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MODELING THE FORMATION OF CHLORINATION BY-PRODUCTS DURING ENHANCED COAGULATION

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Abstract. Because of increasing need to balance health risks for pathogen control and disinfection by-products (DBP) formation in drinking water supplies, water utilities are forced to closely examine and optimize their disinfection practices. This research was designed to investigate the effects of independent variables of dissolved organic carbon (DOC), ferric chloride dosage, chlorine dose, and reaction time on trihalomethanes (THMs) formation in Terkos Lake Water (TLW) of Istanbul City. A statistically-based empirical model was developed for predicting THM formation during enhanced coagulation. The R^2 and F value of model were 0.762 and 460, respectively. The model was found to be statistically significant for all four variables, and model predictions appear to be most accurate for this study. A multiple linear model exhibited the best fit of data. It was observed that THM formation depended primarily on DOC removal. Model calibration, testing and validation were accomplished by using independent data set.

Keywords: disinfection by-products (DBPs), trihalomethanes (THMs), enhanced coagulation, dissolved organic carbon (DOC) removal

1. Introduction

A major objective of drinking water treatment is to provide microbiologically safe water to consumers. The combination of water treatment and disinfection processes has proved to be one of the major public health advances in modern times. In water industry, chlorine is most often the final disinfectant added to treated water for disinfection purposes before it is discharged into water distribution systems (White, 1992). On the other hand, chlorination of organic precursor compounds in water, such as humic and fulvic acid substances results in formation of trihalomethanes (THMs), haloacedic acids (HAAs) and other disinfection by-products (DBPs) (Rook, 1974; Bellar *et al.*, 1974; Crozes *et al.*, 1995; Krasner *et al.*, 1995). Generation of these organic by-products has been shown to be a function of various water quality and operational parameters, including dissolved organic carbon (DOC), type of coagulant and dosage, chlorination level, and reaction time (Sinha, 1998; Uyak and Toroz, 2005).

In natural waters there are many types of organic compounds. All these compounds together are called natural organic matter (NOM). The quantity of NOM may be presented by amount of organic carbon. Approximately 50 percent of DOC, which is defined operationally as the fraction of NOM less than 0.45 μm in diameter, has been identified as humic substances, which is the dominant group of organic compounds in water (Thurman, 1985). Humic substances, primarily humic and fulvic acids, constitute most of NOM in water supplies. The major component of aquatic humic substances is fulvic acid, which is soluble at pH 1. Another component of humic substances is humic acid which is insoluble at pH 1 and has higher molecular weight than fulvic acid.

The approach for THM control employed in this research was enhanced coagulation of precursor removal before chlorination. Several alternatives are available for precursor removal. Enhanced coagulation was selected as a best available technology by United States of America (USA) Environmental Protection Agency (EPA) for precursor control process (USEPA, 1998). Removing humic substances with enhanced coagulation may allow the continued use of chlorine as a disinfectant in the water treatment plants and reduce the numbers and amounts of chlorination by-products (Uyak and Toroz, 2005). Further more, removal of organic compounds can lower chlorine dose required for disinfection purposes because of the reduction of chlorine demand exerted by the organic precursor compounds.

As most of the complex chemistry behind THM formation is unknown, and some difficulties exist in using simple stoichiometry/reaction rate theory to model THM formation under multiple variable conditions. The objective of this research was to examine enhanced coagulation effects on THM formation. Based on THM formation experiments, a data base was established for modeling THM formation following chlorination of enhanced coagulated water. Terkos Lake Water (TLW) in Istanbul city was employed in the jar test experiments. Model variables were coagulant dosage, chlorine dosage, reaction time, and raw water DOC value. The goodness of fit was evaluated through examination of various statistical parameters, including R^2, the F statistic, and α value (significance) for linear regression.

2. Material and Methods

2.1. SAMPLING

The natural water source used in this study was TLW, which originates at Terkos watershed delta. Table I summarizes the raw water quality data encountered during testing facilities. The raw waters were collected prior to any pretreatment. The raw water samples were collected as grab samples, and they were shipped to a laboratory on the same day. When the samples were received at the laboratory, they were kept in the dark in a refrigerator at 4 °C to retard biological activity prior to use. On the other hand, all glassware used during analysis were placed in a detergent bath over night, rinsed four times with hot tap water, rinsed two times ultra pure water and acetone and then placed in an oven at 150 °C overnight.

TABLE I
Terkos Lake water quality parameters

Variables	Unit	Minimum	Maximum	Averages	Std. Deviation
DOC	mg/L	4.10	5.95	4.66	0.58
UV_{254}	1/cm	0.080	0.180	0.127	0.05
SUVA	L/mg-m	1.95	3.02	2.72	0.74
Alkalinity	mg $CaCO_3$/L	98	142	120	31.11
Br^-	μg/L	180	360	270	127.27
pH	–	7.65	8.45	8.05	0.57
Temperature	°C	12.30	24.20	18.25	8.41
THMFP	μg/L	182	290	236	78.79

2.2. Enhanced coagulation jar test procedure

Jar tests were performed using Phipps and Bird six paddle jar test apparatus. The jars were round beakers with one liter capacity. The reagent grade ferric chloride ($FeCl_3 * 6H_2O$) doses were varied 20 to 120 mg/l. The jar test mixing conditions were as follows: rapid mix at 150 rpm for 2 minutes, flocculation at 30 rpm for 30 minutes. After coagulation, jars were allowed to settle for 60 minutes prior to filtration through a 0.45 μm membrane filter. The filtrate from each jar was used to perform DOC, UV_{254}, and THM formation potential (THMFP) studies.

2.3. Sample chlorination procedure

After filtration with 0.45 μm membrane filter, chlorination of TLW samples was carried out at pH 7.0 with phosphate buffer solution. An appropriate amount of concentrated sodium hypochlorite dosing solution was added to the raw and coagulated waters to obtain the desired chlorine dose. The chlorinated sample was then incubated in a dark room for 6, 12, and 24 h at 20 °C. The free chlorine residual was eliminated in the sample bottle, using ammonium chloride solution. Subsequent to the reaction period, the chlorine residual concentrations were measured using the DPD ferrous titrimetric method in accordance with the Standard Methods (APHA, 1998).

3. Experimental Analysis

3.1. Chlorine analysis

Chlorine measurements were conducted using the DPD titrimetric method of 4500-Cl-F (APHA, 1998). This method involves addition of an indicator solution of diethylphenyldiamine (DPD) to the sample after addition of a phosphate buffer. The DPD turns red when oxidized by the chlorine. The sample is then titrated with ferrous ammonium sulfate (FAS), which reduces the DPD. The chlorine concentration is determined from the volume of FAS titrant required to return the DPD to colorless form.

3.2. DOC ANALYSIS

DOC measurements were performed with a Shimadzu TOC-5000 analyzer equipped with an auto-sampler, according to the combustion-infrared method as described in the Standard Methods (APHA, 1998). The sample is injected into a heated reaction chamber packed with a platinum-oxide catalyst oxidizer to oxidize organic carbon to CO_2 gas. Inorganic CO_2 is measured by non-dispersive infrared analyzers and related to an equivalent concentration of organic carbon. The instrument provided reliable, accurate, and reproducible data with a minimum detection limit of 2 μg/LC.

3.3. UV_{254} ANALYSIS

UV_{254} absorbance was measured using a Shimadzu 1601 UV/Vis spectrophotometer at a wavelength of 254 nm with a 1 cm quartz cell. The samples were first filtered through a prewashed 0.45 μm filter to remove turbidity, which can interfere with this measurement, and, a blank with distilled ultra filtered (DIUF) water was run prior to sample analysis. The cell was rinsed with DIUF water and sample prior to being filled with sample for each absorbance measurement. Duplicate analyses were performed on each sample, and the average was reported.

3.4. THM ANALYSIS

THM concentrations were determined using liquid-liquid extraction gas chromatography (GC) according to EPA 551 method (USEPA, 1990). The sum of the four trihalomethanes (chloroform, bromodichloromethane, dibromochloromethane, and bromoform) mass concentrations was reported as TTHM in micrograms per liter. 35 mL THM samples were pipetted into a 40 mL screw cap vials, after that, 3 mL of pentane were transferred to each vial. The vials were then shaken vigorously phase separation. The pentane extract was analyzed by a Hewlett Packard Gas Chromatography 6890 Series II with an electron capture detector (ECD) and capillary column. The system was supported by a HP Chemstation software. The carrier gas used was helium and the make-up gas was nitrogen. The column used for gas chromatography electron capture detector (GC-ECD) analysis was fused silica DB-1, 30 m $\times 0.32$ mm I.D.$\times$ 1.0 μm film thicknesses. The injection technique was split/splitless and the carrier gas flow was 1.6 ml/min. The minimum reporting levels (MRLs) obtained with this method are 0.1 μg/L.

4. Statistical Methods

4.1. Model data base generation

During model construction, TLW was studied in a series of experiments that encompassed the model variables and ranges of conditions. Three values Cl_2 dose was used to represent water treatment plants applied chlorine dose ranges. 2 mg/L was represented as a lower applied dose, while 4 mg/L was used as an average applied chlorine level, and finally 6 mg/L was applied as a higher chlorine dosage for treatment operations. On the other hand, reaction times were varied between 6 h and 24 h. 6 h was represented the minimum time that consumer receive treated water from water treatment plant, while 12 h was used as an average water receiving time for consumer, and finally 24 h was used as higher travel time to consumer from the treatment plant. Moreover, 6 values of ferric chloride dosage were used to represent the water treatment plants coagulation operation ranges. They were 20, 40, 60, 80, 100, and 120 mg/L. 20 mg/L ferric chloride was used as a lower coagulant dosage during treatment operations, and 60 mg/L was used as an average coagulant dosage for coagulation processes, and finally 80, 100, and 120 mg/L ferric chloride dosage were used as an enhanced coagulation dosage conditions for water treatment.

Kinetic experiments were conducted under various combinations of experimental conditions. A base line condition was conventional coagulation with ferric chloride dosages of 20, 40, and 60 mg/L. While, enhanced conditions was enhanced coagulation by ferric chloride dosages of 80, 100, and 120 mg/L.

4.2. Model development

In this study, multiple linear regression analysis was used to model THM formation during enhanced coagulation process. The general strategy adopted to develop model equation was to describe the roles of DOC precursor, chlorine dose, and reaction time in formation of THM. The model coefficients obtained were used to predict THM formation. The concentrations of THM are expressed as (μg/L), the DOC as mg/L, the coagulant and chlorine dose as mg/L, while the contact time as hour (h). The model development was based on the THM concentrations from chlorination of coagulated water. Before model construction, log transformations were applied to all data. Then, a multiple linear regression model for THM formation was created using statistical package for social sciences (SPSS) software (SPSS, 2002). Multiple regression analysis was applied to evaluate the statistically significant variables of the system. The level of significance (α) for the inclusion of a variable in the model was 0.05. Throughout the process of model development, several linear and non-linear regression analyses were performed. Independent sets of data from same sources were collected to validate the model. A total 60 samples were collected from raw water for purpose of model development and validation.

5. Results and Discussion

The model building process requires an understanding of the effects of individual parameters of independent variables on total THM formation. The effects of DOC concentration, coagulant dosage, chlorine dose, and reaction time were discussed individually. The characteristics of TLW used in this study are shown in Table I. The raw water DOC concentrations ranged from 4.10 and 5.95 mg/L, and the UV_{254} absorption ranged from 0.080 to 0.180 cm^{-1}. The values are in the range of typical surface waters.

Enhanced coagulation reduced the DOC level by 62%, and the UV_{254} absorption by 74%, suggesting that enhanced coagulation with ferric treatment preferentially removed organic compounds with higher UV absorption (Figure 1). From Figure 1, the percent reductions of UV_{254} absorbance were higher than those of DOC in TLW. The humic fraction, which absorbs UV light at 254 nm, is more effectively removed by coagulation than the nonhumic fraction (Edzwald *et al.*, 1985; Najm *et al.*, 1994; Krasner *et al.*, 1995). This explanation also accounts for the greater percent removal of UV_{254} absorbance and DOC in TLW. As stated in Table I, specific ultraviolet absorbance (SUVA) value was 2.72 representing average humic content for TLW. The moderate humic fraction in TLW results in greater humic fraction removal and greater overall NOM removal. The UV_{254} generally decreases with increasing ferric dose, indicating preferential removal of the humic fraction. As a comparison with Musikavong *et al.*, study, they found that the removal of aromatic substances by alum and ferric chloride coagulation is more effective than the removal of DOC,

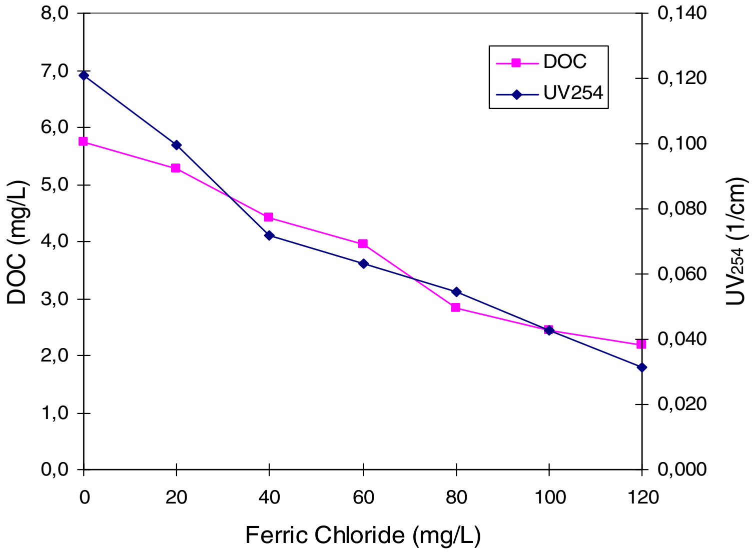

Figure 1. THMs precursors removal as a function of ferric chloride dose.

and it can be concluded from these results that bigger aromatic molecules are removed more effectively than both smaller aromatic molecules (Musikavong *et al.*, 2005). This result supports the conclusion that TLW's NOM was as amenable to chemical coagulation because of its moderate humic fraction. Therefore, enhanced coagulation is very effective for this water source.

5.1. EFFECTS OF DOC CONCENTRATION ON THM FORMATION

Enhanced coagulated samples contained DOC ranging from 5.75 to 2.17 mg/L; Figure 1 shows the DOC reduction during enhanced coagulation. Ferric chloride coagulation removed DOC content of TLW 62%. On the other hand, it is shown in Figure 2 and Table III, a linear relationship (0.732) between DOC and THM formation was obtained with ferric chloride under experimental kinetic conditions (pH = 7.0, Br^- = ambient, reaction time = 6, 12, and 24 h, and chlorine dose = 2, 4,and 6 mg/L). Most researchers found that THM formation rose with increasing soluble humic substances in surface water supplies (Uyak *et al.*, 2005). Moreover, the rate of THM formation is equal to that of DOC consumption, and a first order reaction was reported with respect to DOC (Milot and Rodriquez, 2000). In other words, higher level of DOC will provide more THM formation if enough residual chlorine is available. As shown in Figure 3, increased DOC removal in enhanced coagulated TLW sources resulted in 62% reduction in THM levels. Previous studies indicated that DOC may be used as a surrogate of THM precursors because humic substances typically comprise 50% of DOC in surface water supplies. Adin *et al.* (1991) investigated the formation of THM and their experimental results indicated

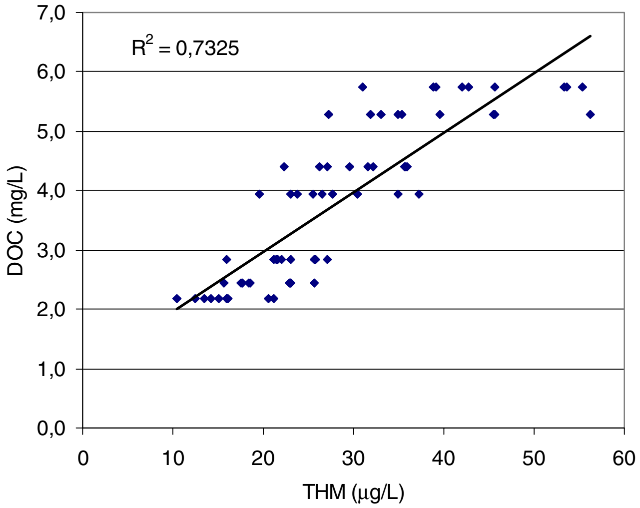

Figure 2. Measured THM values versus DOC values in coagulated TLW.

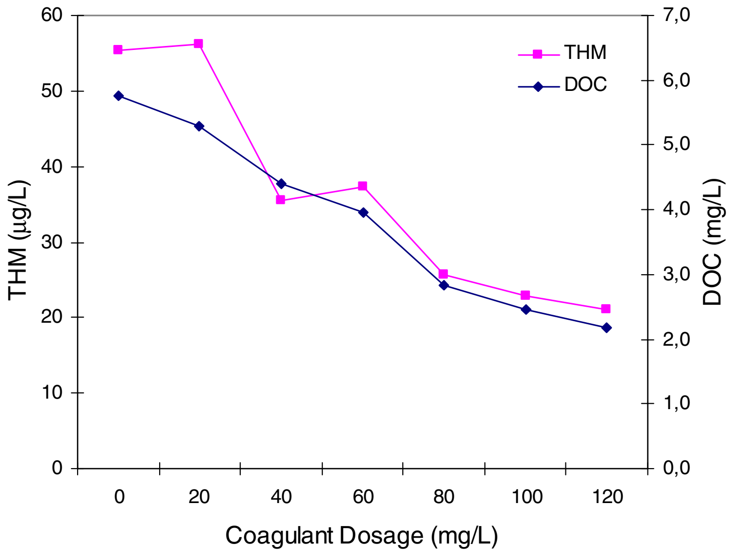

Figure 3. DOC removal and THM formation in coagulated TLW.

that under fixed chlorine levels and reaction times of 24 h, THM production increased until the chlorine was depleted and then decreased while fulvic or humic acids concentrations were increasing consistently (Adin *et al.*, 1991). However, most investigators found that at low concentrations of humics, in the presence of excess chlorine, THM formation rose with increasing dissolved organic matter with a pseudo first order reaction of DOC (Kavanaugh *et al.*, 1980; Babcock and Singer, 1979).

5.2. Effects of coagulant dosage on THM formation

Enhanced coagulation of THM formation removal was usually improved by increasing the ferric dosage, and the most effective THM removals (62%) were achieved at the highest ferric dosages of 120 mg/L with a pH value of 6.3 (Figure 3) for 24 h reaction time. Increasing the dosage of ferric coagulant not only increases the availability of ferric hydroxides potentially able to adsorb THMFP, but also increase the proton concentration, i.e., decreases the pH. The change in pH may be as or more important in enhancing THMFP removal. The effect of $FeCl_3$ dose on the removal of THM species in TLW is shown in Figure 4. It was found that, similar to total THM, the percent removals of THM species increase as the $FeCl_3$ dose increases. From Figure 4, the maximum removals attained for chloroform the most dominant specie at 120 mg/L $FeCl_3$ are 70%. At maximum ferric dose of 120 mg/L, enhanced removal is due to increased NOM adsorption onto amorphous ferric hydroxide precipitates (Krasner and Amy, 1995).

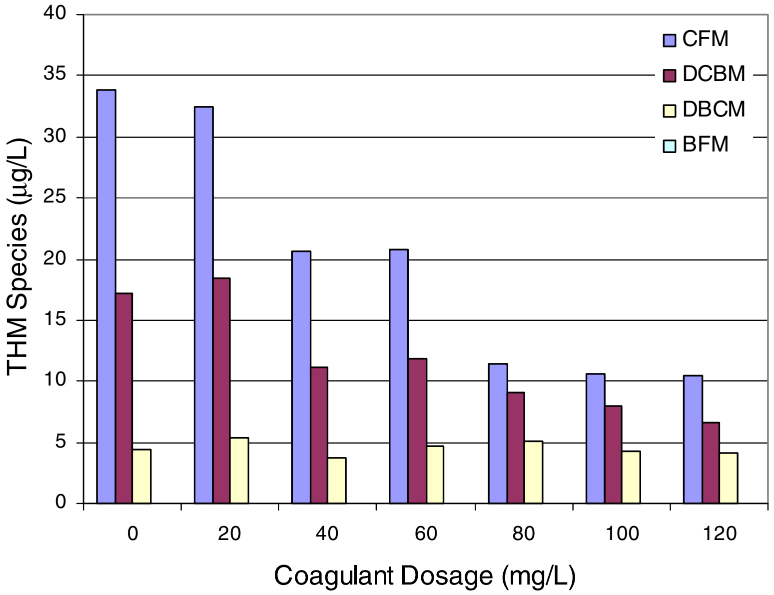

Figure 4. THMs species formation as a function of ferric chloride dose.

5.3. EFFECTS OF CHLORINE DOSAGE ON THM FORMATION

Attempts were made to determine the effect of chlorination dosage on the production of THM in enhanced coagulated TLW (Figure 5). Using Pearson correlation method, a low but definite small relationship ($R^2 = 0.365$) was obtained between THM formation and total chlorine dosage for TLW (Table IV). Table III shows the regression test for correlation of THM formation with chlorine dosage for this water supply. Addition of chlorine to water leads to the formation of hypochlorous acid (HOCl) and hypochloride ion (OCl^-). The formation of these species depends on the pH. In acidic solution HOCl is dominant, whereas in the alkaline solution formation of OCl^- dominates. In the case of enhanced coagulation for TLW, the pH value of the drinking water ranges from 8.05 to 6.30. As such hypochlorous acid is more prevalent chlorine specie which is responsible for the formation of THM. As such when THM concentration increases, the concentration of HOCl decreased.

5.4. EFFECTS OF REACTION TIME ON THM FORMATION

In this study, a series of kinetic experiments was conducted for raw, and each coagulated water samples to provide THM formation versus three reaction times (6, 12, and 24 h) under a chlorine range of 2, 4, and 6 mg/L, and for incremental additions of coagulated water (20 to 120 mg/L). The reaction time value ranged from 6.0 h to 24 h which was associated to the formation of THMs values in the range

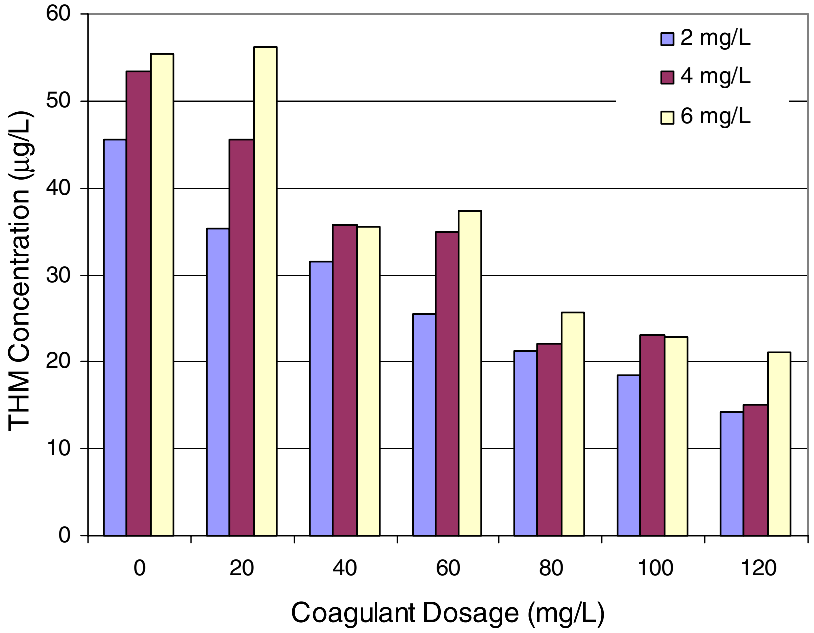

Figure 5. THMs formation as a function of chlorine dose.

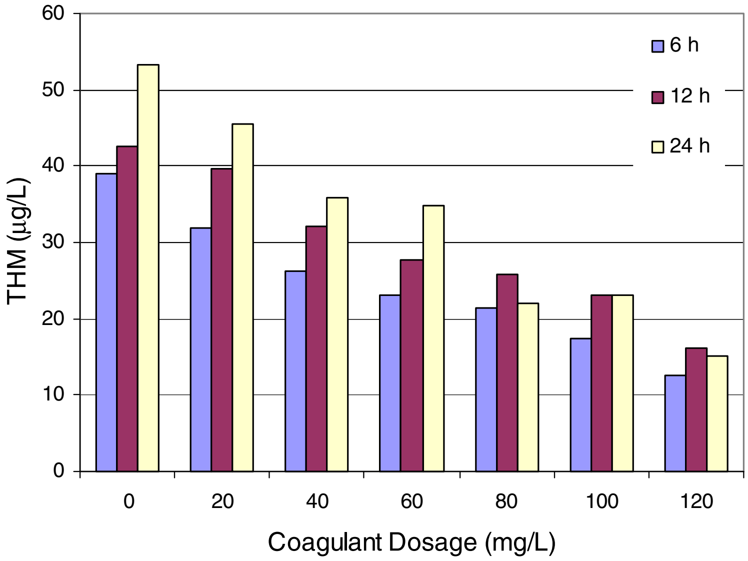

Figure 6. THMs formation as a function of reaction time.

of 10 μg/L-56 μg/L (Figure 6). Since ferric coagulation removed the NOM from raw water, and this influence the amount and rate of THM formation in coagulated TLW (Figure 6). Figure 6 provides THM data versus reaction time for TLW. THM formation rates were initially rapid, corresponding with the rapid consumption of chlorine, followed by a slower, declining rate of production. Approximately, 77% of the THM was formed within the first 6 h, and 94% of THM formation correspond 12 h reaction time in comparison to final 24 h THM formation potential. These findings are similar to the results of Krasner *et al.* (1989), and Sinha (1999) also reported that about 70 to 90% of the THM was formed within the first 24 h when compare to 172 h THM formation potential. However, Pearson regression tests indicated weak correlation ($r = 0.141$) between reaction time and the formation of THM for enhanced coagulated water. THM formation is thought to be a multi-stage process. The initial reaction of chlorine with NOM produces intermediates, which react further via various pathways to produce THM, and other final products (Urano *et al.*, 1983). The trends of THM formation depends on some parameters, such as chlorine dose, and the concentration of NOM.

5.5. MULTIPLE LINEAR THM FORMATION MODELING

Modeling consists of measured THM with the operational and water quality parameters which are responsible for THM formation. The data base created for modeling THM formation in enhanced coagulated samples was based on TLW. Using a SPSS statistical procedure, THM formation model was developed. THM was predicted from raw and ferric coagulated DOC value, ferric dosage, applied chlorine dose, and reaction time. The predictive linear coagulated water model for THM formation was formulated as shown below. Table II shows the model accuracy. The R^2 and F value of model were 0.762 and 460, respectively. This model was found to be statistically significant for all four variables (Table III).

$$\begin{aligned}\mathrm{TTHM} = {} & 10^{1.414} \times (\mathrm{DOC} - 2.1)^{0.199} \\ & \times (\mathrm{Ferric} + 1.56)^{-0.066} \times \mathrm{Cl}_2^{0.242} \times \mathrm{Time}^{0.210}\end{aligned}$$

On the other hand, Figure 7 shows the normal probability plot of measured *vs.* the predicted values of THM. In this figure, all the points are near the straight line with a R^2 value of 0.877. Besides, Figure 8 demonstrates the comparison of measured and predicted THM values. As seen in this figure, predicted THM curve overlaps the measured THM curve in most cases. Moreover, model predictions appear to be most accurate for this study.

TABLE II
Model Summary

R	R^2	Adjusted R^2	F value	Standard error of the estimate
0.873	0.762	0.745	460	0.09459

TABLE III
Regression analysis coefficients

Variables	β	Standard errors	t-values	p-level
Constant	1.354	0.124	10.921	0.000
$\log(\text{Ferric}+1.56)^{0.2}$	-0.253	0.100	-2.525	0.014
$\log(\text{Cl}_2)$	0.343	0.060	5.704	0.000
log(Time)	0.107	0.049	2.202	0.032
log(DOC-2.1)	0.210	0.025	8.422	0.000

TABLE IV
Pearson correlation matrix for model variables

	Ferric	Cl_2	Time	DOC	THMs
Ferric	1.000				
Cl_2	0.000	1.000			
Time	0.000	0.000	1.000		
DOC	-0.563	0.000	0.000	1.000	
THMs	-0.563	0.365	0.141	0.763	1.000

Q2

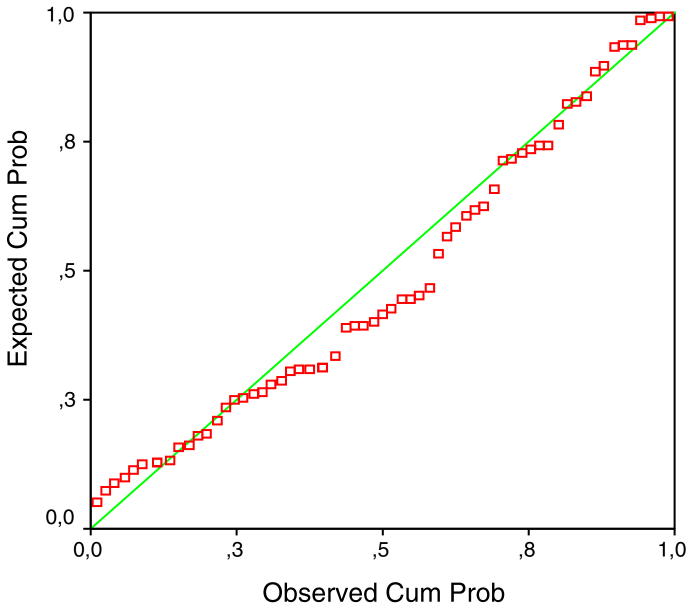

Figure 7. Goodness of the fit of the model for predicted and measured values of THMs.

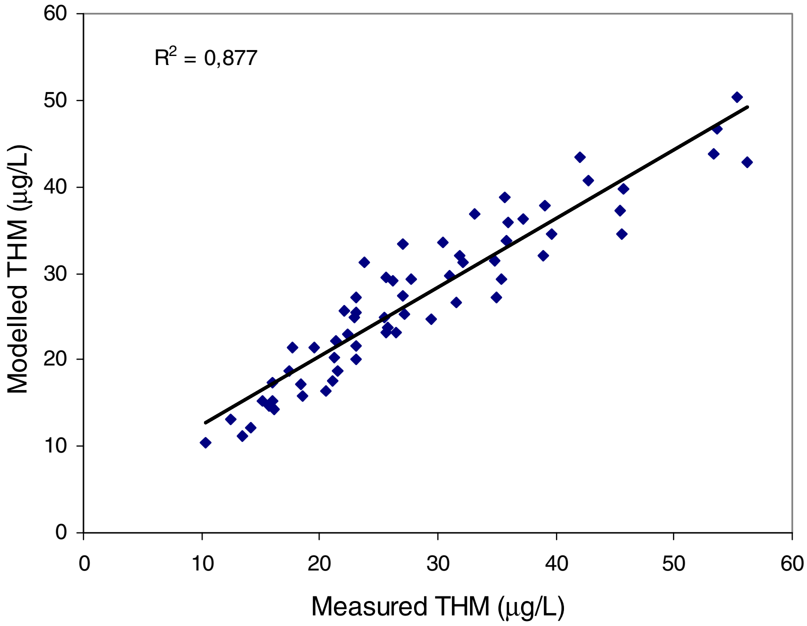

Figure 8. Validation of the model for predicted and measured values of THMs.

5.6. MODEL VALIDATION

Validation of a fitted regression model is the confirmation that the model is sound and effective for the purpose for which it was intended. Validation model requires assessing the effectiveness of the fitted equation against an independence set of data, is essential if confidence in the model is to be expected (Abdullah *et al.*, 2003). Analyses were done to determine the mean square error of prediction (MSEP). MSEP is defined as the average square difference between independent observation and prediction from the fitted equation for the corresponding values of the independent variable (Drapper and Smith, 1981). Using independent set of data obtained from experimental chlorinated TLW and based on regression coefficients, and the calculation of percentage error in prediction, it was found that linear model have higher R^2 value of 0.857. Qualitatively, a visual inspection of a plot of measured values vs. predicted values can serve as a measure of model fitness (Uyak *et al.*, 2005). The goodness of the fit was evaluated through examination of various statistical indicators, including R^2, and F-statistic, while validation was focused on the slope and the intercept of predicted versus measured values of R^2 (Adin *et al.*, 1991). The results of the validation analysis are shown in Figure 8, which shows measured vs. predicted THM values in chlorinated TLW. The model validation indicates reasonable predictions of THM with R^2 value of 0.857.

6. Conclusion

Enhanced coagulation reduced the DOC level by 62%, and the UV_{254} absorption by 74% from TLW, suggesting that enhanced coagulation with ferric treatment preferentially removed organic compounds with higher UV absorption. This result supports the conclusion that TLW's NOM was as amenable to chemical coagulation because of its moderate humic fraction. Enhanced coagulation of THM formation removal was usually improved by increasing the ferric dosage, and the most effective THM removals (62%) were achieved at the highest ferric dosages of 120 mg/L with a pH value of 6.3. Increasing the dosage of ferric coagulant not only increases the availability of ferric hydroxides potentially able to adsorb THMFP, but also increase the proton concentration, i.e., decreases the pH. Moreover, increased DOC removal in enhanced coagulated TLW sources resulted in 62% reduction in THM levels. This article discusses the formulation of a model for predicting THM levels in ferric coagulated TLW in Istanbul. With the use of multiple linear regression techniques, it is possible to develop models for simulating and predicting THMs during the enhanced coagulation treatment.

Based on THM formation experiments, a data base was established for modeling THM formation following chlorination of enhanced coagulated water. The general strategy adopted to develop model equation was to describe the roles of DOC precursor, chlorine dose, and reaction time in formation of THM. The R^2 and F value of model were 0.762 and 460, respectively. This model was found to be statistically significant for all four variables, and model predictions appear to be most accurate for this study. The model validation indicates reasonable predictions of THM with R^2 value of 0.857. Future researches in this area should be directed to formation and modeling of HAAs in Istanbul water supplies.

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