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EFFECT OF PERSULFATE AND ACTIVATED PERSULFATE ON THE CHARACTERISTIC OF NATURAL ORGANIC MATTER FROM ALUM COAGULATION

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ABSTRACT

Surface water is mainly used as source water for drinking water purposes, and it may contain natural organic matter (NOM), which causes some problems in water treatment.The economically feasible method to remove NOM is coagulation.However, NOM is still remaining after coagulation. The remaining NOM can be further removed by enhanced coagulation and pretreatment coagulation through preoxidation. Persulfate has received increasing attention due to its activation will generate sulfate free radical, the strongest oxidants with the highest oxidation potential. The objective of this study was to characterize and examine dissolved organic matter in source and treated water, through persulfate and activated persulfate only, coagulation preceded by persulfate preoxidation with and without activation. Source water samples from the river was treated by coagulation, preceded by preoxidation persulfate with and without ferrous ion activation. Sample were analyzed for dissolved organic carbon (DOC), ultraviolet light absorbance at 254 nm (UV_{254}), specific UV absorbance (SUVA), and its functional group by using fourier-transform infrared (FTIR). The results showed that coagulation preceded by activated persulfate generated the highest removal of DOC and UV_{254}, and the relatively lower infrared absorption intensity than other treatments. It was conjectured that effect of the preoxidation by activated persulfate with ferro ion, which generated in situ-formed ferric ions, and alum coagulation contributed to the improved rejection of the organic compounds. Formation of sulfate radicals or in situ-formed ferric ions in activated persulfate without coagulation has a better performance to remove organic compound at 1200 cm^{-1} and 3440 cm^{-1} than alum coagulation only. This study concluded that activated persulfate could be used to enhance coagulation performance in removing organic matter in source water.

KEY WORDS : Activated persulfate, Coagulation, Spectroscopy, Organic matter surrogates

INTRODUCTION

Surface water is mainly used as source water for drinking water purposes, and it may contain natural organic matter (NOM). The amount and characteristic of NOM can vary in water differs with season and its sources, such as affected by rainfall intensity, hydrological regime, and other environmental factors. the character of NOM can vary with source and time (season), including raising air and surface water temperature, increasing in rainfall intensity (Sillanpää *et al.*, 2015). Discharge of untreated and treated wastewater into drinking water sources can change the composition of NOM. Coagulation, flocculation, sedimentation and sand filtration is the economically feasible and most commonly method to treat NOM. Coagulation has various mechanism processes to remove organic matter, algae species, and inorganic compound (Sillanpää *et al.*, 2018). However, NOM is still remaining after coagulation, due to low molecular weight and hydrophilic fraction in NOM is in ineffective to be removed by coagulation (Hidayah *et al.*, 2016). The remaining NOM can be further removed by enhanced coagulation and pretreatment coagulation through preoxidation (Xie *at al.*, 2016;

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Sillanpää *et al.,* 2018).

Preoxidation is widely used in water and wastewatern treatment, and it was known as an effective method in improving coagulation efficiency for the subsequent removal ofalgal cells and organic matter (Xie *et al.,* 2016; Hidayah *et al.,* 2017). It has been observed that preoxidation, which is performed through permanganate, ozone, chlorine dioxide, chlorine, could change the structure of algae cells and deactivate cell to promote their aggregation (Xie *et al.,* 2016). The mechanism of preoxidation were different for various oxidants, for example: preoxidation destroys the organic coating on the surface of particle and it causes the changing of zeta potential, damages the bond between particles and their adsorbed organics, reduces the higher to lower molecular weight of organic compound. In addition, polymerization of NOM via peroxidation may cause particle aggregation through bridging reactions. Permanganate preoxidation also produced manganese dioxides (MnO_2) in-situ, which can adhere to the cells and promote their settling (Hidayah *et al.,* 2017), however the higher dosage of permanganate could caused color problem in the treated water. Another preoxidant, chlorine formed a disinfectant by-products (DBPs), which have been identified as a carcinogenic and mutagenic compound (Hua and Reckhow, 2007).

Recently, the application of short-lived radical species as oxidant in disinfection and in remediation technology for soil and groundwater has gained attention due to their high redox potential and high reactivity. Persufateis one of the strongest oxidants and has the high oxidation potential (2.12 V), has received increasing attention (Ling *et al.,* 2017). Reaction of persulfate with NOM and micro pollutants are generally slow at ambient temperature, and it can be activated to produce sulfate free radical (SO_4^{-}), a stronger oxidant with a redox potential of 2.6 V, by thermal, chemical and photochemical technique (Liu *et al.,* 2018). Method of persulfate activation would influenced the rate of oxidant reaction.

Numoerous studies have investigated the effect of activated persulfate to remove pollutants. Thermal activation resulted in the highest reduction of polycyclic aromatic hydrocarbons (PAHs) under low persulfate consumption during thermal activation varied (Zhao *et al.,* 2013). It has been proposed that ultraviolet (UV) light could be applied to activate persulfate and the products has a potential to oxidize anthropogenic chemicals in water and emerging organic compound (Yang *et al.* 2017; Hou *et al.,* 2017). Persulfate preoxidation was very effective to control the formation of disinfection by-product (DBPs) in chlorination, while for chloramination, persulfate with ultraviolet activation has potential to reduce DBPs formation of in wastewater-impacted waters (Chu *et al.,* 2016). The study of radical-based advanced oxidation technologies has been observed as a promising methods to treat wastewater and in situ groundwater containing levofloxacin, with a particularly higher potential for the combined Fenton/persulfate system than Fe(II)-activated persulfate ($S_2O_8^{2''}/Fe^{2+}$)(Epold *et al.,* 2015). Another study showed that the Fe^{2+}-activated persulfate oxidation process can effectively degrade toluene in aqueous solutions (Long *et al.*, 2014) and increase membrane permeability (Cheng *et al.,* 2017). According to previous studies, it is summarized that persulfate as a novel oxidant has many advantages over the other well-known oxidants, especially the activated persulfate with ferrous ion. As a natural substrate, ferrous ion (Fe^{2+}) has been widely applied as a catalyst to activate persulfate effectively, instead of being inexpensive and nontoxic (Wu *et al.,* 2012; Long *et al.,* 2014; Cheng *et al.,* 2017). Moreover, activated persulfate by ferrous ion would generated ferric ions, which can act as *in-situ* coagulants, to promote the agglomeration of algal organics (Ma *et al.,* 2012).

In this study, persulfate was applied for preoxidation to enhance coagulation in water treatment processes. In our recent study (Hidayah *et al.,* 2017), coagulation preceded by permanganate oxidation was found to cause a greater reduction in all organic fractions as specified based on their molecular weight, than with coagulation alone. Since sulfate radicals has the higherstandard redox potential (Yan *et al.,* 2013) than permanganate, therefore sulfate radicals could be considered as alternative preoxidant for coagulation process in order to remove organic matter. In addition, according to our knowledge, it is a very limited research on the application of activated persulfate for coagulation pretreatment. NOM in source water and the treated water, from persulfate and activated persulfate only, coagulation preceded by persulfate preoxidation with and without activation using ferrous ion (Fe^{2+}), were examined by fourier-transform infrared (FTIR) besides bulk parameters analysis, such as dissolved organic carbon (DOC),

UV_{254}, and specific ultraviolet absorbance (SUVA) value.

MATERIALS AND METHODS

Source water samples was collected from Jagir River in Surabaya, during February to March 2018, which was taken once a week. As this study focused on the removal of dissolved organic carbon during peroxidation and coagulation, the collected source water was filtered through 1.2/0.5 µm and 0.45 µm (both from Millipore Corporation, USA) to eliminate any suspended solids before further usage. Coagulation with persulfate oxidation was carried out with pre-filtered source water to examine the effect of activation with and without ferrous ion (Fe^{2+}).

Stock solution of 100 mM sodium persulfate ($Na_2S_2O_8$) andiron (II) hepta hydrate ($FeSO_4.7H_2O$) (Merck, Germany) was prepared fresh. Persulfate with and without ferrous ion activation were conducted with a six-paddle gang stirrer jar test equipment (Phipps and Bird, Richmond, Virginia, USA). A specific aliquot from stock solution was diluted in the batch reaction to achieve an initial concentration 0.5; 1; 5; 10 mM persulfate and 0; 1; 2; 4 mM ferrous sulfate and added into the water-filled jar. The contact time was 5; 10; 20; 30; 40; 50; 60 minutes, under slow mixing at 35 rpm.

The same jar test apparatus was used for coagulation study. Six 1.0 L jars were filled with either persulfate with and without ferrous ion activation. Under 100 rpm rapid mixing, stock solution of alum ($Al_2(SO_4)_3.18H_2O$, Merck, Germany) were added with dosage 20 mg/L as Al. Rapid mixing was conducted at 100 rpm for 3 min, followed by reduction speed to 35 rpm for 15 min, then folowed by settling process for 30 min. The supernatants were collected, and filtered through 0.45 µm filter (cellulose acetate, Toyo Roshi, Japan) before further analysis.

The source water was measured for general physicochemical properties, such as dissolved organic carbon (DOC) by using total organic carbon analyzer (Model TOC-500, Shimadzu, Kyoto, Japan), ultraviolet absorbance at 254 nm (UV_{254}) by using UV/vis spectrophotometer (Model U-2001, Hitachi, Japan). Those measurement methods referred to the procedures from Standard Methods (APHA, 2012). SUVA values was obtained by dividing the UV_{254} absorbance value by the concentration of NPDOC. The structure, functional groups and organic characteristics was identified by FTIR spectroscopy (Thermo Nicolet NEXUS 670), scanning from 4000 cm^{-1} to 400 cm^{-1}. KBr pellets were prepared by grinding the powder samples (1%, wt%) of raw and treated water, with spectrographic grade KBr in an agate mortar. Freeze-drying method was used to obtain the powder sample.

RESULTS AND DISCUSSION

Effect of persulfate with and without ferrous ion activation on dissolved organic carbon

The removal efficiencies of DOC with various treatment processes was showed in Figure 1. As compared between Figure 1a and Figure 1c, when persulfate was activated by Fe, DOC removal became increased to almost 30%. This result indicated that activated persulfate exhibited a better result in DOC removal than without persulfate activation. Previous study found that activation of persulfate by Fe(II) simultaneously led to the formation of ferric ion Fe(III) andsulfate radical (SO_4^{-}). The formed ferric ions could acted as in-situ coagulant, which might enhance the agglomeration of organics (Xie *et al.,* 2016). Meanwhile, formation of sulfate radical, which is a strongest oxidant and reacted very rapidly with aromatic compounds as do hydroxyl radicals and H atoms with higher selectivity, could destroyed the organic coating on their surfaces through oxidation and change its zeta potential. Furthermore, reduction of DOC concentration probably due to sulfate radical oxidation could transformed higher molecular weight into lower molecular weight oforganic matter which is shown as reduction of DOC concentration (Hidayah *et al.,* 2017; Cheng *et al*. 2017).

Further, Fe(II)/PS with coagulation reduced the organic matter DOC higher (about 65%) than Fe(II)/PS only with less than 30% and coagulation preceded by persulfate peroxidation only with about 30%, as shown in Figure 1d, Figure 1c, and Figure 1b, respectively. This was probably due to effect of the preoxidation by activated persulfate with ferro ion, which generated in situ-formed ferric ions, and alum coagulation contributed to the increased rejection of the organic compounds (Wang *et al.,* 2016; Bu *et al.,* 2017). Alum coagulation could generated chemical interaction between alum as coagulants and functional groups present in the

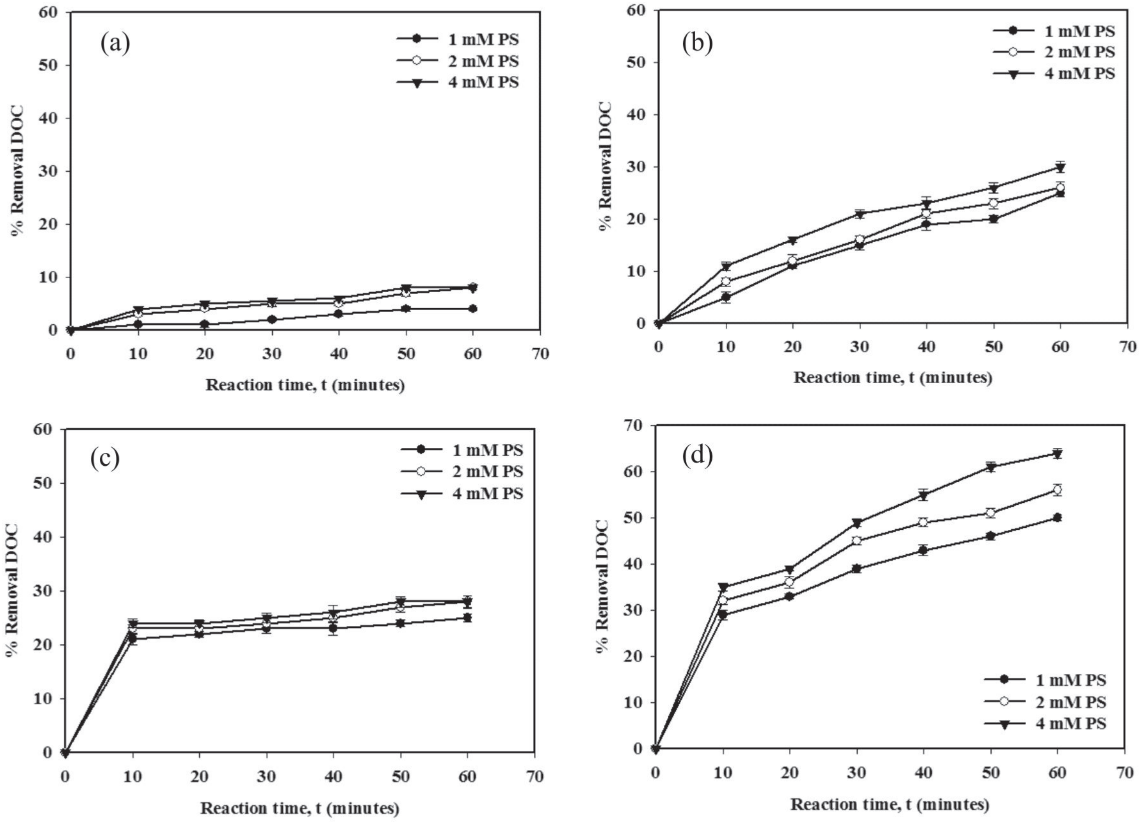

Fig. 1. Effect of persulfate concentration (a) without ferrous ion activation and without coagulation (NANC), (b) without ferrous ion activation and followed by cogulation (NAC), (c) with ferrous ion 4 mM activation and without coagulation (ANC), and (d) with ferrous ion activation 4 mM and followed by coagulation (AC), on DOC removal under various reaction time.

organic matter. The coagulation mechanism with alum probably occurred by charge neutralization through positively charged hydrolysed species or due to adsorption of humic substances on the the hydroxide precipitate (Edwards and Amirtharajah, 1985; Rigobello *et al.*, 2011). Another possibility of organic removal removal was described as complexation, which is Al within the flocs is in the form of uncondensed monomers. The Al speciation within aggregates formed in the presence of organic compound where the Al monomers were found to be the predominant species (Masion *et al.*, 2000). Meanwhile, increasing ferrous iron dosage corresponded to a higher available Fe^{2+} concentration, which could increased the in-situ formed ferric ions, and produced more sulfate radical than less dosages to activate persulfate.

NOM surrogates removal by alum coagulation preceded by persulfate oxidation

The performance of activated persulfate pretreatment followed by coagulation in the removal of DOC (Figure 2a), removal of UV_{254} (Figure 2b) and SUVA value (Fig. 2c) during water treatment with reaction time 30 minutes. First, the results suggested that the reduction of both parameters increased with increasing Fe(II) activation dosage and persulfate dosage. The highest DOC removal by activated persulfate/coagulation is about 55%, which indicated that persulfate could improved coagulation performance. It has been studied that DOC removal by coagulation about 20-40% (Edzwald, 1993), while $KMnO_4$ preoxidation followed by coagulation could remove DOC about 45-50% (Hidayah *et al.*, 2017). Second, Figure 2b showed the percentage of UV_{254} removal is about 70%, which is higher than that of DOC removal. It seems that activated persulfate demonstrated the best performance in UV_{254} reduction, which was probably caused by strong degradation by sulfate radical. UV_{254} featured molecules with aromatic structures and double bonds carbon, which was more UV light per unit DOC concentration, i.e. have a higher SUVA value, than the non-aromatic substances (Edzwald, 1993). It has been well-established in the literature that aromatic structures and conjugated C=C double bonds are more amenable to removal by preoxidation and or coagulation than non-aromatic compounds (Cheng *et al.*, 2017; Tian *et al.*, (a)

Third, Figure 2c showed SUVA value of the source water was 3.84 L/mg.m, which indicated that organic matter of these source water was to be composed mainly of mixture of non-humic and humic compound or mixture of hydrophilic and hydrophobic matter, though the value tends in an more dominant of humic and hydropbohic matter. Activated persulfate followed by coagulation process has in significantly performance in SUVA value, it shows decreasing SUVA value. However the organic properties is still between 2 – 4 L/mg.m, which still indicated mixture of hydrophilic and hydrophobic matter. Decreasing SUVA value probably represented the removal hydrophobic matteer and or humic compound. SUVA is an indicator of NOM composition of water has been denoted on the previous study (Edzwald and Tobiason, 2011). Source water with SUVA values ≥ 4 indicatedmainly of humic or hydrophobic matter, while waters with SUVA less than 2 contain mainly non-humic or hydrophilic NOM.

Fourier-transform infrared (FTIR) spectroscopic analysis

FTIR analysis was performed to determine whether organic compounds were removed and remained on the sample. The spectrum from the FTIR analysis of the raw water and treated water DOC sample was shown in Figure 3a. Major spectra bands or dominant functional groups were assigned as follows: 3440 cm^{-1} (aromatic N-H stretching; alcohols and carboxylic acid O-H stretching), 2430 cm^{-1} and 2340 cm^{-1} (nitrile Ca-N stretching), 1650 cm^{-1} (aromatic C=C and conjugated carbonyl C=O), 1200 cm^{-1} and 1120 cm^{-1} (alcohols and carboxylic C-O stretching; aliphatic amine C-N stretching). Diagnostic bands at 620 cm^{-1} is indicated as inorganic functional group PO_4^{-3} bending and sulfate bending (Smith, 1999). Therefore, the absorption spectrum clearly illustrated that the dominant functional groups alcohol, carboxylic acid and amino acids are the main functional group

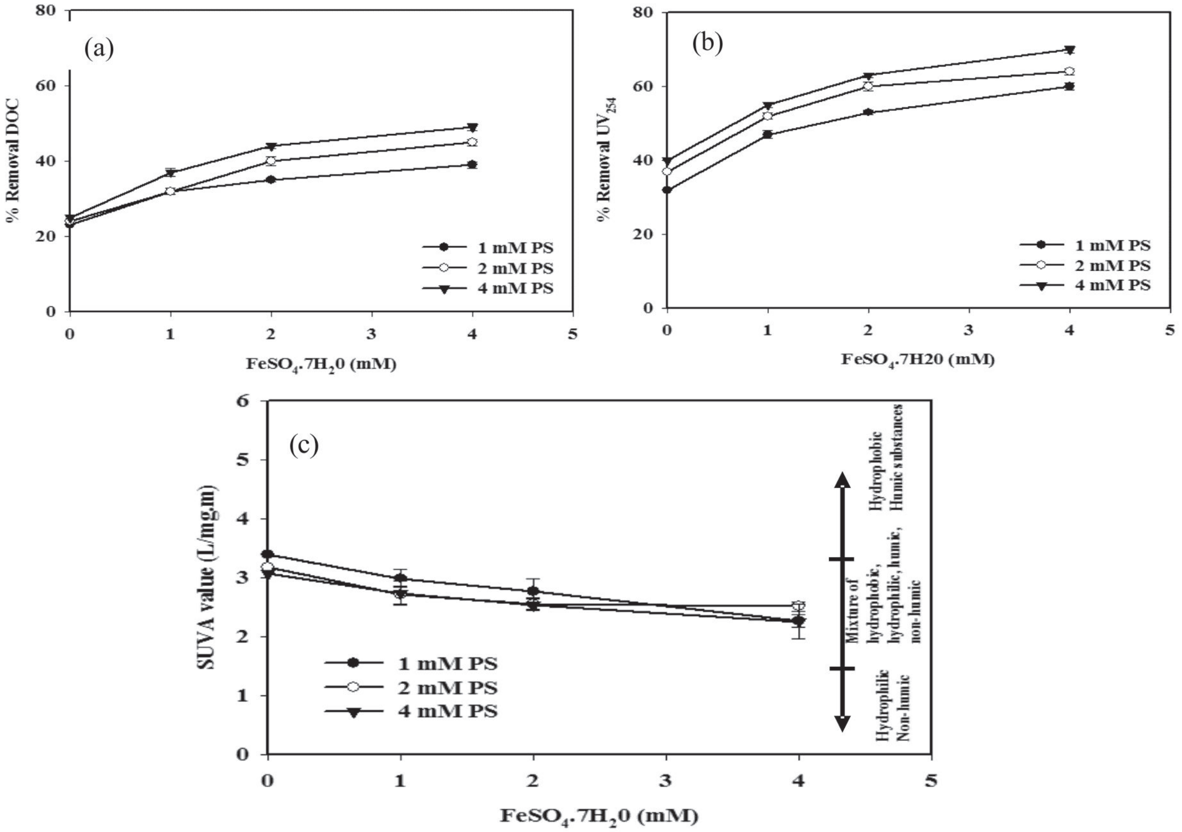

Fig. 2. Performance of coagulation preceded by various activated PS oxidation (AC) with reaction time 30 minutes for removing organic matter as represented by (a) DOC removal, (b) UV_{254} removal, (c) SUVA value

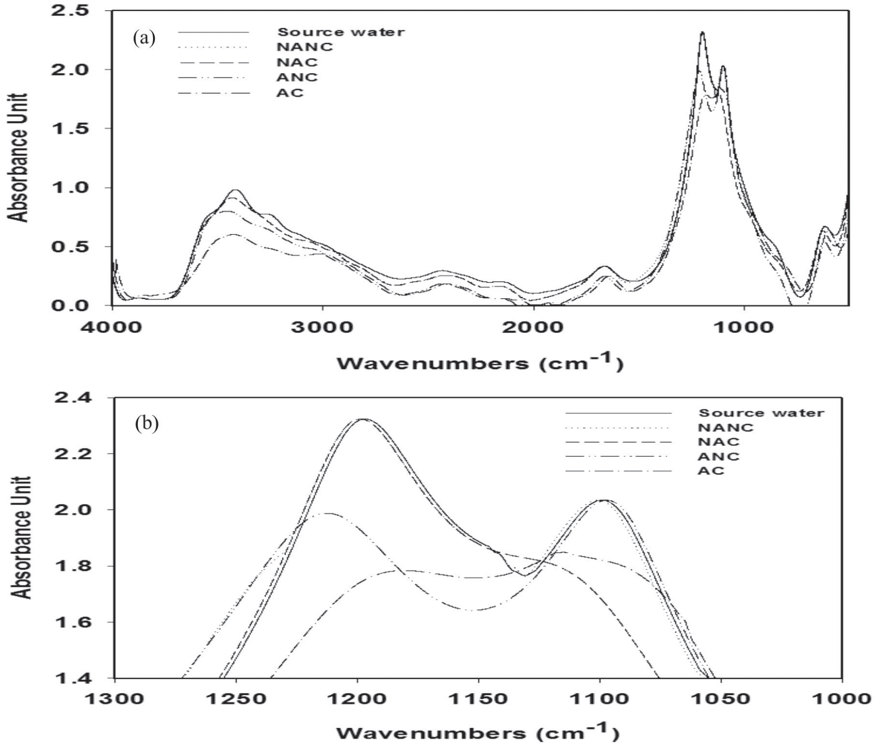

Fig. 3(a). FTIR spectrum of source water and treated water with persulfate 4 ppm under reaction time 30 minutes. (b) Magnification of spectrum at 1300 cm^{-1} to 1000 cm^{-1}

present in the raw water.

Combination results between bulk organic of DOC and UV$_{254}$ removal over FTIR spectral analysis of the organic indicated that activation of persulfate and coagulation process obviously affect the characteristic of organic matter in raw water. Activation persulfate followed by coagulation (AC) expectedly displayed a relatively lower infrared absorption intensity than other treatments, which showed a consistentcy with the highest removal of TOC and UV$_{254}$. While, treatment without activated persulfate and without coagulation (NANC) had the highest absorption intensity, even similar with raw water intensity. It showed a little decreasing of absorption spectra at 2430 cm^{-1} and 2340 cm^{-1}. As shown in the magnification of spectrum at 1300 cm^{-1} to 1000 cm^{-1} (Figure 3b), the absorption intensity at 1120 cm^{-1} showed insignificantly removal under activated persulfate without coagulation process. This result was contrast with coagulation without preceded by activation persulfate. It seems that hydrolyzed species of alum has a higher capacity in removing functional groups at 1120 cm^{-1} than in situ-formed ferric ions or sulfate radicals. However, either formation of sulfate radicals or in situ-formed ferric ions (activated persulfate without coagulation) has a better performance to remove organic compound at 1200 cm^{-1} than alum coagulation (without activated persulfate with coagulation), and remove functional groups at 3440 cm^{-1} as well. These results suggested that applied treatment processes in this study has various mechanism removal for the functional groups of organic matter.

CONCLUSION

This study found that coagulation preceded by preoxidation with activated persulfate was found to cause a greater reduction in all organic bulk parameters and spectrum from the FTIR analysis. Reduction of bulk organic parameters is probably due to activation of persulfate by Fe(II) simultaneously caused of the generation of ferric ion Fe(III), and sulfate radical ($SO_4^{\cdot-}$). Both of generated product could enhance coagulation performance in removing natura organic matter. Combination results between bulk organic of DOC and UV$_{254}$ removal over FTIR spectral analysis shown that coagulation preceded by activated persulfate displayed a relatively lower infrared absorption intensity than other treatments, which is consistent with the highest removal of TOC and UV$_{254}$. The proxidant activatedpersulfate and coagulation process obviously affect the characteristic of organic matter in raw water. In order to remove organic matter in water treatment, activated persulfate could be considered as alternative preoxidant for coagulation process, and as pretreatment for membrane process. Further, effect of activated persulfate to DBPs formation could be conducted in the future study since the characteristic oforganic matter, a precursors of DBPs formation, has been changed through preoxidation by activated persulfate.

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