

# Jase 23\_Fluorescence Organic Carbon Components and its Correlation with Bulk Organic Parameters in Water Treatment Processes

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## Fluorescence Organic Carbon Components and its Correlation with Bulk Organic Parameters in Water Treatment Processes

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Water containing natural organic matter (NOM) has an adverse effect on the aesthetic water qualities, and may cause some problems in water treatment processes. The aim of this research is to assess the changing of fluorescence dissolved organic carbon by using Fluorescence Excitation Emission Matrices (FEEMs) analysis through Parallel Factor (PARAFAC) modelling. In addition, this study purposed to examine the correlation between organic fluorescence with bulk parameters concentration, such as dissolved organic carbon (DOC), ultraviolet absorbance at 254 nm (UV<sub>254</sub>), and specific UV absorbance (SUVA) during water treatment processes. The results show humic-like compound, such as tryptophan-like and fulvic acids-like are the two major organic components contained in the water. Organic matter fluorescence identified that fulvic acid-like shows a higher removal than aromatic protein-like. The correlations of organic fluorescence are higher with DOC than with UV<sub>254</sub> and SUVA value. Combination between bulk parameters and FEEMs methods could be used to give reinforced information about the presence of the main organic fractions in source and treated waters.

**Keywords:** Dissolved organic matter; Water treatment; Fluorescence; Parafac

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### 1. Introduction

Dissolved organic carbon (DOC) presence in the water has adverse effect on the water aesthetic, such as color, taste, and odor [1]. Furthermore, DOC has drawbacks in drinking water treatment which has an effect on membrane fouling due to DOC can be a major constituent causing significant loss of membrane permeability [2]. DOC may act as a precursor of harmful disinfectant by-products (DBPs) formation during chlorination process, such as trihalomethanes (THMs) and haloacetic acids (HAAs), which have carcinogenic properties [3]. Generally, the mostly feasible process to reduce DOC is conventional treatment, including coagulation, followed by flocculation, sedimentation/floatation, and sand filtration [1]. Some identification methods of organic matter have been developed to gain a better understanding of the kinds of DOC existed in water,

and their subsequent transformation during water treatment process. Recently, fluorescence excitation-emission matrices (FEEMs), a fluorescence spectroscopy method, has been employed to support an information of different kinds and sources of DOC in waters [1, 4]. Fluorescence spectroscopy identified organic matter based on both its molecular structure and composition, thus it could performed the chemical characteristics of DOC. Three-dimensional of FEEMs has been implemented to differentiate organic matter types of DOC in aquatic waters, because of simple, low cost, very sensitive tool, require small sample volume and no complicated sample preparation [5]. Parallel factor (PARAFAC) analysis is the most widely used application of multivariate statistical methods that has been coupled with fluorescence intensity data [6]. PARAFAC method makes the identification of different types of organic matter into a number of components. The independent components pre-

senting diversity in the composition and source component among water samples. Each component reflects a group of fluorophores with specific and similar characteristics in term of an assess of emission and excitation loadings of each component, and a relating separately to each of two or more fluorophore group in the form of a sample score [5, 7]. FEEMs with PARAFAC analysis have been used to track the changing and removing of DOC fractions in batch-scale, pilot-scale, and in water treatment plants [6, 8, 9]. Several studies have exhibited the application of EEMs with PARAFAC method to characterize organic matter and to initiate the correlations between attained components and the formation of DBPs [7, 10]. DOC is mostly presented quantitatively by total organic carbon (TOC), or absorption of UV-light (UV<sub>254</sub>) for practical purposes, however less information of its characteristics. Therefore, this study use combined methods, FEEMs by PARAFAC analysis, TOC, UV<sub>254</sub> parameters on the same sample simultaneously, in order to track the changing of organic component and to assess the relationships among the organic components, TOC, and UV<sub>254</sub>.

## 2. Materials and Methods

Water' sampling collection had been conducted at Lujhu Water Treatment Plant (LJWTP). LJWTP has maximum capacity to treat water approximately 157,500 m<sup>3</sup>/day. The plant uses a conventional treatment process which consists of intake (raw water), pre-settling, slow mix (coagulation process), sedimentation, rapid sand filter, and clarifier (finally chlorination). In Polyaluminium chloride (PACl) is used as a coagulant, with the average total amount of PACl is 2,844 kg/day. In addition, the total time in the coagulation process is 27.5 minutes, with rapid mixing for 2.1 minute and slow mixing for 25.4 minutes, followed by settling processes for 89.7 minutes. Chlorine was injected in the pre-settling and in the clarifier, while the types of chlorine used in the pre-settling (or known as prechlorination) and in the clarifier (or known as chlorination process) is sodium hypochlorite (NaOCl), with average total amount of NaOCl 1,603 kg/day. Sample were taken at September and lasted until April in the following year with sampling frequency once per month, however sampling in October was done twice due to heavy rain in October.

All samples were filtered through pre-rinsed 0.45 μm cellulose acetate (CA) membrane prior to analyze with the aim to separate particulate contained in the water. The water samples were analyzed for DOC concentration using TOC analyzer (Model TOC-500, Shimadzu, Kyoto, Japan), ultraviolet absorbance at 254 nm (UV<sub>254</sub>) using a UV/Vis spectrophotometer (Model U-2001, Hitachi, Japan) with a 1

cm quartz cell. The specific ultraviolet absorbance (SUVA) was calculated by dividing the UV<sub>254</sub> over TOC concentration. SUVA indicates the amount of humic substances and non-humic substances in the natural organic matter [11].

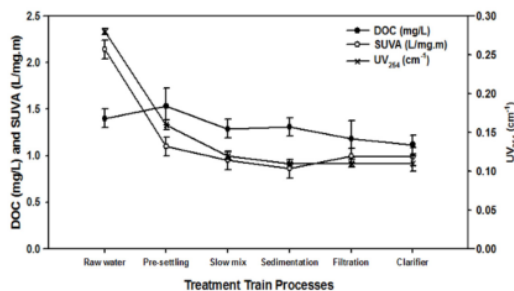
Perkin Elmer LS-55 luminescence spectrometer was used for fluorescence measurement. Excitation wavelengths (Ex) between 200 and 400 nm at interval of 10 nm and emission wavelengths (Em) between 280 and 550 nm at intervals of 0.5 nm was set up for all samples, as described in Hidayah [7]. Fluorescence measurements obtained 48 data set, which is represented samples from LJWTP, then it were used to construct parallel factor (PARAFAC) models with drEEM (<http://www.models.life.ku.dk/drEEM>) toolbox in Matlab® [6, 7]. The statistical analysis with Pearson correlations was determined using SPSS Statistics 17.0 software (IBM, Armonk, NY, USA). The Pearson correlation coefficients were used to measure the strength of the linear relationship among DOC, UV<sub>254</sub>, SUVA, and maximum fluorescence intensity (F<sub>max</sub>) of the PARAFAC component [7].

## 3. Results and Discussion

### 3.1. DOC, UV<sub>254</sub>, and SUVA Value

Figure 1 shows the DOC value of collected samples through water treatment process train. It describes the average and range of DOC, UV<sub>254</sub>, and SUVA value through the water treatment process train. It can be explained that DOC concentration of raw water varied from 1.05 mg/L, which occurred in October, to 1.8 mg/L in April, with an average of 1.30 mg/L. This is consistent with the change of seasons from wet to dry, which due to the decrease in river flow in dry season, as the river flow is lower, the dilution effect from dissolved matters is also lower. First, it shows pre-settling increased the DOC concentration to 1.53 m/L, it is probably due to the dosing of chlorine in the inlet to pre-settling basin. Chlorine could react with suspended particles, for example algae cells, and transform those into dissolved forms, and, therefore, the increase in DOC. The coagulation step shows the highest DOC reduction compared to other steps. This indicates the coagulation PACl used in this plant is effective in DOC removal.

Second, UV<sub>254</sub> shows a general trend of reduction. Regarding to DOC in pre-settling, reduction of UV<sub>254</sub> indicated that aromatic compound have been oxidized by chlorine in the inlet to pre-settling basin. This is consistent with decreasing SUVA value to 1.1 L/mg.m, which indicated the existence of non-aromatic and hydrophilic remain in the pre-settling basin. For SUVA value, the raw water has an average value of 2.14 mg/L, which is representative of intermediate hydrophobicity (2-4 L/m.mg) [11]. The general



**Fig. 1.** The concentration of DOC, UV<sub>254</sub>, and SUVA value through the water treatment process train.

trend is a reduction in SUVA, which shown the significant reduction in SUVA value through pre-settling basin. It is probably due to the dosing of chlorine in the inlet as oxidation would transform hydrophobic organics into hydrophilic ones. The further decrease in SUVA value is the following treatment step is also reasonable as hydrophobic organics are more easily removed by conventional water treatment process than hydrophilic organics [3, 12]. The increase SUVA value through filtration due to biological activity generated from filter media [13].

### 3.2. Component of Organic Fluorescence

An initial exploratory analysis was performed for 48 FEEMs water sample from LJWTP. Performance of 48 data set in PARAFAC analysis have validated 2 components which compose the water samples. Table 1 shows the main peaks of the two components with its excitation and emission wavelength pairs in this study, which referred to previous studies.

The result of identifying components with the spectral contour shown in Figure 2. Component C1 is fluorophores of humic-like which may derived from terrestrial or anthropogenic (originating in human activity) origin [11].

However, component C1 was initially assessed to have originate only from the marine environment, but lately has been tracked to be existed in freshwater due to agricultural contributions [16]. Component C2 is protein-like fluorophores, tend to tryptophan-like fluorophore. After validation of the two models of components, the component through the water treatment process was determined using fluorescence intensity maximum ( $F_{max}$ ).  $F_{max}$  gives an estimation of the relative concentration of each component. Figure 3 shows the average of  $F_{max}$  of each component through the water treatment process. It can be seen that  $F_{max}$  was higher for humic-like component C1 in raw

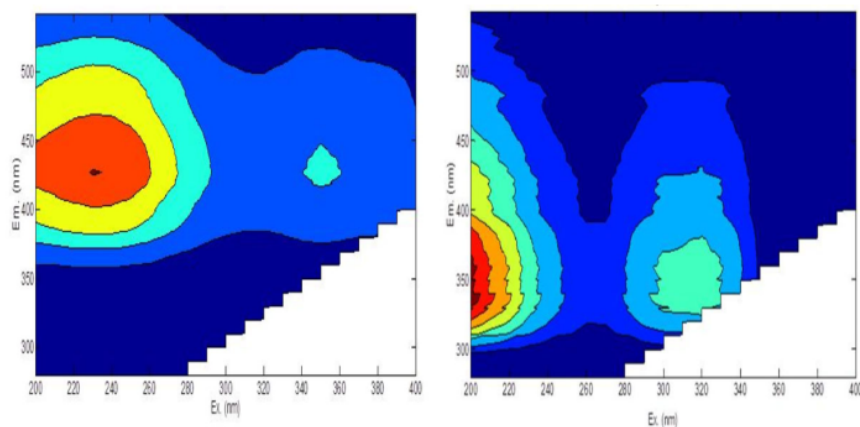
water and pre-settling, with  $F_{max}$  was 0.296 R.U., and 0.411 R.U., respectively. Increasing  $F_{max}$  of C1 and C2 fractions after pre-settling is probably due to the adding of chlorine dosage in the inlet of pre-settling tank. Chlorine could react with fluorophores in term of suspended particles, for example algae cells, and transform those into dissolved forms, and therefore increasing DOC in term of humic-like and protein-like, as shown in this study [17]. This results is in accordance with DOC concentration in pre-settling, as shown in Figure 1. Coagulation could reduce the fluorophores of humic-like down to 0.237 R.U., with reduction about 50%. This shows that the high molecular weight component C1 could be effectively removed by coagulation. The final treated water in clarifier has shown an increasing of  $F_{max}$  of C1 fraction, it is probably due to the effect of chlorination, which was injected in the clarifier. Chlorination and pre-chlorination in the pre-settling could break the bond between particle and adsorbed organics, and reduce the molecular weight of these organic matter, which would results in the desorption of stabilized organic coatings and the reduction of steric effect and electrostatic barrier [17, 18]. In addition, low molecular weight compound from sand filtration effluent may also produced metabolic products of microbes existing the sand filter. Those released of organic matter could be oxidized into lower molecular weight and more soluble, hence, those components tend to increase in the effluent of clarifier. Figure 3 also shows that component C1 has higher removal than component C2 through the coagulation, sedimentation, and filtration process. This can be explained by the generally lower removal efficiency of hydrophilic fraction organics, such as the protein-like component C2 than that of more hydrophobic humic substances fraction [8, 9, 19]. In addition, NOM consists of a number of components with wide various of chemical charges, chemical composition, molecular size, molecular weight, and structure. Therefore, the degree of organic matter or humic substances treatment in water treatment processes depends on the characteristic of NOM. Therefore, it is necessary to be able to recognize and divine the reactivity of NOM or its compounds during different phases of the water treatment.

### 3.3. Correlation Between Bulk Organic Parameters and Fluorescence Organic

Correlation analysis was conducted using  $F_{max}$  as a surrogate of the concentration of the PARAFAC components, with DOC, UV<sub>254</sub> concentration, and SUVA value. The degree of correlation is shown in Table 2. The correlations of the humic-like fluorophores C1 ( $r = 0.87$ ;  $p < 0.05$ ) and protein-like fluorophores C2 ( $r = 0.90$ ;  $p < 0.05$ ) are higher

**Table 1.** The two components characterized in this study compared to components from previous studies. Values in the brackets represent secondary peaks.

Component	Ex/Em	Description	References
C1	230 (350)/430	Marine and terrestrial humic substances	
		Component 1: 240 (310)/400-450	[14]
		Component C3:250 (340)/420	[8]
C2	<250 (300-320)/ 340-360	Amino acids, free or protein bound	
		Component C7: 240 (300)/338	[6]
		Component 4: <260 (305)/378	[15]



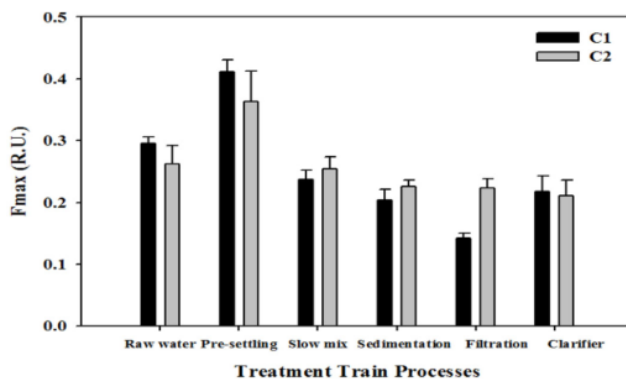
**Fig. 2.** PARAFAC model from FEEMs data identified two components.

with DOC than with UV<sub>254</sub> and SUVA value. This result is as expected, since DOC represent a detection of all organic carbon, including humic and non-humic substances, while UV<sub>254</sub> is more related to aromatic structures and conjugated C double bonds. The aromatic organic fraction absorbs more UV light per unit concentration of DOC organic carbon [11]. This results is in accordance with previous studies which found that DOC, UV<sub>254</sub>, SUVA value and Fmax demonstrate a correlation though chromatographic methods, which apply a fractionation technique based on molecular weight, could provide the higher correlation than the former [8, 19]. In addition, The correlation among C1 and C2 with UV<sub>254</sub> and SUVA indicated that C2 correlates with UV<sub>254</sub> and SUVA than C1 does. Higher UV<sub>254</sub> is a surrogate indicator of the relative density of aromatic functional groups and conjugated double bonds, while higher SUVA indicates to higher aromaticity and hydrophobicity. It means that protein-like fluorophores has a stronger correlation with aromatic bond and hydrophobicity of organic fractions than humic-like fluorophores. This results is because humic structures may incorporate protein-like fluorophores as a result of weak interactions

based on  $\pi - \pi$  or van der Waals forces between the dissolved organic matter components. Previous studies indicate that proteins and humic supramolecules containing certain structures, which was attained from phenol or aniline, may contribute to protein-like fluorescence. Therefore, it is conjectured that protein-like fluorophores showed a higher correlation than humic-like fluorophores [18, 20].

#### 4. Conclusion

Tracking the concentration of DOC and its constituents through the Lu Jhu water treatment plant with pre-chlorination, coagulation, sedimentation, rapid filtration, and post-chlorination, the DOC removal through the whole treatment process train is about 20%. Based on the result from PARAFAC analysis of the FEEMs spectra of samples from water treatment process train, humic substances, mainly fulvic acid-like and aromatic protein-like (II) (tryptophan-like) are the two major DOC components. FEEMs identified that fulvic acid-like shows a higher removal than aromatic protein-like. The correlations of the fulvic-like fluorophores and protein-like fluorophores are



**Fig. 3.** The average of maximum fluorescence intensity ( $F_{\max}$ ) of PARAFAC components through water treatment processes.

**Table 2.** Correlation analysis between organic components and bulk parameters.

	Component C1	Component C2	DOC	UV <sub>254</sub>	SUVA
Component C1	1.00	0.50	0.87	0.79	0.72
Component C2		1.00	0.90	0.83	0.75
DOC			1.00	0.89	0.78
UV <sub>254</sub>				1.00	0.75
SUVA					1.00

higher with DOC than with UV<sub>254</sub> and SUVA value. In addition to bulk parameters analysis, FEEMs are useful tools to elucidate the DOC constituents in the raw water, and to trace their changing during various water treatment processes.

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