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Removal characteristics of organic matter during pretreatment for membrane-based food processing wastewater reclamation

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Abstract. In this study, we investigated coagulants such as polyaluminum chloride (PACl) and ferric chloride ($FeCl_3$) and the combination of a coagulant and powdered activated carbon (PAC) for the removal of dissolved organic matter (DOM) from fish processing effluent to reduce membrane fouling in microfiltration. The efficiency of each pretreatment was investigated through analyses of dissolved organic carbon (DOC) and ultraviolet absorbance at 254 nm (UVA_{254}). Membrane flux and silt density index (SDI) analyses were performed to evaluate membrane fouling; molecular weight distributions (MWD) and fluorescence excitation-emission matrix (FEEM) spectroscopy were analyzed to assess DOM characteristics. The results demonstrated that $FeCl_3$ exhibited higher DOC and UVA_{254} removals than PACl for food processing effluent and a combination of $FeCl_3$ and PAC provided comparatively better results than simple $FeCl_3$ coagulation for the removal of DOM from fish processing effluent. This study suggests that membrane fouling could be minimized by proper pretreatment of food processing effluent using a combination of coagulation ($FeCl_3$) and adsorption (PAC). Analyses of MWD and FEEM revealed that the combination of $FeCl_3$ and PAC was more efficient at removing hydrophobic and small-sized DOM.

Keywords: adsorption; coagulation; food processing wastewater; microfiltration; organic matter

1. Introduction

Water scarcity is one of the greatest global challenges today, because intensive industrialization, urbanization and rapid population growth, particularly in cities, have increased the pressure on water resources. Cho (2002) reported South Korea has experienced water-related issues in terms of both water quantity and quality. Water resource limitations due to population growth and industrialization can result in conflicts between stakeholders (Labadie 2007). In order to resolve water scarcity issues and to maintain a sustainable water supply, reuse of reclaimed wastewater is a promising solution (Garcia *et al*. 2015). Water reuse can not only address urbanization and water supply scarcity, but also attain efficient resource usage and environmental and public health protection (USEPA 2012).

For each ton of production, the food processing industry consumes much more water than other industrial sectors (Mavro *et al*. 2000). The food industry accounts for over 30% of manufacturing water use overall (Department of Agriculture Australia 2014). Furthermore, since these facilities use high-quality water and are commonly located near urban areas, it is important to emphasize wastewater reclamation and reuse in the food processing industry. The food processing industry produces a great deal of effluent, which can cause adverse environmental impacts, if not handled appropriately (Meneses *et al*. 2017). In many countries, strict environmental regulations have been applied to the food industry, thus increasing costs for wastewater treatment.

In general, industrial wastewaters are difficult to reuse, mainly due to the presence of hazardous chemicals and heavy metals; this is not the case for food industry wastewater. Wastewater from the food industry has some unique properties compared to municipal and other industrial wastewaters. It generally contains high levels of both organics and nutrients, which are generally biodegradable and nontoxic (Kotsanopoulos and Arvanitoyannis 2015). Therefore it can be readily treated by conventional biological treatment processes. Examples addressing the aforementioned conditions include: rinse water produced from start-up operations and final product rinse (Balannec *et al*. 2002), condensed water through evaporation (Vourch *et al*. 2008), cheese whey (Rektor *et al*. 2004), wastewater management in the food industry (Gugala *et al*. 2015), fresh-cut vegetable processing (Selma *et al*. 2008) and wastewater from poultry products (FAO/WHO 2007).

There are various technologies to remove dissolved salts and fine particles from food processing wastewater. Among them, membrane filtration has been widely used for reclamation because it can effectively remove particulate matter and microorganisms. Moreover, it has stable and perfect separation performance, even for salts and dissolved organic matter (DOM), when using reverse osmosis (RO)

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and nano-filtration (NF) membranes (Iaquinta *et al*. 2009, Galambos *et al*. 2004, Lee *et al*. 2009). However, high pressure requirements for membrane filtration systems result in high costs, a drawback of these types of systems. Membrane fouling can aggravate the cost and operational issues (Lee *et al*. 2005).

Membrane fouling in wastewater treatment is especially caused by improper pretreatment (Ishii *et al*. 2012, Shon *et al*. 2006). It is critical to reduce DOM contents during pretreatment prior to membrane filtration (ultrafiltration and/or microfiltration) for reclamation. While a combination of physicochemical and biological treatment is often used for pretreatment to reclaim wastewater (Gao *et al*. 2005), coagulation is an effective technique to remove organic matter and suspended solids in which coagulants, mainly iron and/or aluminum, are used (Chi *et al*. 2006). A combination of coagulation/sedimentation followed by adsorption has recently been attracting attention in the water treatment field to overcome the membrane fouling problem (Sakol *et al*. 2004).

In order to assess the best available pretreatment for membrane filtration in a food processing wastewater reclamation system, we investigated the removal efficiency of DOM by different coagulants and its impact on membrane fouling reduction. In this study, we proposed a coagulation/sedimentation technique using polyaluminum chloride (PACl), ferric chloride ($FeCl_3$) and a combination of $FeCl_3$ and powdered activated carbon (PAC) to minimize the membrane fouling encountered during the reclamation of food processing wastewater.

2. Materials and methods

2.1 Raw water and chemicals

Water samples were collected from a fish processing wastewater treatment plant in Pohang, South Korea. The facility treated 500 m^3/day of wastewater with a biological treatment process and the effluent from the facility was used as raw water for this study. The water quality characteristics of the effluent are summarized in Table 1. Coagulants such as PACl and $FeCl_3$ were supplied by Sigma Aldrich, U.S.A and the PAC was from Calgon Carbon, U.S.A.

2.2 Coagulation, adsorption and membrane filtration tests

Jar tests were conducted to evaluate the organic matter removal efficiencies by coagulation with PACl and $FeCl_3$. Also, a combination of $FeCl_3$ and PAC was tested with the same apparatus to evaluate the removal efficiencies by both coagulation and adsorption. The experiments were carried out in a six-jar tester (Nova Etica, Model 218 LDB) with digital mixing rod rotation control and simultaneous addition of reagents and sample collection. When jar testing PACl, $FeCl_3$ or a combination of $FeCl_3$ and PAC, we injected a 1 L-sample of raw water with a range of 0-100 mg PACl/mg Dissolved Organic Carbon (DOC), 0-50 mg

Table 1 Characteristics of raw water

Parameter	Value
pH	7.8
DOC (mg/L)	5.0
UVA at 254nm	0.089
SUVA	1.78
TDS (mg/L)	1,870
Alkalinity (mg/L)	135

$FeCl_3$/mg DOC and the same dosing rate for the $FeCl_3$ and Table 1. Characteristics of raw water PAC combination. First, rapid mixing was conducted at 120 rpm for 1 min, then slow mixing at 20 rpm for 20 min (Konradt *et al*. 2008), then the samples were allowed to settle for 1 h. Efficacy was calculated based on the removal efficiency of DOC and UVA at 254 nm (UVA_{254}). The membrane filtration tests were conducted at a constant transmembrane pressure of 100 kPa. A microfiltration membrane made from polyvinylidene fluoride (PVDF) with an effective area of 0.18 m^2 and a pore size of 0.1 μm was used. The feed water was forced through the membrane under pressure and the permeate was collected in a vessel mounted on an analytical balance. If not indicated otherwise, tests were conducted at 20°C.

2.3 Analytical methods

To examine water characteristics we analyzed pH, turbidity, electrical conductivity (EC) and other typical water quality parameters mainly using Standard Methods. The pH and EC were measured by a pH meter (Orion Star A series, Thermo Scientific, U.S.A) and a conductivity meter (Orion Star A212, Thermo Scientific, U.S.A), respectively. Alkalinity was measured by Standard Method No. 2320. Samples were pre-filtered through 0.7 μm glass fiber filters (GF/F) before DOC and UVA_{254} analysis. DOC was measured by a TOC analyzer (TOC-V, Shimadzu, Japan). UVA_{254}, an extensively used indicator of DOM (Humbert *et al*. 2005), was measured via UV spectrophotometer (HS3300, Humas, Korea).

Organic characteristics were assessed by fluorescence excitation-emission matrix (FEEM) and size-exclusion chromatography (SEC). FEEM analysis was conducted using a spectro-fluorophotometer (RF5301, Shimadzu, Japan) equipped with xenon lamps (slit interval: 10 nm, excitation range: 220-400 nm and emission range: 250-600 nm). SEC measurement was conducted through high performance liquid chromatography (HPLC, Futecs, Korea). The column used for the test was the Protein-Pak 125 (10 μm, 7.8 x 300 mm). The mobile phase was prepared with Na_2HPO_4 (1.135671 g), $NaH_2PO_4.H_2O$ (1.103938 g) and NaCl (23.37899 g) dissolved in 4 L of distilled water. Air in the mobile phase was removed using a sonicator. The flow of the mobile phase was 0.8 mL/min and 20 μL of sample was injected. The detector wavelength was 254 nm (Absorbance Detector UV730D, Younglin, Korea).

To evaluate membrane fouling potential, the silt density index (SDI) was evaluated using Eq. (1).

$$SDI = \frac{\left[1 - \left(\frac{t_i}{t_f}\right)\right]}{T} * 100 \quad (1)$$

Where T is the total elapsed flow time in min (typically 15 min), t_i is the initial time required to collect 500 mL of sample (s) and t_f is the time required to collect 500 mL of sample after test time, T (Kim *et al*. 2016).

3. Results and discussion

3.1 Removal of organic matter by PACl, $FeCl_3$ and PAC

The DOC and UVA_{254} analysis results for both coagulants (PACl and $FeCl_3$) are presented in Fig. 1. Overall, both DOC and UVA_{254} levels decreased with an increase in coagulant dosing rates and UVA_{254}, which represents the proportion of aromatic organic compounds, exhibited higher removal rates than DOC. The iron-based coagulant ($FeCl_3$) showed higher removal rates for both DOC and UVA_{254} than the aluminum-based coagulant (PACl). The addition of 50 mg $FeCl_3$/mg DOC achieved 50% removal of DOC, while the same dosing of PACl resulted in only a 10% DOC reduction. A similar trend was observed in the UVA_{254} analysis results for the $FeCl_3$ and PACl coagulants (Fig. 1). In this study $FeCl_3$ outperformed PACl in removing DOM from fish processing effluent. Thus, $FeCl_3$ was used for further investigation.

The high values of UVA_{254} observed indicated enrichment of water samples with hydrophobic and high molecular weight (MW) organic matter that could be removed through chemical coagulation (Yan *et al*. 2008). To reduce membrane fouling, the proportion of aromatic organic compounds should be reduced during pretreatment. For better membrane filtration performance, we investigated the influence of PAC addition on DOM removal during $FeCl_3$ coagulation. Fig. 2 represents DOM removal efficiencies (by UVA_{254}) by $FeCl_3$ coagulation and a combination of $FeCl_3$ and PAC. The results demonstrate that increasing the $FeCl_3$ dose reduced UVA_{254} and illustrate that PAC addition can enhance UVA_{254} removal, possibly by the adsorption of aromatic DOM by the PAC (Yan *et al*. 2008).

UVA_{254} removal efficiency was about 20% upon the addition of PAC alone at 10 mg/L, but the removal efficiency was enhanced to 40% or more in combination with 20 mg $FeCl_3$/mg DOC (Fig. 2). With higher dose of PAC (i.e., 30 mg/L), DOM removals further increased. Our results indicate that a combination of $FeCl_3$ and PAC provide comparatively better results than simple $FeCl_3$ coagulation for DOM removal from fish processing effluent.

3.2 Microfiltration of raw and pretreated waters: Flux and SDI

A flux reduction analysis was conducted to evaluate microfiltration performance after pretreatment. Fig. 3 presents the flux reduction over time for raw water and

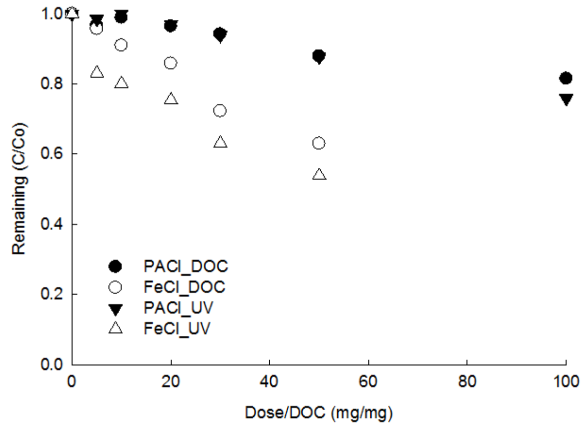


Fig. 1 Comparison of PACl and $FeCl_3$ to remove organic matters (DOC, UVA_{254})

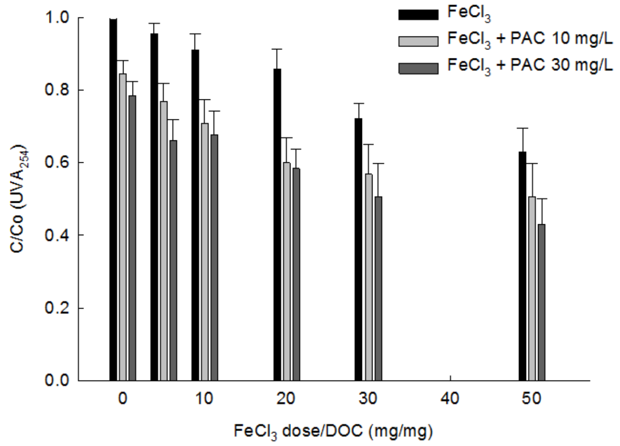


Fig. 2 Influence of PAC addition on the removal of DOM during $FeCl_3$ coagulation

pretreated water samples by 30 mg/L $FeCl_3$, 30 mg/L $FeCl_3$ + 10 mg/L PAC and 30 mg/L $FeCl_3$ + 30 mg/L PAC. Raw water caused the maximum flux reduction, followed by the 30 mg/L $FeCl_3$ treated water. The addition of PAC during $FeCl_3$ coagulation diminished the membrane flux reduction (Fig. 3). These results indicate the relationship between organic matter present in water samples and the flux reduction during microfiltration. Flux reduction was reduced as the concentration of organic matter decreased during pretreatment in the following sequence: 30 mg/L $FeCl_3$, 30 mg/L $FeCl_3$ + 10 mg/L PAC and 30 mg/L $FeCl_3$ + 30 mg/L PAC. Flux for raw water declines around 70% after 200 L/m^2 of filtration, while flux decline was 50% in case of a pretreated (30 mg/L $FeCl_3$ + 30 mg/L PAC) water (Fig. 3). The combination of $FeCl_3$ and PAC removed DOM more effectively in raw water versus either coagulant alone ($FeCl_3$), thus minimizing membrane flux reduction. For better understanding of the relationship between membrane flux and organic matter characteristics, SDI, molecular weight distribution (MWD) and FEEM were further investigated.

SDI can predict the efficiency of pretreatment systems prior to a microfiltration membrane process (Vial *et al*. 2003). The SDI value is considered to envisage the membrane fouling propensity of water samples as follows: SDI < 1 confirms clean operation and no colloidal fouling;

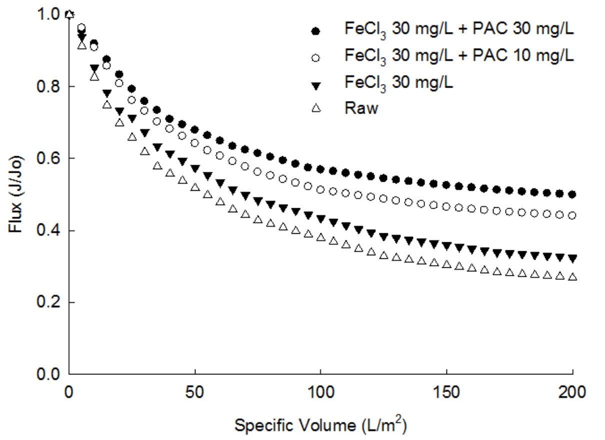


Fig. 3 Comparison of flux reduction for raw and pretreated waters

Table 2 Silt Density Index (SDI) results

Sample	SDI
Raw water	4.3-4.5
$FeCl_3$ 30 mg/L	3.8-4.0
$FeCl_3$ 30 mg/L + PAC 10 mg/L	3.4-3.6
$FeCl_3$ 30 mg/L + PAC 30 mg/L	2.9-3.1

SDI < 3, cleaning is required after months of operation; SDI 3-5, the fouling tendency is on higher side and the system requires frequent cleaning; SDI > 5, additional pretreatments are required (Park *et al*. 2007). In this study, SDI averaged 4.4 in raw water and reduced to 3.9 by the addition of $FeCl_3$-treated water (Table 2). The addition of PAC reduced SDI values as well: 3.5 with 30 mg/L $FeCl_3$ + 10 mg/L PAC and 3.0 with 30 mg/L $FeCl_3$ + 30 mg/L PAC (Table 2). These results demonstrate that SDI decreased via the coagulation process, especially by using a combination of $FeCl_3$ and PAC. This was likely due to the adsorption of organic matter and colloidal particles by PAC (Park *et al*. 2007). To identify the SDI difference between $FeCl_3$ and the combination of $FeCl_3$ and PAC-treated water, we investigated the characteristics of organic matter by performing MWD and FEEM analyses.

3.3 Characteristics of organic matter: MWD and FEEM

The MWDs of raw and pretreated waters are presented in Fig. 4. Most compounds in raw water were in the range of 100-10,000 daltons (Da). Although normalized signals for high MW (>50 kDa) do not show a distinct difference between raw and treated waters in Fig. 4, DOM with high MW (>50 kDa) was readily removed by coagulation (Table 3). More than 75% of compounds with MW > 50 kDa were removed by a combination of 30 mg/L $FeCl_3$ and 30 mg/L PAC. Molecules between 10-50 kDa were difficult to remove by $FeCl_3$ coagulation and PAC adsorption and portions of molecules 10-30 kDa even increased after coagulation. These were possibly formed by small molecules during coagulation that remained and did not settle (Sakol D. *et al*. 2004). Coagulation with $FeCl_3$ alone

Table 3 Percent remaining of DOM by MW after pretreatment

MW (Da)	$FeCl_3$	$FeCl_3$ 30 mg/L + PAC 10 mg/L	$FeCl_3$ 30 mg/L + PAC 30 mg/L
>100,000	43.1%	36.8%	9.7%
80,000-100,000	58.9%	46.6%	25.0%
50,000-80,000	79.7%	45.2%	25.5%
30,000-50,000	83.5%	106.0%	83.8%
10,000-30,000	165.3%	120.5%	146.3%
1,000-10,000	86.7%	76.9%	70.5%
100-1,000	94.6%	80.6%	67.9%
<100	106.9%	48.6%	36.0%

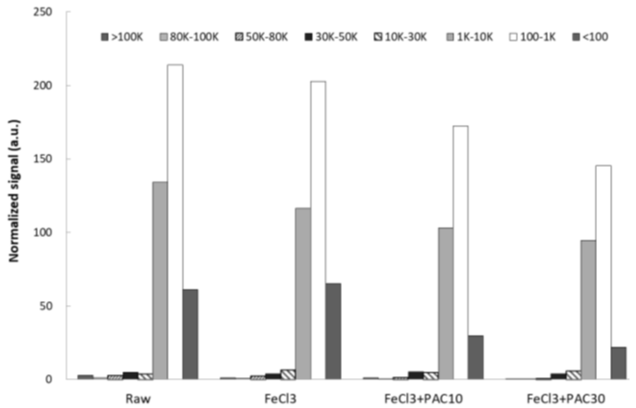
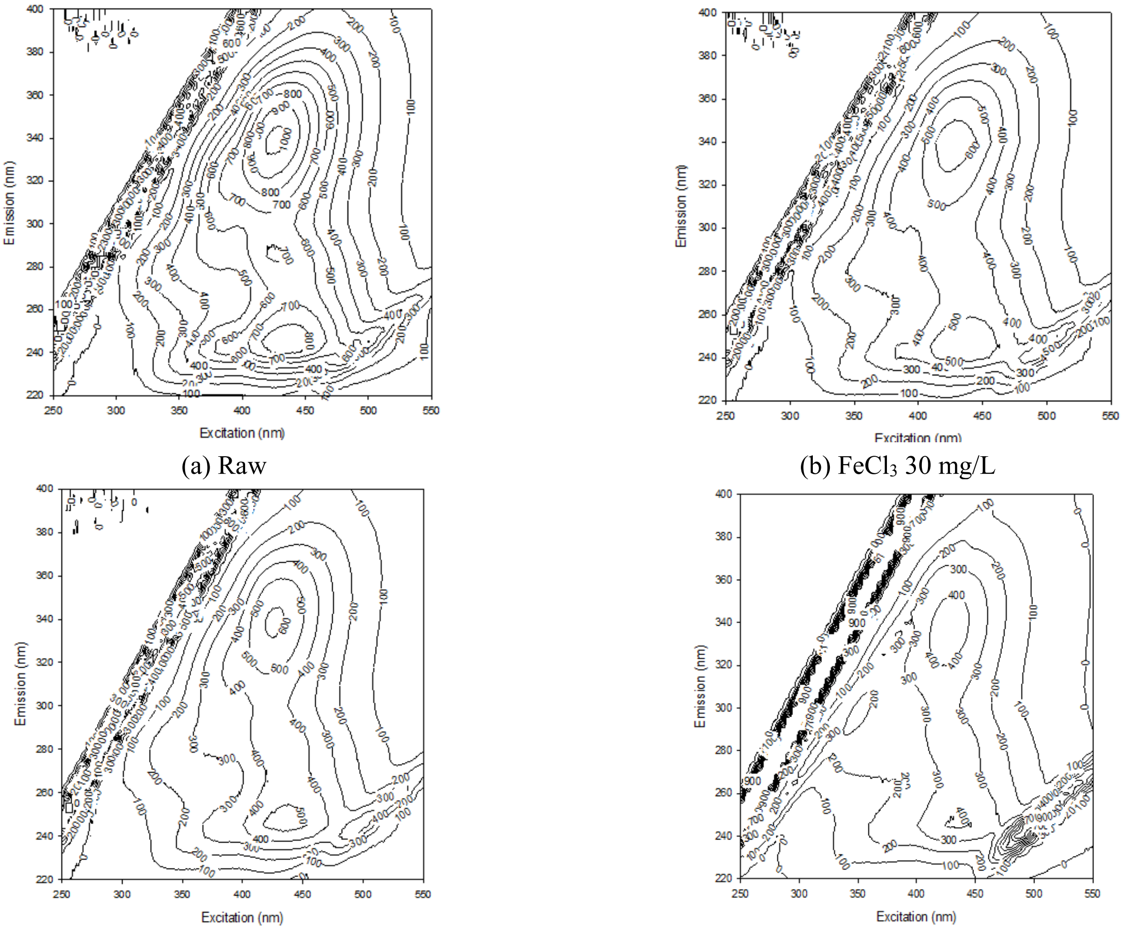


Fig. 4 Molecular weight distributions of raw and treated waters ($FeCl_3$ dose = 30 mg/L; PAC 10 means 10 mg/L of PAC; PAC30 means 30 mg/L of PAC)

could not efficiently remove DOM with low MW (< 1000 Da). On the other hand, the combination of $FeCl_3$ and PAC was more efficient at removing this organic matter because the PAC adsorbed the small-sized DOM (Lee *et al*. 2009).

The FEEM analysis of raw and pretreated waters was conducted to evaluate the types of organic matter in the water samples. Two distinct peaks observed for each sample lie in the humic acid-like (Excitation (ex) and emission (em), ex/em 430/340 nm) and fulvic acid-like (ex/em 430/245 nm) regions (Fig. 5). Raw water presented peak intensities of 1000 at the humic acid-like and 867 at the fulvic acid-like regions. The two peaks of the $FeCl_3$-coagulated sample dropped to 667 and 580, respectively, an approximately 35% reduction (Fig. 5b). The addition of PAC during $FeCl_3$ coagulation did not reduce the peaks significantly, although a higher dose of PAC achieved a relatively higher reduction. The two peak values of a sample with 30 mg/L $FeCl_3$ and 10 mg/L PAC were 615 and 535, respectively (Fig. 5c); the peaks were 490 and 410, respectively, with 30 mg/L of PAC addition during $FeCl_3$ coagulation (Fig. 5d). Thus, a major reduction in the organic materials in these regions was by $FeCl_3$ coagulation. These results imply different coagulation mechanisms for organic matter in fish processing effluent and charge neutralization may play a significant role in removing macromolecular substances that might belong to the organic regions (Jarusutthirak *et al*. 2007).



(a) Raw (b) $FeCl_3$ 30 mg/L

(c) $FeCl_3$ 30 mg/L + PAC 10 mg/L (d) $FeCl_3$ 30 mg/L + PAC 30 mg/L

Fig. 5 FEEM of (a) raw and (b, c, d) coagulated WWTP effluent

4. Conclusions

DOM removal efficiency by different coagulants and its impact on reducing membrane fouling were investigated for food processing wastewater reclamation.

- $FeCl_3$ exhibited higher DOC and UVA_{254} removals than PACl for the food processing effluent.
- The addition of PAC during $FeCl_3$ coagulation can significantly improve organic matter removals and it can be applied when influent organic concentrations are high.
- This study indicates that membrane fouling can be minimized by proper pretreatment of food processing effluent using a combination of coagulation (by $FeCl_3$) and adsorption (by PAC).
- WD and FEEM analyses revealed that the combination of $FeCl_3$ and PAC was more effective at removing hydrophobic and small-sized DOM. For real-life application of this process, long-term microfiltration performance with the proposed pretreatment needs to be evaluated.

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