest THM-PT was observed in the HA model water as well as in water with the mixture of humic and fulvic acids (HA + FA). The HA model water treated with the MIEX®DOC process has exhibited the lowest decrease of THM-PT, which could be caused by the lower ion exchange rate of hydrophobic anion organic compounds with the molecular weights above1,000 Da such as parts of humic acids. The results presented elsewhere [21] showed the presence of compounds with the molecular weights of 300–3,000 in the HA model water. More efficient removal of THM precursors in the FA model water and surface water can indicate that these waters consist of the mixture of hydrophobic and hydrophilic organic compounds with lower molecular weights, which are removed by the ion exchange on the MIEX®resin.

The THM-FP removal depends also on the quality of raw water. In Singer and Bilyk's research [16], carried out on nine water types obtained from water treatment facilities, the THM-FP removal with 60 min contact with the MIEX resin oscillated from 71% to 84%, depending on the type of treated water, and the highest level of removal was observed in waters with highest $SUVA_{254}$ parameter. In other research [17], also conducted on waters from water treatment facilities, with 30-min contact and 10 ml L^{-1} dose of the MIEX® resin, the THM-FP reduction was achieved in range 39%–86%, and in the study of Boyer et al. [20], the results were 47%–69%. In the studies conducted in waters rich in humic acids, Fearing et al. [13] achieved the reduction of THM-FP at the level of 70%.

3.3. Haloacetic acids formation potential

In Fig. 3, the haloacetic acids formation potential (HAA-FP) is presented for all analysed waters, both before and after treatment in the MIEX®DOC process. HAA-FP is a sum of formation potentials of monochloroacetic acid, monobromoacetic acid, dichloroacetic acid, trichloroacetic acid, bromochloroacetic acid and dibromoacetic acid.

Results comparable with those achieved for the THM group were obtained in the raw waters. The highest HAA-FP value prior to treatment was achieved for the HA model water (575.02 μg L^{-1}), while the lowest for the KG surface water sample. HAA-FP values correlate well with $SUVA_{254}$ results (Table 1). It proves that the main precursors of HAA formation are hydrophobic organic compounds, which are efficiently removed by the MIEX®DOC process.

After the process of ion exchange on the MIEX® resin, HAA-FP for the analysed samples decreased to 76% (for KG surface water sample) and 83% (for HA model water sample). For the FA model water sample, very high HAA-FP removal efficiency was achieved (82%), while for HA + FA model sample the efficiency of the MIEX®DOC process was 78%.

In research conducted by other authors, the HAA-FP removal in the ion exchange process on the MIEX resin was also observed. The formation potential of HAA was decreased by more than 60% in the research study by Singer and Bilyk [15], by 42%–87% in Gan et al. [17] by 26%–69% in Boyer et al. [16].

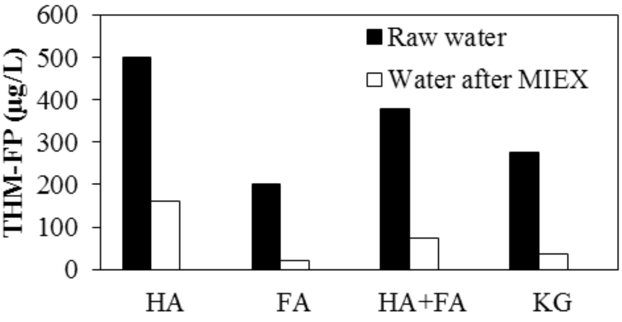

Fig. 2. Trihalomethanes formation potential.

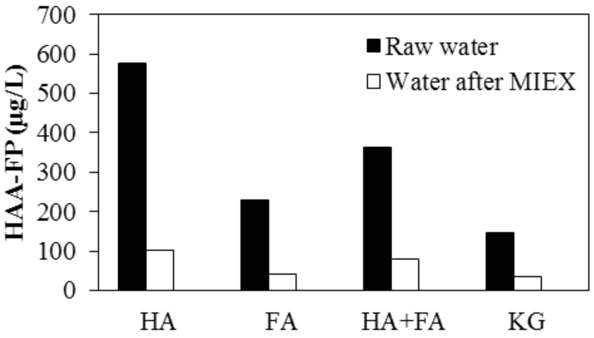

Fig. 3. Haloacetic acids formation potential.

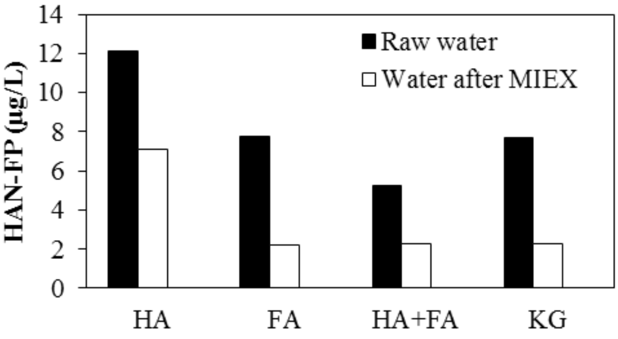

Fig. 4. Haloacetonitriles formation potential.

3.4. Haloacetonitriles formation potential

Haloacetonitriles formation potential (HAN-FP), calculated as a sum of formation potentials of trichloroacetonitrile, dichloroacetonitrile, bromochloroacetonitrile and dibromoacetonitrile, is presented in Fig. 4.

For raw water samples, the highest HAN-FP was achieved for the HA model water (12.11 μg L^{-1}). For the FA model water and the KG surface water, the values of these potentials amounted to 7.75 μg L^{-1} and 7.72 μg L^{-1}, respectively. For the HA + FA model water sample, on the other hand, the lowest value of HAN-FP was achieved, namely 5.25 μg L^{-1}. In the case of humic acids present in the water sample, the efficiency of HAN-FP removal in the MIEX®DOC process was lower. For the HA model water sample, the lowest HAN-FP removal efficiency was achieved (42%), and for the HA + FA model water this efficiency was 57%. The highest level of HAN-FP removal in the MIEX®DOC process was achieved for the FA model water sample (72%), and the removal the KG surface water sample. Haloacetonitriles can be formed while chlorinating free aminoacids, nitrogen from heterocyclic structures in nucleic acid, other protein compounds and aminoacids linked to humic compounds structure [22,23]. Although the removal of organic nitrogen in the MIEX® filter does not have a regular pattern [20], in model samples with fulvic acid the highest removal efficiencies of precursors to the formation of HAN were achieved. High level of HAN-FP removal in the MIEX®DOC process in surface water sample can be the effect of high DON removal in this sample (Fig. 1).

3.5. Haloketones formation potential

Research results of haloketones formation potential (HK-FP), known as a sum of formation potentials of 1,1-dichloropropanone and 1,1,1-trichloropropanone, for the analysed water samples are presented in Fig. 5.

In raw water samples that include fulvic acids, the lowest HK-FP concentrations were achieved. For the FA model water sample, the HK-FP amounted to 5.68 μg L^{-1}; for the HA + FA model water sample, it was slightly higher (7.13 μg L^{-1}); the highest value was achieved for the HA model water (26.16 μg L^{-1}). HK-FP in the surface water sample was also relatively high (17.16 μg L^{-1}). In case of removing precursors to the formation of HK in the MIEX®DOC process, the highest efficiency of HK-FP removal was achieved for the HA model water sample and the KG surface water sample (73% and 54%, respectively), and the lowest for water samples containing fulvic acids (26% for the FA sample and 37% for the HA + FA sample). Such tendency of removing HK precursors can be explained by the fact that the precursors to the formation of HK are hydrophobic acids [24,25], including mainly humic acids, which are relatively efficiently removed in the MIEX®DOC process. For that reason, in the model water samples containing humic acids, the HK-FP removal efficiency was higher than in water with fulvic acids. Relatively high removal efficiency of precursors to HK from the KG surface water sample can result from the fact that smaller hydrophilic compounds, such as ketones, which are also precursors to the formation of HK, were removed.

3.6. Chloral hydrate formation potential

Research results on the chloral hydrate formation potential (CH-FP) for samples of water before and after treatment in the MIEX®DOC process are presented in Fig. 6.

The CH-FP in water samples before the MIEX®DOC process was the highest in the FA model water sample (40.42 μg L^{-1}). Similar CH-FP results were achieved for KG, HA + FA and HA water samples, reaching 27.95 μg L^{-1}, 28.13 μg L^{-1} and 28.83 μg L^{-1} respectively. Significant CH precursor removal efficiency has been achieved in model water samples that included humic acids in their composition (11% for the HA + FA sample and 9% for the HA sample); in case of the FA model sample and the KG sample, the efficiency was 5%. CH is a by-product containing organic carbon; therefore, the CH precursors are removed alongside the DOC removal.

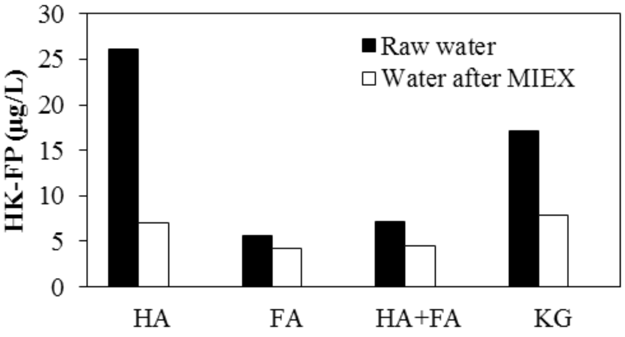

Fig. 5. Haloketones formation potential.

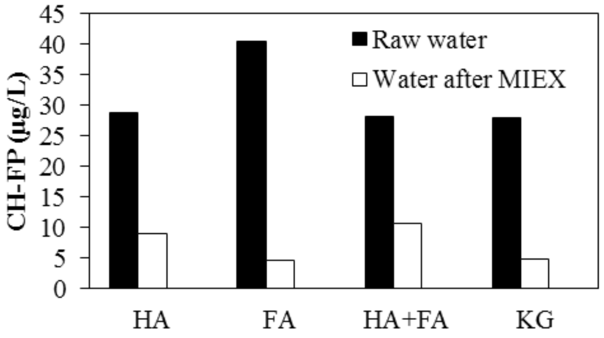

Fig. 6. Chloral hydrate formation potential.

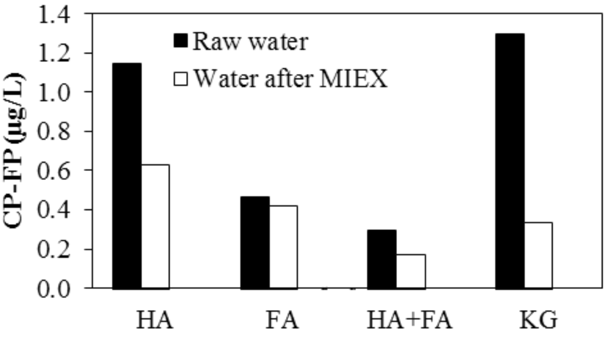

Fig. 7. Chloropicrin formation potential.

CH-FP removal efficiencies in MIEX®DOC process oscillate at a much lower level than THM-FP and HAA-FP. Precursors to the formation of CH are aminoacids belonging to hydrophilic alkali fractions, and products of their oxidation are aldehydes, which are neutral hydrophilic compounds [26]. Decrease in the CH-FP in the MIEX®DOC process can be caused, to a large extent, by the removal of hydrophilic DOC fractions from water. This hypothesis is supported by high efficiency of removing CH-FP from the KG surface water and the FA model water, which are the samples of relatively high hydrophilicity.

3.7. Chloropicrin formation potential

Chloropicrin formation potential (CP-FP) results in all analysed water samples are presented in Fig. 7.

In raw waters, the CP-FP values were low and oscillated between 0.29 μg L^{-1} for the HA + FA model water sample and

1.30 µg L^{-1} for the KG surface water sample. The CP-FP was 1.15 µg L^{-1} for the HA model water sample and 0.46 µg L^{-1} for the FA sample. The highest removal level of CP-formation precursors in the MIEX®DOC process was observed in the treatment of the KG surface water sample (74%). For water samples containing humic acids (HA and HA + FA) the CP-FP removal efficiency was 45% and 40%, respectively. For the FA water sample, the lowest level of CP-FP removal was observed (9%).

In the study conducted by Gan et al. [17] on waters from water treatment facilities (30-min contact and 10 ml L^{-1} dose of the MIEX® resin) the reduction of halonitromethanes formation potential was achieved in the range 9%–33%. In that work, the formation potential of trichloronitromethane (chloropicrin) was not analysed. It is clear, however, that in comparison with the removal of THM and HAA precursors, the efficiency of CP-FP removal in the MIEX®DOC process is lower. CP precursors are organic compounds containing nitrogen. Neutral hydrophilic compounds and hydrophilic alkaline compounds are particularly rich in nitrogen [27], so relatively high efficiency of CP precursors removal in the MIEX®DOC process for the KG surface water might result from the removal of these fractions from treated water.

4. Summary and conclusions

The conducted research allowed evaluating the efficiency of removing precursors to the formation of organic water chlorination by-products in the MIEX®DOC process. The research was conducted on four types of raw water, and a strong correlation between raw water quality and removal levels of the disinfection by-product precursors has been observed. Taking into consideration the analyses of tested by-product groups and the precursors removal efficiency in the MIEX®DOC process, the study has concluded that:

- the most easily removed were precursors of compounds from the trihalomethanes group (68%–90%), haloacetic acids (76%–82%) and chloral hydrate (62%–88%),
- the less easily removed were precursors of haloacetonitriles (42%–72%),
- the least easily removed were precursors of compounds from haloketones (26%–73%) and chloropicrin (9%–74%) groups.

It has been confirmed that the organic matter content in raw water has a great impact on precursors formation. Therefore, prior to application of the MIEX®DOC process in water treatment facilities, it would be recommended to carry out research on the efficiency of removing disinfection by-products formation potential for raw water.

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