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The Influence of Organic Matter Origin on the Chlorine Bulk Decay Coefficient in Reclaimed Water

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Abstract: Using reclaimed water has been increasing to manage water shortages arising due to climate change. Research has been conducted on reclaimed water production, but few studies have investigated the pipe network and supply of reclaimed water. Reclaimed water contains greater amounts of organic matter, nutrients, and ionic substances compared to tap water. Therefore, it is highly likely to cause problems, such as water pollution due to microbial propagation in pipes, and leakage due to pipe corrosion, which interfere with water supply system operations. This study investigated the residual chlorine decay characteristics of chlorine disinfectants applied to a control biofilm in reclaimed water pipe networks. The bulk decay coefficient was compared between reclaimed water and a humic acid solution, and the origin of organic matter was analyzed using fluorescence excitation-emission matrices. The experimental results show that residual chlorine was consumed because protein and amino acid-based organic matter reacted more rapidly with chlorine than natural organic matter, such as humic acid. Moreover, chlorine bulk decay occurred rapidly in reclaimed water when total organic carbon was 3 mg/L or higher. These results confirm that removing organic matter during reclaimed water treatment may affect the management of the pipe network system.

Keywords: reclaimed water; residual chlorine; bulk decay coefficient; organic matter

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1. Introduction

Climate change has intensified the differences in regional water resource availability, resulting in regional water shortages [1–3]. The impacts of climate change are difficult to predict, but regular floods and droughts and the associated shortages of available water resources may cause problems in the water supply system. Therefore, the development of alternative water sources, such as rainwater, reclaimed water, groundwater, and seawater desalination, has been researched as methods to prevent water shortages [4–7].

The quantity of reclaimed water is constant throughout the year in urban areas, and reclaimed water can be a viable alternative water source. Most studies related to reclaimed water have focused on reclaimed water treatment, such as membrane filtration, ozone treatment, and activated carbon treatment, for reclaimed water production [8–10]. By contrast, most studies conducted on the water supply pipe network have focused on tap water subjected to water treatment [11–13]; however, few studies have been conducted on reclaimed water with different water quality characteristics [14]. Reclaimed water is used for non-drinking domestic applications, such as cleaning, toilet flushing, and gardening, and thus, it is highly likely to contact the human body. Therefore, it is necessary to investigate the characteristics of microorganisms in the reclaimed water pipe network and conduct research on control measures.

Over time, biofilms form on the inner surface of the water supply pipe network. As biofilm formation increases the distribution of microorganisms and causes an increase in harmful microorganisms, such as *Legionella* sp., it is necessary to continuously monitor

the condition of the pipe network and control microorganisms inside the pipe by regular cleaning [15,16]. Moreover, as reclaimed water is rich in nutrients compared to tap water that is subjected to water treatment, it can support increased biofilm formation in the pipe network. In a previous study, reclaimed water was circulated through a polyvinyl chloride pipe reactor and the biofilm was sampled to evaluate the impacts of reclaimed water on microbial communities [17]. In contrast to tap water, it was found that microbial species diversity decreased over time, and ammonium oxidizing bacteria, nitrite-oxidizing bacteria, and denitrifying bacteria were found in large quantities, indicating that the nitrogen component present in reclaimed water affected the microbial distribution in the biofilm [17].

To use reclaimed water as a public water source, disinfection is essential for controlling the growth of microorganisms that may cause health problems. Moreover, disinfectants, such as residual chlorine, should be retained in the pipe network. During disinfection in the pipe network, microorganisms are likely to propagate if the input of chlorine is insufficient. Conversely, if the chlorine input is excessive, carcinogenic disinfection by-products, such as trihalomethane, are generated [18,19]. Proper maintenance of residual chlorine is also important for the water supply network. Residual chlorine is consumed due to reduction reactions with bulk water and the pipe wall. In particular, during the reduction of chlorine by bulk water, fast and reduction reactions have been examined [20–22]. Previous studies on the residual chlorine reduction in reclaimed water indicated that the reaction between reclaimed water and chlorine exhibited different characteristics compared to tap water because reclaimed water contains more organic matter and ionic substances than tap water. According to Kang et al., reclaimed water had more than 20 times higher electrical conductivity, and more than double the organic matter content, than tap water [23]. Moreover, Hu et al. reported that assimilable organic carbon (AOC), dissolved organic nitrogen, and dissolved organic carbon (DOC) of reclaimed water were higher than those of tap water [24]. As the organic matter present in tap and reclaimed waters have different origins, they may affect the reaction characteristics of residual chlorine differently in the pipe network.

This study investigated the decay characteristics of residual chlorine, which is required to control biofilms in the reclaimed water pipe network in bulk water. Additionally, the influence of the organic matter origin of reclaimed water on chlorine bulk decay was evaluated. Our findings have implications for pipe network management when using reclaimed water as a source for public water supply, which will be important in the future to address water shortages due to climate change.

2. Materials and Methods

2.1. Relclaimed Water Samples

Experiments of chlorine bulk decay were performed using three water samples: two samples from the reclaimed water of the Songdo sewage treatment plant in Incheon-Si, South Korea (Incheon-si reclaimed water, IRW-A and IRW-B) and one sample from the reclaimed water of the sewage treatment plant in Gumi-Si, Gyeongsangbuk-do, South Korea (Gumi-Si reclaimed water, GRW). The total organic carbon (TOC) concentration was 3.71, 1.27, and 3.44 mg/L for IRW-A, IRW-B, and GRW, respectively. The IRW facility is located in coastal landfills. The water is used as reclaimed water after going through chlorine disinfection involving a bioreactor, secondary settling basin, sand filtration, and activated carbon processes. The reclaimed water from the GRW facility is discharged after passing through a bioreactor and secondary settling basin for coagulation, sand filtration, and UV disinfection. Both IRW and GRW samples were tested in the reclaimed water of the sewage treatment plant before the disinfection process.

2.2. Natural Organic Matter (NOM) Standard Solution

A NOM standard solution was prepared using a humic acid standard reagent to evaluate the influence of organic matter origin on chlorine bulk decay. To prepare the NOM standard solution, H16752 reagent (Sigma-Aldrich, Saint Louise, MO, USA) was placed in

distilled water to achieve a concentration of 1000 mg/L, and the pH was adjusted to 10 using 0.1 N NaOH solution. The solution was used after being stirred for 24 h, filtered with GF/C filter, and then diluted to 1–5 mg/L required for the experiment.

2.3. Bulk Decay Coefficient (k_b) Measurement Method

To measure the chlorine bulk decay coefficient, the experiment was performed in a constant-temperature chamber by setting the water temperature to 5, 15, and 25 °C and the chlorine injection concentration condition to 1, 2, and 4 mg/L. A 150 mL serum bottle and a Teflon-coated silicone stopper were used in the experiment and were washed with distilled water and dried at 105 °C for more than one hour before use.

Sodium hypochlorite solution (8%) was diluted to 4:1 (v/v) by deionized water and was injected according to the chlorine injection concentration condition, and free residual chlorine was measured over time. For each sample, measurements were taken after 0, 3, 5, 10, 20, 30, 60, and 120 min during the first 2 h, every 2 h for the next 6 h, and then every 4 h for the remainder of the experiment (up to 84 h).

For residual chlorine analysis, color development was conducted using the DPD (N,N-diethyl-p-phenylenediamin) colorimetric method of Standard Methods for the Examination of Water and Wastewater, and the free residual chlorine concentration was measured using Pocket Colorimeter^{TM} II (HACH, Loveland, CO, USA). In each test, three measurements were taken and averaged.

2.4. Analysis of Water Quality Parameters and Fluorescence Characteristics

Table 1 shows the analysis parameters and analysis methods for determining the quality of reclaimed water. For each sample, a Shimadzu RF5301 fluorescence spectrophotometer was used to obtain a fluorescence excitation-emission matrix (FEEM) for the fingerprint of organic matter. The excitation wavelength region of FEEM ranged from 220 to 400 nm and its emission wavelength region ranged from 250 to 600 nm. Measurements were performed at 10 nm intervals.

Table 1. Analytical equipment and methods of the water quality parameters.

Parameter	Unit	Equipment/Method
Temperature	°C	Istek 720P, Istek, Seoul, Korea
pH		pH meter, Istek pH-250L
Cl^-	mg/L	DR 4000, HACH, USA
$SO_4{}^{2-}$	mg/L	DR 4000, HACH, USA
$NH_4{}^+$-N, $NO_3{}^-$-N	mg/L	DR 4000, HACH, USA
TOC, DOC	mg/L	TOC-VCPH, Shimadzu, Kyoto, Japan
Hardness	mg/L as $CaCO_3{}^-$	DR 4000, HACH, USA
Alkalinity	mg/L as $CaCO_3{}^-$	Standard method (2320)
Cu^{2+}, Fe^{2+}, Mn^{2+}, Zn^{2+}	mg/L	IRIS intrepid II XDL, Thermo Electron Corp, Waltham, MA, USA

The samples before analysis were filtered through a 0.22-μm membrane filter. A xenon lamp was used as the light source, and the excitation-emission slit width was set to 10 nm. Every sample was measured after the DOC (dissolved organic carbon) concentration was diluted to 2 mg C/L. The EEM measurement data of the samples were corrected for the fluorescence spectrum, which appeared in the liquid sample by default, using the EEM measurement value of distilled water. In addition, fluorescence EEM analysis was performed using Sigmaplot software.

3. Results and Discussion

3.1. Residual Chlorine Decay Characteristics of Reclaimed Water

Three samples produced from the two sewage treatment plants were used to investigate the chlorine bulk decay characteristics of reclaimed water. Table 2 shows the water

quality analysis results for reclaimed water. The IRW samples showed higher chloride ion concentrations than the GRW sample because the sewage treatment plant was located in a coastal landfill, and thus, seawater penetrated into the sewer pipe. A comparison between TOC and dissolved organic carbon (DOC) concentrations shows that most of the organic components in the samples were in the dissolved state. In addition, the water quality factors that affected the consumption of residual chlorine in the general water supply network were reductive inorganic substances, such as ammonia nitrogen (NH_4^+-N), Fe^{2+}, and Mn^{2+}. However, for reclaimed water, these water quality components were not expected to have a significant influence because most were removed during the sewage treatment process. In addition, the low concentration of ammonia nitrogen shows that biological nitrification was well performed in the sewage treatment plants.

Table 2. Water quality in the reclaimed water samples.

Parameter	Tap Water	IRW A	IRW B	GRW
Temp (°C)	15.2	22.7	16.0	17.5
pH	7.25	7.50	6.89	7.32
Cl^- (mg/L)	10.1	1197.0	1011.0	357.0
SO_4^{2-} (mg/L)	12.0	171.5	130.0	143.0
NH_4^+-N (mg/L)	0.07	0.03	1.40	0.00
NO_3-N (mg/L)	1.10	7.35	7.85	12.80
TOC (mg/L)	1.75	3.710	1.270	3.444
DOC (mg/L)	1.62	3.675	1.174	3.127
UV_{254} (abs./cm)	0.021	0.055	0.032	0.060
Hardness (mg/L as $CaCO_3$)	82	440	420	280
Alkalinity (mg/L as $CaCO_3$)	30	85	77	89
Cu^{2+} (mg/L)	0.004	0.0239	0.0231	0.0037
Fe^{2+} (mg/L)	0.010	0.0293	0.0436	0.0065
Mn^{2+} (mg/L)	0.009	0.0418	0.0413	0.0009
Zn^{2+} (mg/L)	0.068	0.0247	0.0256	0.0046

Warton et al. (2006) applied an exponential first-order decay model proposed to calculate k_b [25]. The model formula is as follows:

$$C_t = a + b(e^{-kb \bullet t}) \quad (1)$$

The parameters a, b, and k_b were determined using curve-fitting software (OriginLab Origin Version 8.0). The chlorine bulk decay characteristics of each reclaimed water sample were investigated by temperature (5, 15, and 25 °C) and hypochlorite injection concentration (1, 2, and 4 mg/L) to calculate the chlorine bulk decay coefficient (k_b). The corresponding results are shown in Table 3.

Table 3. Bulk decay coefficients of residual chlorine in reclaimed water samples.

Temp. (°C)	Initial Chlorine ion Injection (mg/L)	k_b (day^{-1})			
		Tap Water	IRW-A	IRW-B	GRW
5	1	1.785	605.290	2.018	397.138
	2	1.454	459.130	1.669	60.350
	4	1.326	320.458	1.432	11.362
15	1	2.658	694.886	3.122	512.510
	2	2.211	542.074	2.675	97.157
	4	1.762	427.608	2.159	19.181
25	1	3.276	906.12	3.887	652.882
	2	2.878	754.661	3.488	118.411
	4	2.501	555.134	3.095	29.837

For all samples, k_b increased with temperature, which was expected to follow the trend in the Arrhenius equation (Equation (2)), where the chemical reaction rate increases with temperature.

$$k_b = Ae^{-(E/RT)} \quad (2)$$

where k_b is the residual chlorine decay coefficient (day^{-1}), A is the frequency factor or pre-exponential factor (mol/day), E is the activation energy (J), R is the universal gas constant (J/mol·K), and T is the absolute temperature (K).

In addition, k_b decreased in all samples as the chlorine injection concentration increased. Rossman (2006) reported that the chlorine bulk decay reaction follows a first-order reaction, as shown in Equation (3), and that the first-order reaction rate constant (k_b) of Equation (4) becomes a function of the initial chlorine concentration (C_0) and a stoichiometry constant (α); therefore, k_b increases as C_0 decreases under the condition that the concentration of reactants (X_0) does not change [26].

$$\frac{dC}{dt} = -k_b C \quad (3)$$

$$k_b = k_0(X_0 - \alpha C_0) \quad (4)$$

3.2. Influence of Organic Matter Characteristics on Residual Chlorine Decay

To examine the influence of organic matter characteristics on chlorine bulk decay, the experiment was performed using tap water and reclaimed water (IRW-A, IRW-B, and GRW) at 5 and 25 °C, respectively. The results are shown in Figure 1. The reclaimed water exhibited much faster chlorine bulk decay than tap water.

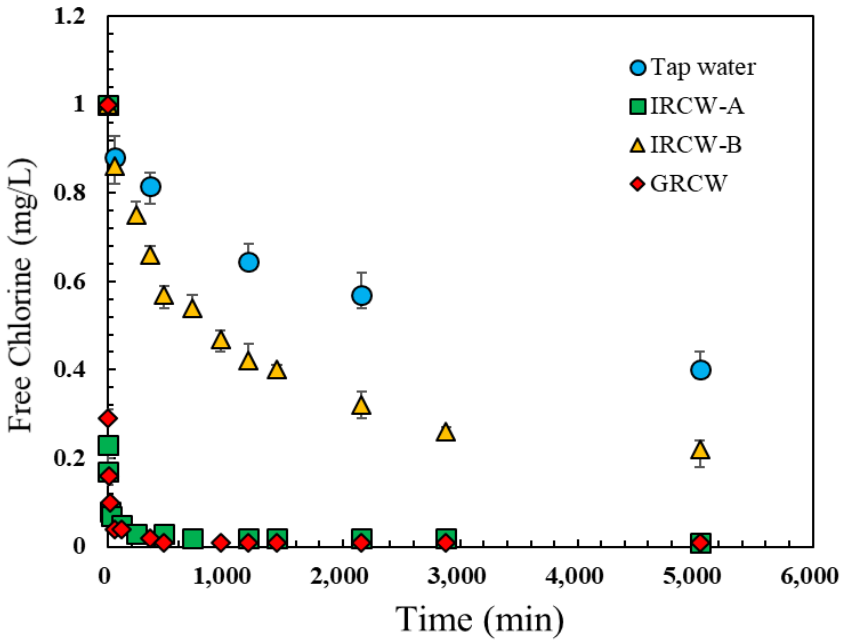

Figure 1. Comparison of residual free chlorine in reclaimed water and tap water.

TOC is divided into particulate organic carbon and DOC. DOC is divided into non-easily biodegradable refractory DOC (RDOC) and easily biodegradable labile DOC (LDOC) [27]. The organic components of tap water mainly consist of RDOCs, such as polyaromatic compounds, and show a higher absorbance during UV254 measurements than organic materials that mainly consist of LDOCs [28]. Figure 2 shows the k_b values according to $SUVA_{254}$ (Specific UV_{254}; calculated by dividing UV_{254} by DOC and multiplying by 100) and TOC concentrations. As k_b increased, TOC increased, whereas $SUVA_{254}$ decreased. The lower the k_b, the higher the $SUVA_{254}$, indicating that the organic matter

in the samples had a high proportion of RDOC. For reclaimed water, non-biodegradable RDOC is the main component if the TOC concentration is low because most biodegradable organic components are removed, whereas easily biodegradable organic components exist if the TOC concentration is high [29].

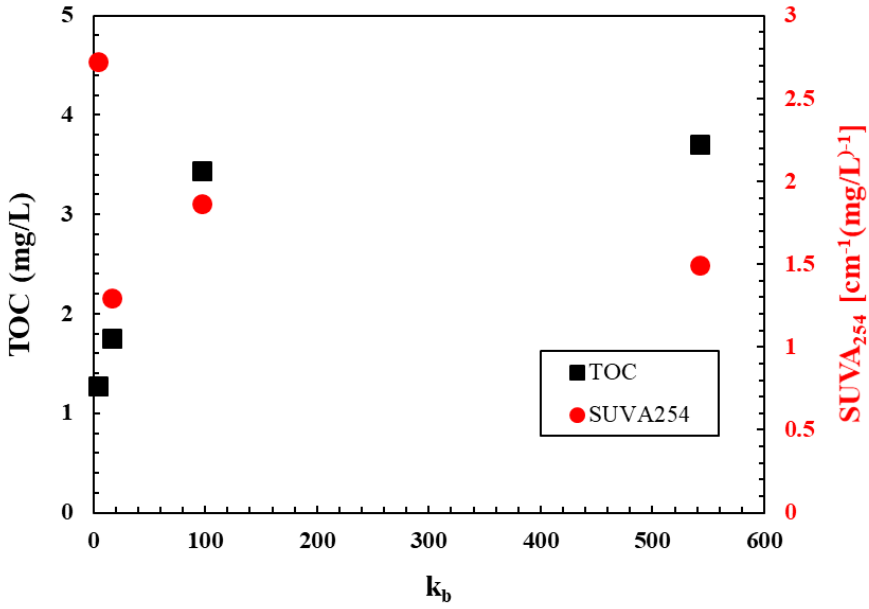

Figure 2. Relationship between k_b and total organic carbon concentration and $SUVA_{254}$ in reclaimed water.

3.3. Influence of Natural Organic Mater on Chlorine Bulk Decay

To investigate chlorine bulk decay characteristics according to the organic matter origin, a NOM solution that reflected the characteristics of organic matter in the tap water sample was prepared, and chlorine bulk decay characteristics according to the TOC concentration in NOM were examined. As shown in Figure 3, residual chlorine rapidly decreased as TOC increased up to 2000 min. Moreover, the chlorine bulk decay coefficient increased as TOC increased, as shown in Table 4.

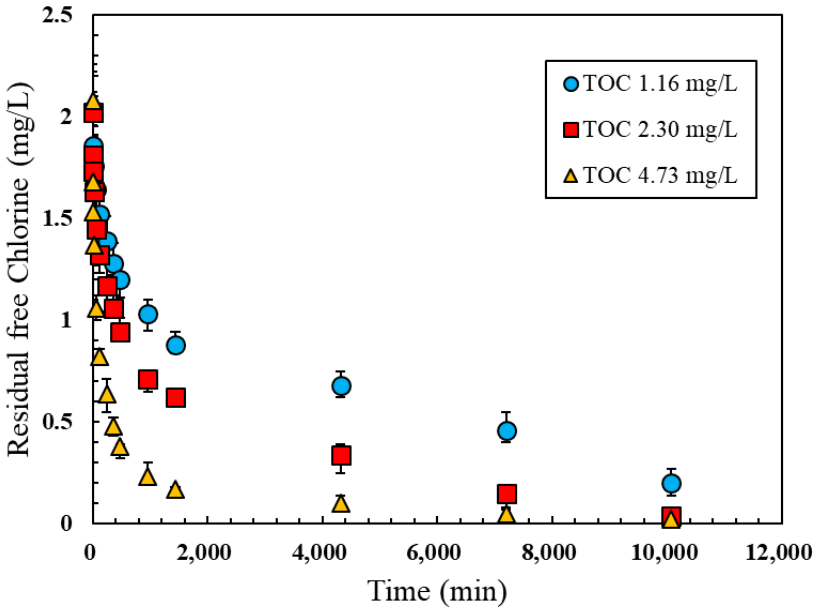

Figure 3. Reduction of residual free chlorine with differing amounts of natural organic matter.

Table 4. Bulk decay coefficients in NOM solution.

TOC (mg/L)	1.16	2.30	4.73
k_b (day^{-1})	0.512	2.119	9.173

3.4. Comparison of Bulk Decay Coefficients between Tap Water, Reclaimed Water, and NOM

Based on the above results, the chlorine bulk decay coefficients of the reclaimed water samples and NOM samples obtained under the same experimental conditions (an initial chlorine concentration of 2 mg/L and a water temperature of 15 °C) are shown in Figure 4. The chlorine decay rate increased as the TOC concentration increased for each of the reclaimed water and NOM samples. Figure 4 shows separate plots for reclaimed water, NOM, and tap water. Correlations to TOC and k_b can be seen between reclaimed water samples, and between NOM and tap water samples. The correlation between reclaimed water samples differed from that between the NOM and tap water samples, possibly attributed to the properties of the organic matter that reacted with chlorine. Kohpaei et al. [30] suggested a parallel second-order kinetic model, which distinguished the chlorine bulk decay coefficient between organic matter having a fast reaction rate with chlorine and organic matter with a slow reaction rate. The R^2 value for TOC and k_b between reclaimed water samples was 0.93, whereas it was 0.96 for TOC and k_b between the NOM and tap water samples (Figure 4). Thus, the reaction characteristics with chlorine were different among the reclaimed water, NOM, and tap water samples. This suggests that among the organic components of reclaimed water, organic matter that reacted quickly with chlorine, and those that reacted slowly, coexisted. In this study, a large k_b appeared for the sample TOC (>3 mg/L), indicating the existence of organic matter that reacted quickly with chlorine.

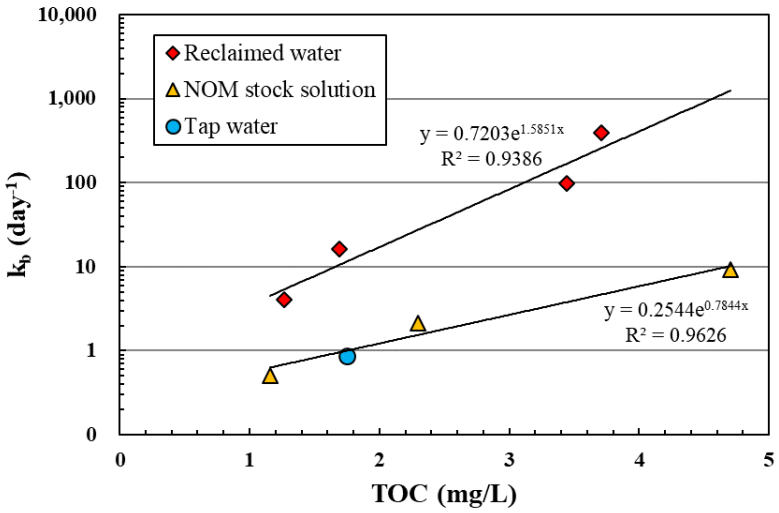

Figure 4. Comparison of k_b between reclaimed water and NOM solution.

3.5. Influence of Organic Matter Origin in Reclaimed Water on Chlorine Bulk Decay

The origin of organic matter that exists in water can be identified through FEEM analysis [31]. It is possible to determine whether the organic matter in water is a humic acid-like or fulvic acid-like polyaromatic compound, or protein-like or amino acid-like organic matter. Therefore, FEEM was measured for GRW, which exhibited high k_b among the reclaimed water samples, and IRW-B and NOM samples, which showed low k_b values. The results are shown in Figure 5.

In general, humic acid-like organic matter shows strong emission intensity in the 420–450 nm range during excitation in the 230–260 nm and 320–350 nm ranges, and protein-like or amino acid-like organic matter exhibits strong emission intensity in the 300–305 nm, and 340–350 nm ranges during excitation in the 220–275 nm range [32].

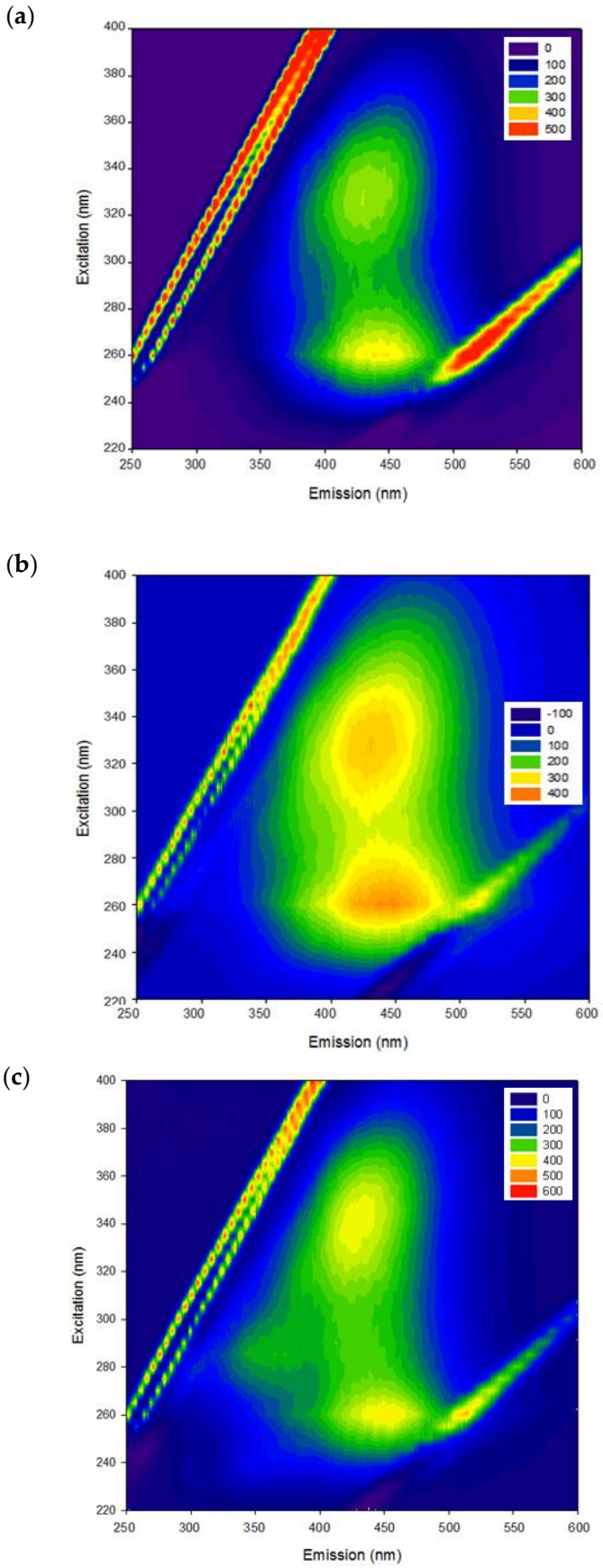

Figure 5. FEEM images. (**a**) NOM solution, (**b**) Incheon reclaimed water A (IRW-A) sample, (**c**) Gumi reclaimed water (GRW) sample.

Figure 5a shows that the NOM sample showed strong emission intensity in the 420–450 nm range during excitation in the 230–260 nm and 320–350 nm ranges. Figure 5b shows the FEEM results of IRW-B, which had a TOC concentration (1.690 mg/L). IRW-B showed strong emission intensity in the 420–450 nm range during excitation in the 230–260 nm and 320–350 nm ranges. The peak intensity shown in Figure 5b represented strong emission intensity in the 410–470 nm range during excitation in the 250–270 nm and 310–350 nm ranges, and was similar to that of Figure 5a, which was prepared using humic acid.

Figure 5c shows the FEEM results of GRW, which exhibited a high chlorine bulk decay coefficient. Partial fluorescence of protein-like or amino acid-like organic matter can be observed in the figure, and the results are different from those shown in Figure 5a,b. It was found that residual chlorine in water has a low reaction rate with humic acid-like or fulvic acid-like organic matter, such as polyaromatic compounds, and a high reaction rate with protein-like or amino acid-like organic matter.

Jemba et al. [33] reported that chlorine in reclaimed water was rapidly consumed even in the absence of ammonia nitrogen because the organic matter in reclaimed water has lower molecular weight and contains more components that contribute to microbial assimilation than the organic matter in purified water. This is consistent with the results of this study where the chlorine bulk decay reaction occurred rapidly as protein-like and amino acid-like organic matter that originated from sewage rapidly reacted with chlorine. The reaction between organic matter and residual chlorine in reclaimed water can also be explained with the chlorine decay model presented by Funamizu et al. [34], who found that organic matter with a low molecular weight of 3000 Da or less reacts with chlorine much faster than the organic matter with a molecular weight of 3000–10,000 Da.

The high molecular weight organic matter can be reduced to lower molecular weight through reaction with chlorine, which is then quickly consumed through a fast reaction with low molecular weight organic matter. Moreover, when advanced treatments, such as ozone oxidation, are applied to reclaimed water, TOC or DOC can be reduced. However, AOC, which is organic matter that can be assimilated by microorganisms, may increase, thereby decreasing biological stability in the pipe network. Therefore, to prevent the depletion of residual chlorine in the reclaimed water pipe network it is necessary to continuously monitor the organic matter and residual chlorine in reclaimed water and consider introducing additional biological treatment processes, such as biofiltration.

4. Conclusions

The use of reclaimed water is expected to increase in the future as a method to mitigate water shortages caused by climate change. Research on residual chlorine decay that occurs in the reclaimed water supply network is important for the safe use of reclaimed water. In this study, the bulk decay coefficient of reclaimed water was compared to that of a solution prepared with humic acid to investigate the influence of organic matter on chlorine bulk decay in reclaimed water. Chlorine in water was reduced as it reacted more rapidly with the sewage-originated organic matter, such as proteins and amino acids, than NOM, such as humic acid, as in tap water. In addition, in the case of reclaimed water, it was found that chlorine bulk decay occurred rapidly when TOC was 3 mg/L or higher. Based on the above results, it is deemed necessary to continuously monitor organic matter in reclaimed water and adjust the input amount of chlorine because chlorine bulk decay in reclaimed water is significantly affected by organic matter characteristics. These results confirmed that removing organic matter during reclaimed water treatment has significant implications in the management of the pipe network system.

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