Removal of fluorescence and ultraviolet absorbance of dissolved organic matter in reclaimed water by solar light

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ABSTRACT

Storing reclaimed water in lakes is a widely used method of accommodating changes in the consumption of reclaimed water during wastewater reclamation and reuse. Solar light serves as an important function in degrading pollutants during storage, and its effect on dissolved organic matter (DOM) was investigated in this study. Solar light significantly decreased the UV_{254} absorbance and fluorescence (FLU) intensity of reclaimed water. However, its effect on the dissolved organic carbon (DOC) value of reclaimed water was very limited. The decrease in the UV_{254} absorbance intensity and FLU excitation–emission matrix regional integration volume (FLU volume) of reclaimed water during solar light irradiation was fit with pseudo-first order reaction kinetics. The decrease of UV_{254} absorbance was much slower than that of the FLU volume. Ultraviolet light in solar light had a key role in decreasing the UV_{254} absorbance and FLU intensity during solar light irradiation. The light fluence-based removal kinetic constants of the UV_{254} and FLU intensity were independent of light intensity. The peaks of the UV_{254} absorbance and FLU intensity with an apparent molecular weight (AMW) of 100 Da to 2000 Da decreased after solar irradiation, whereas the DOC value of the major peaks did not significantly change.

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Introduction

Wastewater reclamation and reuse plays an important role in solving water shortage problems in cities of many countries, including China. Storing reclaimed water in lakes is an important method of accommodating changes in the consumption of reclaimed water during the reclamation and reuse of wastewater (Marks, 2006; Taigbenu and Ncube, 2005).

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Water quality may change during storage; for example, suspended solids in reclaimed water may settle, and nutrients, including nitrogen and phosphorus, may induce algae growth in the lake (Hernández et al., 2006; Ma et al., 2005). As such, the changes in water quality during storage are important considerations for the final use of reclaimed water.

The photodegradation of pollutants by solar light is an important method of controlling pollutants during the storage of reclaimed water. Light plays an important role in transforming some micro-pollutants, including fluoroquinolone antibiotics and triclosan (Buth et al., 2010; Amine-Khodja et al., 2006; Liu and Guan, 2012). The half-life time of the photodegradation of fluoroquinolone during solar light irradiation in pure water even ranges from 1.25 to 58 min (Ge et al., 2010). Solar light can transform triclosan in surface water into harmful dioxin-like pollutants, which are then concentrated in sediments (Buth et al., 2010). The experiments on the fates of micro-pollutants during solar light irradiation indicate that solar light serves as an important function in transforming pollutants in surface water.

Dissolved organic matter (DOM) is an important component in reclaimed water; it contributes to the formation of the precursors of disinfection byproducts, assimilable organic carbon, and membrane-fouling pollutants (Tang et al., 2010; Wu et al., 2009; Liang and Song, 2007; Lin et al., 2009). Solar light degrades natural humic acid, including Elliott soil fulvic

acid (Halladja et al., 2009). During solar light irradiation, some organic nitrogen pollutants are transformed into ammonium and nitrate, which are associated with the eutrophication of water environments (Bronk et al., 2010). The composition of DOM in reclaimed water is very complex, and soluble microbial products and undegradable pollutants are present in reclaimed water (Barker and Stuckey, 1999). Therefore, the effects of solar light on DOM in reclaimed water may be very complex.

The spectrum, hydrophobicity, and molecular weight of DOM have been used to characterize the potential risk changes of reclaimed water, such as the formation of disinfection byproducts and increase in toxicity (Henderson et al., 2011). Ultraviolet (UV) absorbance and fluorescence excitation-emission matrix regional integration volume (FLU volume) are correlated with the chlorination anti-estrogenic activity formation potential and the formation of disinfection byproducts (Tang et al., 2010, 2014). Research on the precursors of disinfection byproducts shows that during chlorination, DOM with molecular weights of lower than 1 kDa play a major role in the formation of N-nitrosodimethylamine and increase of genotoxicity (Mitch and Sedlak, 2004; Wu et al., 2010). This finding indicates that the molecular weight of DOM is also an important factor that is associated with the formation of disinfection byproducts. However, research on the effects of solar light on UV absorbance, FLU spectra, and DOC in reclaimed water remains limited. The changes in
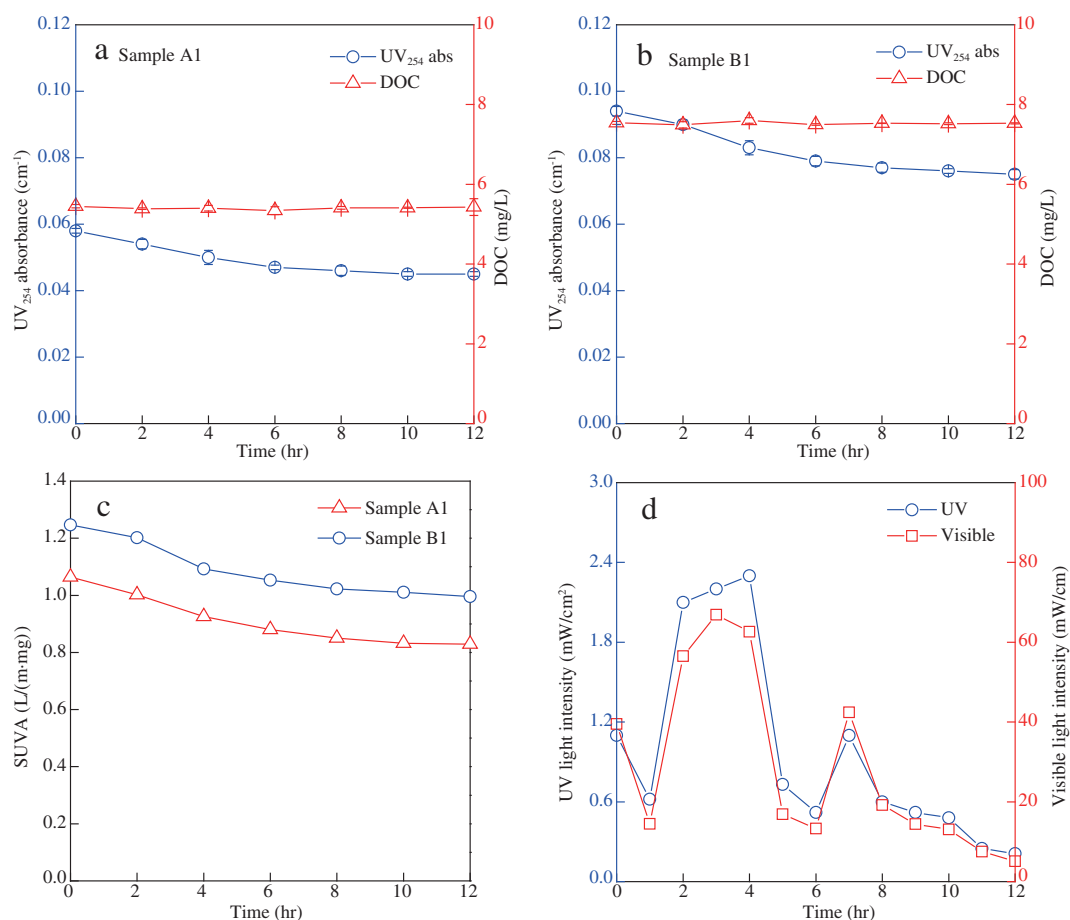

Fig. 1 – Changes of UV$_{254}$ absorbance, dissolved organic carbon (DOC) values, and Specific ultraviolet absorbance (SUVA) values in the reclaimed water samples during natural solar light irradiation.

molecular weight distribution with UV absorbance and FLU, and particularly the DOC of DOM during solar light irradiation, are still unknown.

The present study evaluated the removal of UV absorbance, FLU volume, and DOC in reclaimed water during natural solar light irradiation. The changes in the molecular weight distribution of DOM in reclaimed water during solar irradiation were investigated using size exclusion chromatography and UV_{254} absorbance, FLU, and DOC detectors. The kinetic constants of UV absorbance and FLU for irradiation experiments were obtained, and the effects of light wavelength (λ) and intensity on UV absorbance and FLU volume removal were evaluated.

Fig. 3 – Removal kinetics of UV_{254} absorbance intensity and dissolved organic carbon (DOC) in reclaimed water samples (A2 and B2) after simulated solar light irradiation by xenon lamp. The light intensities of ultraviolet light and visible light in simulated solar light were 2.80 mW/cm^2 and 70.00 mW/cm^2, respectively.

1. Materials and methods

1.1. Water samples

The reclaimed water samples A1 to A3 were obtained at three different times from the UV light disinfection effluent in a reclaimed water treatment plant that employs the anaerobic–anoxic–oxic process, coagulation, filtration, and UV light disinfection. The reclaimed water samples B1 to B3 were also obtained from UV light disinfection effluent of a reclaimed water treatment plant using biological filters, high rate compact water clarification after flocculation with microsand and polymer (ACTIFLO®, VEOLIA), and UV light disinfection at three different times. The UV light disinfection effluents from treatment plants were discharged into rivers
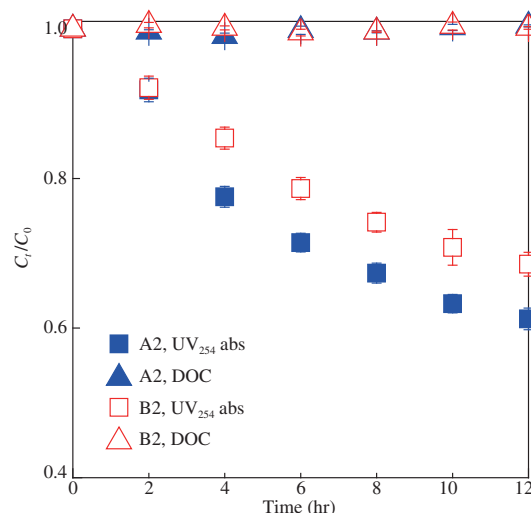
and ponds and then used as reclaimed water samples. The samples were filtered through microfilters (0.45 μm) and then stored at 4°C for irradiation experiments. The water quality of reclaimed water samples is shown in Appendix A Table S1.
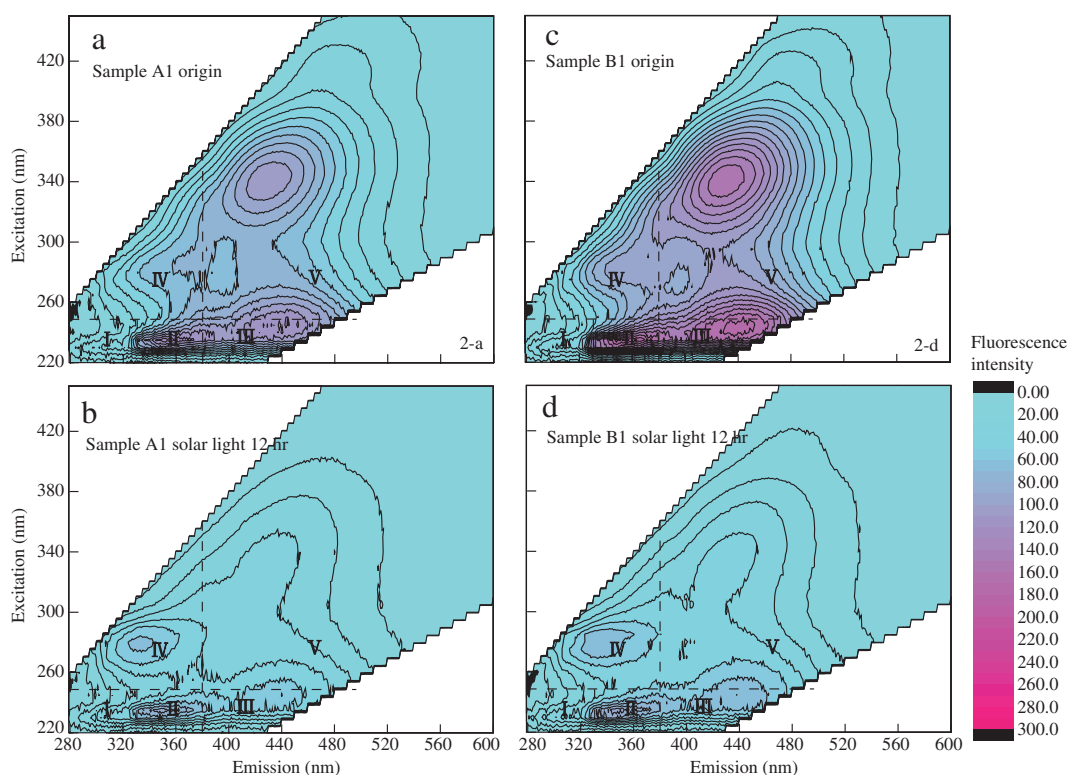

Fig. 2 – Excitation emission matrix (EEM) fluorescence spectra of reclaimed water samples A1 and B1 before (a, c) and after (b, d) natural solar light irradiation for 12 hr. Fluorescence regions I, II, III, IV and V represent tyrosine-like proteins, tryptophan-like proteins, fulvic acid, microbial metabolites and humic acid, respectively.

1.2. DOM irradiation experiments by natural solar light

Quartz glass tubes with 50 mL of reclaimed water were exposed to natural solar light for 12 hr (6:45 A.M. to 6:45 P.M.) in summer at N22°35, E113°58 of Shenzhen (China). The temperatures of the water samples during solar light irradiation ranged from 22.9 to 27.5°C. The light intensities of the solar light with wavelengths of 275 to 330 nm, 320 to 400 nm, and 400 to 1000 nm were found to be 0.06–1.01 mW/cm^2, 0.07–1.31 mW/cm^2 and 5.21–72.34 mW/cm^2 by UV-B, UV-A, and FZ-A irradiance meters (Beijing Normal University Photoelectric Instrument Factory, China), respectively. The experiments involving DOM irradiation by natural solar light were performed in duplicate. The water samples for the UV$_{254}$ absorbance, FLU spectra, and DOC analyses were obtained after light irradiation.

1.3. Kinetic experiments on the irradiation experiments of DOM by simulated solar light

The water samples (50 mL) were stored in quartz glass tubes and were exposed for 12 hr to simulated solar light provided by a xenon lamp (1 kW) in a XPA-7 Photochemical Reactor (Xujiang Electromechanical Plant, Nanjing, China) to obtain the kinetic parameters for the photodegradation of DOM. Kinetic experiments on the irradiation experiments of DOM were performed in duplicate. The temperature and pH of the water samples during light irradiation were maintained at 25.0 ± 0.5°C and 7.0±0.3, respectively. The water quality and light intensity were determined during light irradiation. The light intensities for the wavelengths of 275 to 330 nm, 320 to 400 nm, and 400 to 1000 nm from the xenon lamp were 1.21, 1.59, and 70.00 mW/cm^2, respectively.

The water samples were exposed to visible light ($\lambda > 420$ nm) and UV light to determine the effect of light wavelength on DOM irradiation experiments. Visible light ($\lambda > 420$ nm) was provided through filtration of simulated solar light (xenon lamp) with a UV light filter, and UV light was provided by a mercury lamp with a light filter.

1.4. Analysis of UV absorbance, dissolved organic carbon, and FLU spectra of DOM

The UV$_{254}$ absorbance and DOC values of the water samples after light irradiation were determined using a UV–Vis spectrophotometer (UV-2450, Shimadzu, Japan) and TOC analyzer
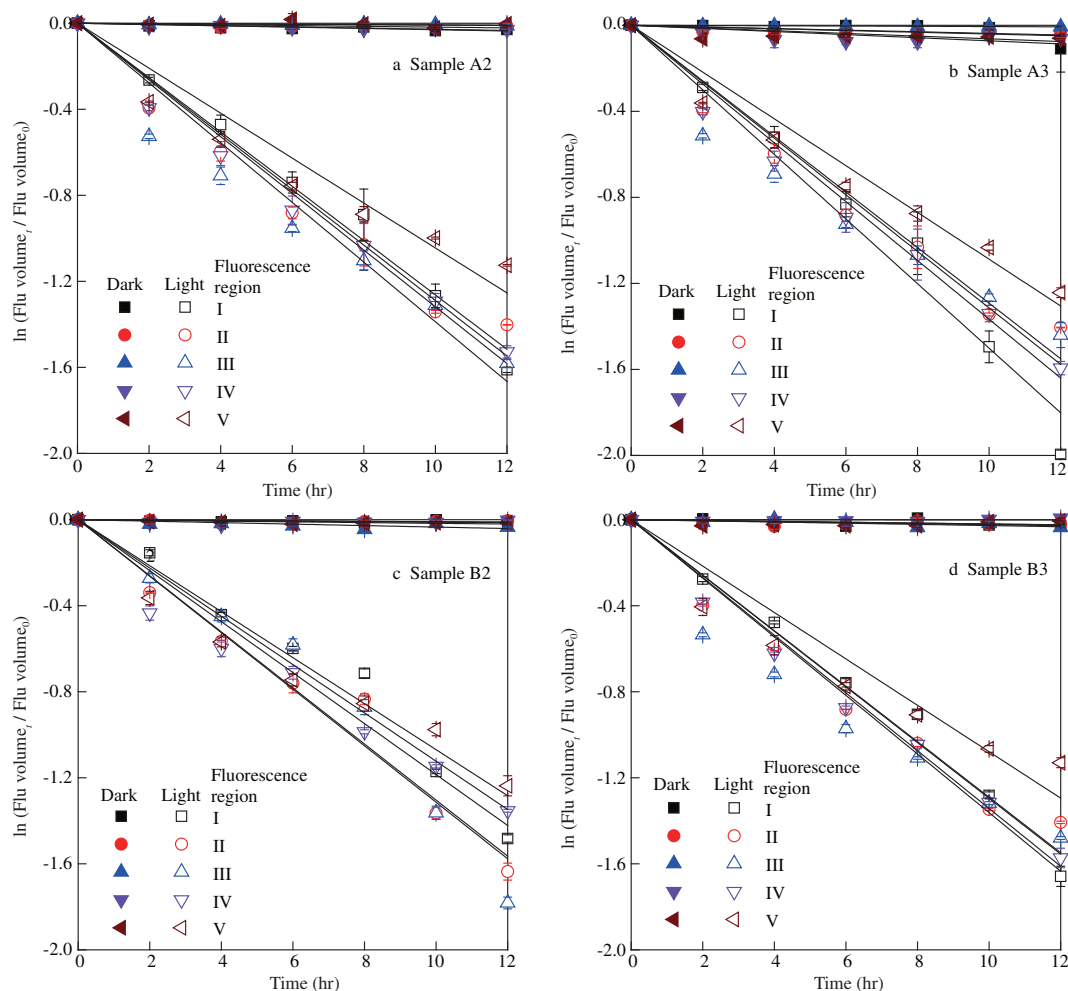

Fig. 4 – Removal kinetics of fluorescence volumes in reclaimed water samples after simulated solar light irradiation by xenon lamp for 12 hr. The light intensities of ultraviolet light and visible light in simulated solar light were 2.80 mW/cm^2 and 70.00 mW/cm^2, respectively.

(TOC-V$_{CPH}$, Shimadzu, Japan). The excitation emission matrix of the FLU spectra of the water samples was determined using a FLU spectrophotometer (UV-5301PC, Shimadzu, Japan), and the fluorescence data were then transformed into FLU volumes of regions associated with tyrosine, tryptophan, and humic/fulvic acids, following previous studies (Tang et al., 2014; Chen et al., 2003). The FLU volume was obtained by integrating FLU intensities of peaks in the related region.

1.5. Size exclusion chromatography (SEC) with UV$_{254}$, FLU, and DOC analyses

The molecular weights of organic matter in the water samples after solar light irradiation were characterized by High Performance Liquid Chromatography (LC20, Shimadzu, Japan) using two sizes of exclusion columns (a combination of Tsk-Gel G3000PWXL and Tsk-Gel G2500PWXL, Japan), UV array, FLU detectors, and a modified Total Organic Carbon detector (Sievers 900, GE, USA), following previous research (Wang et al., 2014; Lou and Xie, 2006). The injection volume was 100 μL. The elution program was performed with a mobile phase solvent of water solution containing NaH$_2$PO$_4$ (2.4 mmol/L), Na$_2$HPO$_4$ (1.6 mmol/L), and Na$_2$SO$_4$ (25 mmol/L) with a flow rate of 0.5 mL/min. The wavelength of the UV detector was 254 nm. The excitation/emission (Ex/Em) wavelength pairs of the FLU detector were 235/300 nm, 235/355 nm, 245/420 nm, 340/430 nm, and 280/355 nm, which were used to characterize the DOM associated with tyrosine-like protein, tryptophan-like protein, fulvic acids, humic acids, and soluble microbial byproducts, respectively (Chen et al., 2003). The apparent molecular weights (AMW) at specific retention times of the SEC chromatographs based on UV, fluorescence, and TOC detectors were calibrated with polystyrene sulfonate standards (3000 Da, 7000 Da, 13,000 Da, and 40,000 Da, American Polymer Standard). With calibration curves, the AMW distributions were calculated from the retention times, and then were divided into three regions as AMW < 400 Da, 400 Da < AMW < 900 Da, AMW > 900 Da. Thereafter, the integration of UV$_{254}$, FLU and TOC intensities in the three regions was performed.

2. Results and discussion

2.1. Changes in UV$_{254}$ absorbance and FLU of reclaimed water during solar light irradiation

The UV$_{254}$ absorbance and DOC values of the reclaimed water samples after irradiation by natural solar light are shown in Fig. 1. The intensities of UV light and visible light in solar light ranged from 0.13–2.31 mW/cm^2 and 5.21–72.34 mW/cm^2 as shown in Fig. 1d. The changes in both the UV$_{254}$ absorbance and DOC values during 12 hr of storage under dark conditions were not significant. The UV$_{254}$ absorbance values of the samples A1 and B1 decreased significantly after 12 hr of solar light irradiation, with a removal efficiency of 22% and 20%. However, the removal of DOC was very limited. Therefore, the Specific ultraviolet absorbance (SUVA) values of the samples A1 and B1 also decreased significantly after solar light irradiation (Fig. 1c). UV$_{254}$ absorbance has been used to characterize chromophores in river water (Stolpe et al., 2010).

In this study, the decrease in UV$_{254}$ absorbance during light irradiation indicated that solar irradiation decreased the chromophores in DOM. These findings are supported by
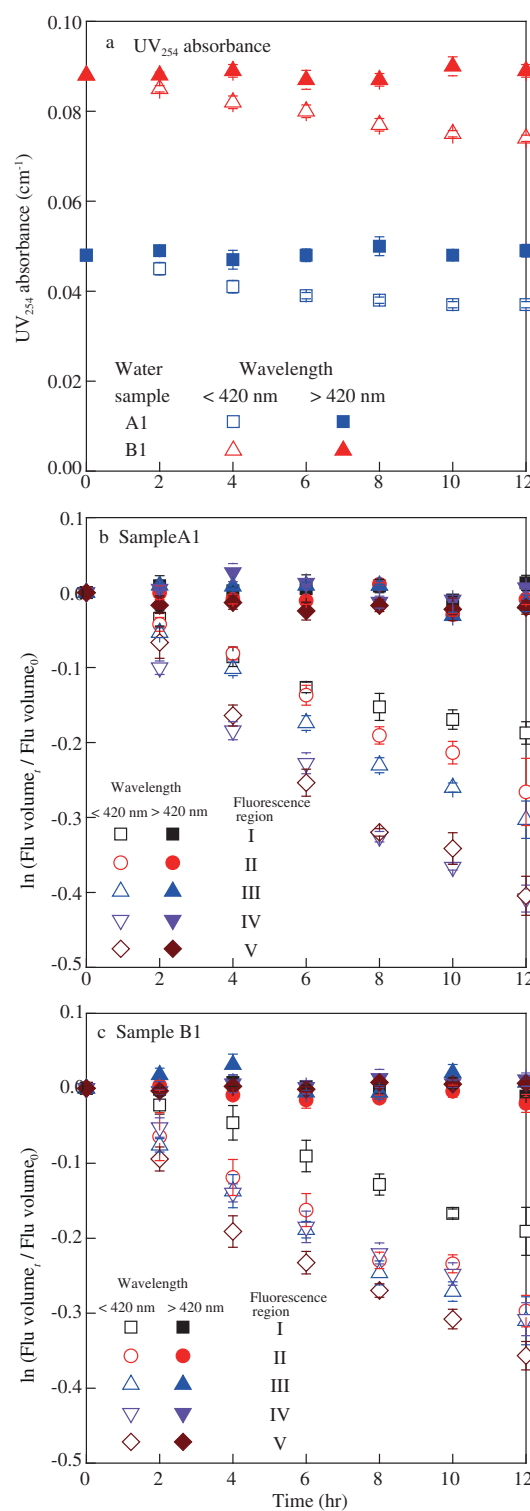

Fig. 5 – Effects of solar light with different wavelengths on UV$_{254}$ absorbance removal in reclaimed water samples A1 and B1 during light irradiation. The light intensities of ultraviolet light and visible light were 0.60 mW/cm^2 and 53.00 mW/cm^2, respectively.

previous studies, which found that natural solar irradiation significantly decreases the UV_{254} absorbance values of DOM in natural rivers, with limited DOC removal efficiency (Corin et al., 2000; Meng et al., 2013). Some previous studies indicated that the disinfection byproduct formation potential of reclaimed water exhibited a correlative relationship with the characteristics of UV_{254} absorbance and fluorescence (Chu et al., 2002; Van Huy et al., 2011).

The changes in the FLU spectra of reclaimed water during natural solar irradiation are shown in Fig. 2. Previous findings detected the presence of humic acid, tryptophan, and tyrosine-like protein/amino acids in reclaimed water before solar irradiation (Chen et al., 2003). The fluorescent intensities of reclaimed water were similar to the original values (data not shown) after stored under dark conditions. However, the FLU intensities of the reclaimed water significantly decreased after solar irradiation.

The changes in the FLU volumes of samples A1 and B1 are shown in Appendix A Fig. S1 in the Supplementary Material. The intensities in region V, which are associated with humic acid in reclaimed water samples A1 and B1, decreased by 49% and 60%, respectively. The removal efficiencies of the FLU volumes related to microbial metabolites, tryptophan-like proteins, and fulvic acid reached 30% to 40% for sample A1 and 40% to 54% for sample B1. Solar light also reduced the FLU intensity of oceanic DOM with a removal efficiency of 70% to 80% after 68 days of irradiation (Helms et al., 2013; Kowalczuk et al., 2013; Mounier et al., 1999).

In the present study, natural solar light quenched the FLU spectra of DOM in reclaimed water, indicating that natural solar light can degrade the fluorescent structure of DOM

and form some products without FLU. During chlorine disinfection, FLU exhibited a positive relationship with the anti-estrogenic activity and disinfection byproduct formation potential of reclaimed water (Tang et al., 2014; Hao et al., 2012). Thus further investigations on the anti-estrogenic activity and disinfection byproduct formation potential of reclaimed water should be conducted, and the relationship of formation potential and fluorescent structures of DOM in reclaimed water should be investigated in solar irradiation conditions.

2.2. Kinetics of UV_{254} absorbance and FLU removal during the irradiation of reclaimed water by simulated solar light

Simulated solar light was used in the irradiation experiments to evaluate the kinetic constant of UV_{254} absorbance and the DOC of reclaimed water. The UV_{254} absorbance and DOC values of the reclaimed water samples after 0 to 12 h of irradiation by simulated solar light are shown in Fig. 3. The intensity of simulated solar light was similar to the highest level of natural solar light as shown in Fig. 1d. After irradiation by simulated solar light, the UV_{254} absorbance values of the samples decreased significantly, whereas their DOC values were similar to the original values.

The decrease in the UV_{254} absorbance value during irradiation by simulated solar light could be fit by pseudo-first order reaction kinetics, as shown in Fig. 3. The kinetic constant of the value of the UV_{254} absorbance removal under irradiation by solar light ranged from 3.40×10^{-2}/hr to 4.60×10^{-2}/hr.

The changes in the 5-region FLU volumes of the reclaimed water samples during irradiation by simulated solar light for 0
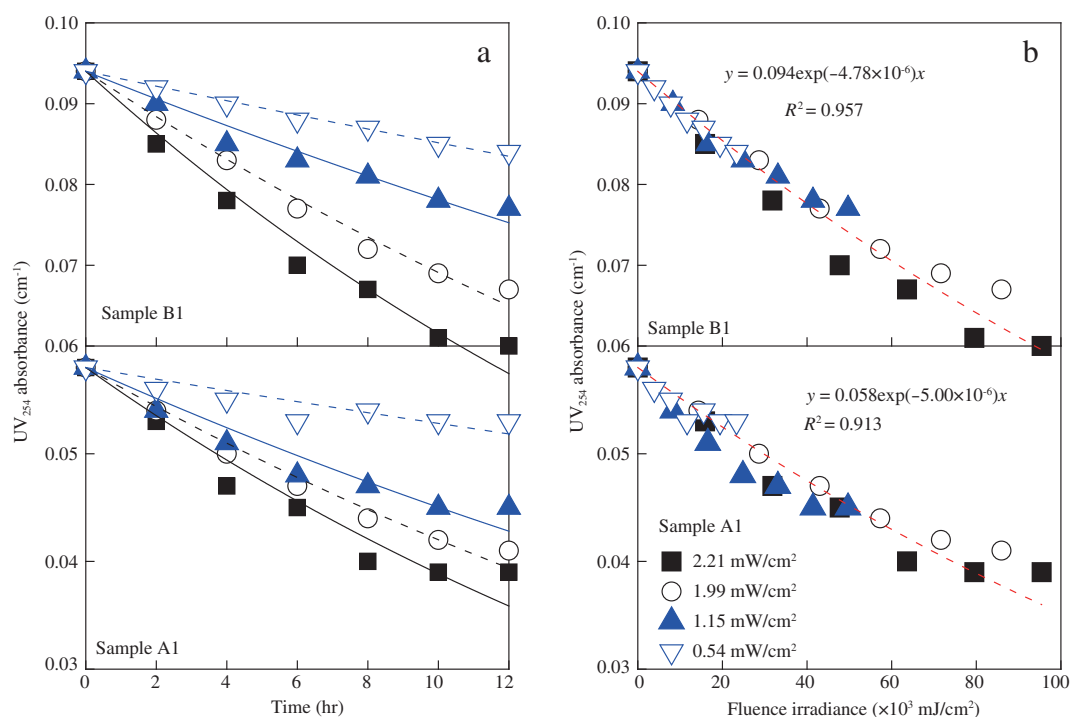

Fig. 6 – Simulated solar irradiation induced degradation of UV_{254} absorbance at different irradiation intensity: (a) irradiation time-based irradiation experiments kinetics and (b) UV light irradiation fluence-based irradiation experiments kinetics. The light intensities of ultraviolet light ranged from 0.54 mW/cm^2 to 2.21 mW/cm^2.
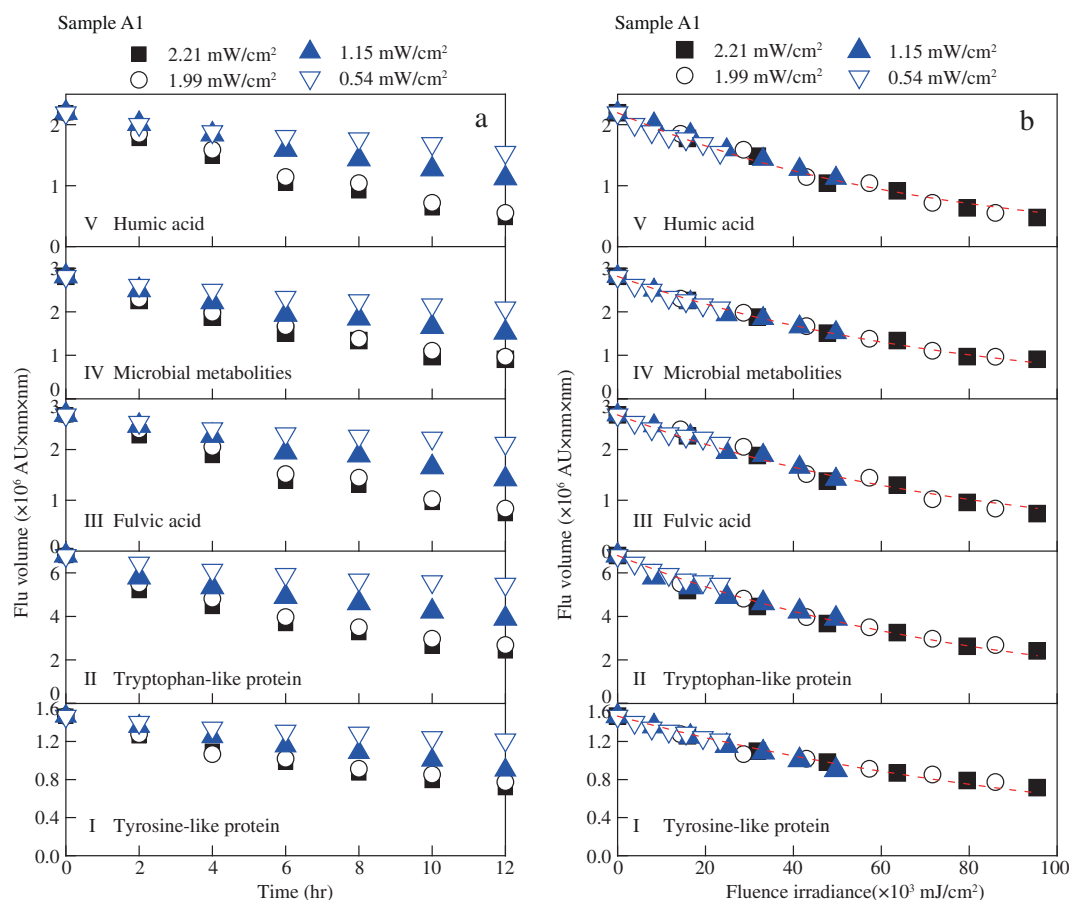
Fig. 7 – Simulated solar irradiation-induced degradation of fluorescence volume in sample A1 at different ultraviolet light irradiation intensities: (a) irradiation time-based irradiation experiments kinetics and (b) irradiation fluence-based irradiation experiments kinetics. The light intensities of ultraviolet light ranged from 0.54 mW/cm² to 2.21 mW/cm².

to 12 hr are shown in Fig. 4. The decrease in the FLU volumes in the FLU spectra was fit by pseudo-first order reaction kinetics. The kinetic constants ranged from 0.10 to 0.15/hr, which were much higher than those of the UV_{254} absorbance changes. These findings indicated that the photodegradation of fluorophoric DOM was much faster than that of chromophoric DOM. The irradiation process changed the molecular structure of the humic acid, after decarboxylation and decolorization reactions, with some damage to the carbon skeleton and chromophores. However, although the intensity of some aromatic or phenolic compounds was decreased, they changed into other aromatic compounds, for example chloro-benzene, 1,3-demethyl-benzene, etc. (Li et al., 1996).

But absorbance in the UV region appears to be influenced by the type and number of double bonds. The phenolic groups were decomposed into double bonds and other intramolecular charge transfer complexes with donor/acceptor moieties (Maleki et al., 2006), which show substantial UV absorbance at 254 nm.

2.3. Effects of the wavelength and intensity of solar light on UV_{254} absorbance and FLU removal

2.3.1. Effects of solar light wavelength
The changes in the UV_{254} absorbance and FLU volumes of DOM under light irradiation with the respective wavelengths

Table 1 – Time-based irradiation experiment kinetic constants of fluorescence volumes in sample A1 under solar light irradiation with different intensities of ultraviolet light.

Ultraviolet light intensity (mW/cm²)	Time-based irradiation experiment kinetic constants (/hr)				
	Region I (tyrosine-like protein)	Region II (tryptophan-like proteins)	Region III (fulvic acid)	Region IV (microbial metabolites)	Region V (humic acid)
0.54	0.02	0.02	0.02	0.02	0.03
1.15	0.04	0.05	0.05	0.05	0.05
1.99	0.06	0.08	0.09	0.09	0.11
2.21	0.06	0.09	0.10	0.10	0.12

of 275 to 420 nm and >420 nm for 0 to 12 h are shown in Fig. 5. The intensity of light at 275 to 420 nm was 0.60 mW/cm², which is equal to 25% of the UV light intensity of simulated solar light. The intensity of light with a wavelength of >420 nm (53.00 mW/cm²) was similar to that of visible light under simulated solar light.

The removal efficiency of the UV_{254} absorbance of the reclaimed water samples was less than 3% after irradiation by visible light ($\lambda > 420$ nm). However, the UV_{254} absorbance of the reclaimed water samples after irradiation by UV light decreased to 80% of the original value. The difference in the changes in the UV_{254} absorbance under irradiation by UV and visible light indicated that UV light irradiation in solar light serves a key function in decreasing the UV_{254} absorbance of reclaimed water.

The FLU volumes significantly decreased, with the kinetic constants of 0.02–0.04/hr for water samples A1 and B1, respectively, after irradiation by UV light with a wavelength of 275 nm to 420 nm. The UV light intensity after the light filter was much lower than that of simulated solar light without a filter, therefore the kinetic constants for FLU volume under UV light were lower than those under the simulated solar light. However, the FLU spectra of the reclaimed water after irradiation by visible light ($\lambda > 420$ nm) were similar to those before irradiation by light, indicating that UV light (i.e., not visible light) degraded the fluorescent DOM of the reclaimed water.

For river water, UV light degrades DOM into carbonyl compounds, and the removal efficiency of humic acid and protein in rivers under visible light is very limited (Davies, 1987; Engebretson, and Von, Wandruszka, 1997; Andley et al., 1984). The UV absorbance spectra of the two water samples from 200 to 900 nm are shown in Appendix A Fig. S2 of the supplementary material. The water samples did not absorb light with a wavelength of more than 420 nm.

2.3.2. Effects of irradiation intensity

The removal kinetics of the UV_{254} absorbance and FLU volumes of the reclaimed water (sample B1) were evaluated under various intensities of simulated solar light irradiation, as shown in Figs. 6 and 7. The light intensities shown in the figures refer to UV light intensities; visible light did not decrease the UV_{254} absorbance and FLU volumes of the reclaimed water. The wavelength of UV light was divided into two parts: UV-A (320 to 360 nm) and UV-B (290 to 320 nm). The contributions of UV-B and UV-A to the total UV light intensity were 5% to 10% and 90% to 95%, respectively.

According to the fitting results of time-based kinetics, the time-based kinetic constants of UV_{254} absorbance increased from 0.01 to 0.04/hr, accompanied by an increase of UV light intensity in the range of 0.54 to 2.21 mW/cm². Details of the time-based degradation kinetic parameters of fluorophore are shown in Table 1. The removal kinetic constants of the FLU volumes also increased with an increase in light intensity.

Quantum yields are amounts of reagents consumed per photon absorbed in photochemical reactions. To verify the influence of light intensity on the quantum yields of chromophore and fluorophore degradation, the relationship between the light fluence-based removal kinetics of UV_{254} absorbance/ FLU volume and light intensity is shown in Figs. 6b and 7b. These

figures compare the removal kinetics of the UV_{254} and FLU volume of the samples under different light intensities. The light fluence-based removal kinetic constants of the UV_{254} and FLU volume were independent of light intensity, indicating that light intensity cannot affect the quantum yield of chromophoric and fluorophoric DOM during solar light for irradiation experiments.

2.4. Effects of solar light on the molecular weight of DOM

The molecular weight of DOM before and after irradiation by simulated solar light is shown in Fig. 8. The molecular weight of DOM in the reclaimed water samples before irradiation by solar light ranged from 100 Da to 2000 Da. Three peaks in SEC
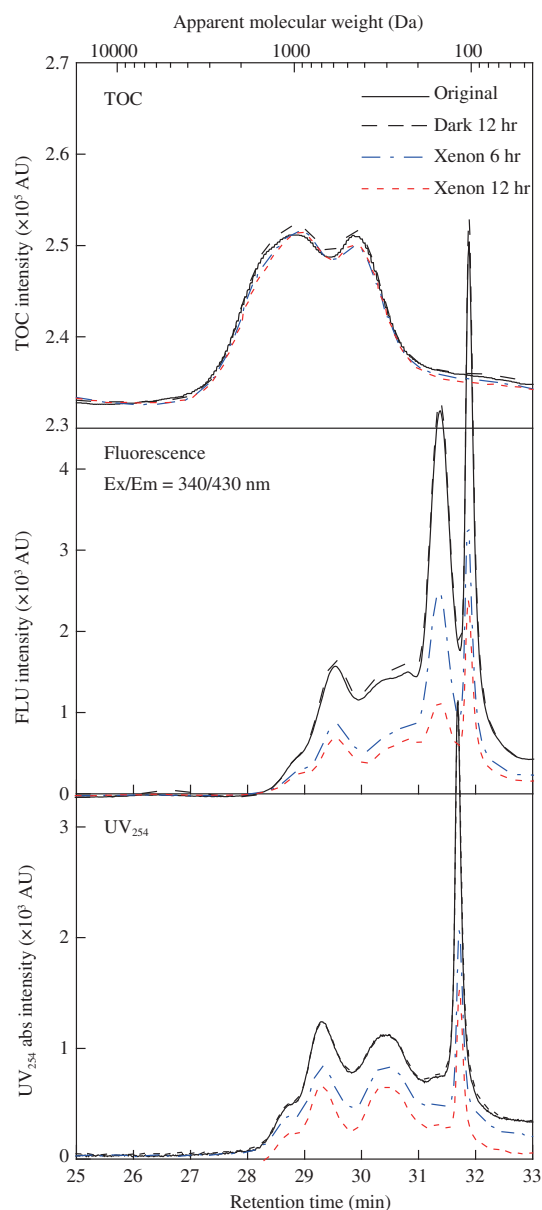

Fig. 8 – Effects of simulated solar light on molecular weight in reclaimed water sample B1. The light intensities of ultraviolet light were 2.21 mW/cm².

with UV_{254} absorbance occurred, but the sharpest peak was a solvent peak. Therefore, the molecular weight of major peaks with UV_{254} absorbance in the SEC of the reclaimed water samples were 290 Da and 780 Da. However, the molecular weights of peaks identified through the DOC detector were 420 and 1130 Da, indicating that some DOM did not exhibit UV_{254} absorbance. The molecular weight of the fluorescent peaks at Ex/Em of 340/430 nm, 235/300 nm, 235/355 nm, 245/420 nm, and 280/355 nm are shown in Figs. 7 and Appendix A Fig. S4. The molecular weights of the fluorescent DOM peaks with Ex/Em = 340/430 nm were 170 Da and 610 Da, which differed from those of the fractions contributing to DOC and UV_{254} absorbance.

The DOC value of the two major peaks in the SEC insignificantly decreased after irradiation by solar light, while the UV_{254} absorbance and FLU intensity of all the major peaks in the SEC of the reclaimed water samples significantly decreased. This finding may be explained by the hypothesis that the simulated solar light possibly transformed the chromophoric and fluorophoric DOM in products with similar molecular weights and lower UV254 absorbance and fluorescence.

The removal efficiency of DOM with different molecular weight in reclaimed water samples B1 during simulated solar light irradiation is shown in Fig. 9 and Appendix A Fig. S5. Among the three fractions of DOM, the removal efficiency of DOM with UV_{254} absorbance and fluorescence with molecular weight of 400–900 Da was the highest. Further research is needed to explain this phenomenon.

3. Conclusions

The effects of solar light on UV_{254} absorbance intensity, DOC values, SUVA value, and FLU spectra of reclaimed water were investigated, and the following conclusions were obtained:

(1) Natural solar light significantly decreases the UV_{254} intensity and FLU spectra of reclaimed water, but has very limited effect on the DOC value of reclaimed water. (2) The decrease in the UV_{254} absorbance intensity and FLU volume of reclaimed water during irradiation by solar light can be fit by pseudo-first-order reaction kinetics. The removal rate of the FLU intensity is much higher than that of the UV_{254} absorbance. (3) UV light in solar light serves a key function in decreasing the UV_{254} absorbance intensity and FLU volume during solar light irradiation. The light fluence-based irradiation experiment kinetic constants of UV_{254} absorbance and FLU volume are independent of light intensity. (4) The majority molecular weight of DOM ranges from 100 to 2000 Da. The UV_{254} absorbance and FLU intensity of all the peaks decrease after solar irradiation, whereas the DOC values of the major peaks do not significantly change. The removal efficiency of the molecular weight of DOM from 400 to 900 Da was the highest.

Appendix A. Supplementary data

Supplementary data to this article can be found online at http://dx.doi.org/10.1016/j.jes.2015.08.021.
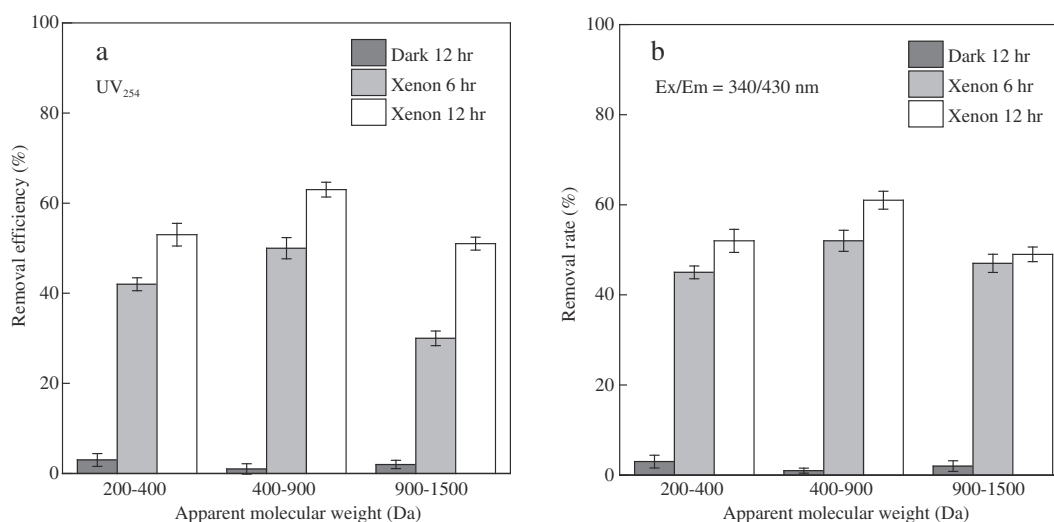

Fig. 9 – The removal efficiency of DOM with different molecular weight in reclaimed water samples B1 during simulated solar light irradiation. The light intensities of ultraviolet light were 2.21 mW/cm².

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