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Critical Study of Multiple Regressions Modelling for Monitoring of Haloacetic Acids in Water Reservoirs

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Abstract Haloacetic acids are disinfection by-products formed during oxidation of natural water. In this study, linear and nonlinear models were developed based on field-scale investigations. These statistical models were applied to predict the formation of haloacetic acids in the treated water. Pearson's correlation was used to check significance of raw water determinants at the probability of 0.05. Multiple forward stepwise regression method was used to develop these models which were then validated using another set of results from the identical source. These predicted results show that linear model was better fit than nonlinear model. A plot of predicted and measured values showed that the Pearson's coefficient and the slope were in good agreement. The developed models can thus be used to reduce the occurrence of haloacetic acids in treated water by means of regulating the raw water quality used for the treatment plant.

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Abbreviations

BCAA	Bromochloroacetic acid
DBAA	Dibromoacetic acid
DBP	Disinfection by-products
DCAA	Dichloroacetic acids
DCBAA	Dichlorobromoacetic acid
HAAs	Haloacetic acids
MAPE	Mean absolute percentage error
MCAA	Monochloroacetic acid
MSEP	Mean square error in prediction
NOM	Natural organic matters
SPSS	Statistical package for social science
SWTP	Semenyih water treatment plant
SWTPR	Semenyih water treatment plant resource
TCAA	Trichloroacetic acid
THMs	Trihalomethanes
TOC	Total organic carbon

1 Introduction

Chlorine disinfectants in surface or raw water interact with organic matter of humic and fulvic acids and form major disinfection by-products like haloacetic acids (HAAs), trihalomethanes (THMs) and many other minor chlorination by-products. Mostly, these HAAs are formed during oxidation and chlorination of natural waters to make it fit for consumption [1]. This formation of THMs, HAAs and other volatile organic compounds (VOCs) has been found to be associated with adverse health effects, and few of them are classified as possible human carcinogens. Globally, these are regulated by several international and national regulatory agencies [2,3].

In the last few years, many modelling attempts have been made to estimate the occurrence of disinfection by-products

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(DBPs) in drinking water. Most of applied empirical models are site-specific, and their predictive capabilities in other water environment under varying conditions remain inappropriate [4,5]. On the basis of laboratory and field data investigations, different models have been developed and proposed [5,6]. The development of a reliable model is mostly recognized as an essential methodological approach for predicting DBP formation in the natural water environment [6]. In the present study, field-scale data have been used to make multiple linear and nonlinear models for HAAs formation. This research was carried out for first time to evaluate the HAAs formation in Semenyih treatment plant of Malaysia, and regression analysis was used to model the occurrence of HAAs. Furthermore, in Malaysia, no such kind of study was carried out for predictive model of HAAs occurrence in water to date. Here, the pH effect, chlorine dose, temperature, contact time, bromide ion concentration and TOC determinants were chosen as variable parameters.

2 Experiments

HAAs formation occurs by reaction of chlorine with natural organic matter (NOM) under the plant-operating conditions including TOC, pH, chlorine dose, contact time and bromide concentration. The real samples were collected for analysis of HAAs from that plant having these operating conditions. The routine analytical and critical parameters for raw water and water fit for treatment plants were the total organic carbons (TOC), ultraviolet light absorbance at 254 nm (UV-254), temperature, chlorine dosage, bromide ion concentration, reaction time and chlorination pH [7]. However, these water quality indicators may vary under different conditions.

2.1 Study Area

Figure 1 shows the location map of Semenyih water treatment plant (SWTP). Water samples were taken at one time in the 3-month intervals from water treatment plant located in Putrajia, Malaysia. This water treatment plant uses conventional water treatment system. Source water was treated to fulfill with World Health Organization (WHO) water quality levels. Further, disinfection of natural organic matter in raw water was also done with the disinfectants.

2.2 Sampling and Storage

Ammonium chloride (dechlorinating reagent) was added as a dry salt solution to the real water sample vial to yield a sample solution of 100 mg/L ammonium chloride. Samples were placed at 50-mL dark-brown glass containers and held at 5 °C and extracted in freezer (10 °C) until further analysis.

All the samples were placed in the organic solvent vapor-free atmosphere.

2.3 Parameters Analysis and Methods

Total organic carbon (TOC) concentrations in water samples were measured according to SM 5310 C procedure of 'Standard Methods 1998', using Aurora-1030, analytical TOC analyzer [8]. Samples were pre-acidified with phosphoric acid. Accuracy was maintained by analyzing routinely a set of calibration standards between 5 and 20 mg/L of TOC. The standards were prepared with potassium hydrogen phthalate in ultra-pure water. A low system background check was performed for ultra-pure water with every run to insure the value less than 0.1 mg/L. The method detection limit was determined to be 0.076 mg/L, by conducting triplicate analysis with 10 percent of the time ensured precision. The average relative standard deviation for the TOC analyzer was 1 %. pH values were measured by the electrometric method according to procedure SM 4500H+B of 'Standard Methods 1998' [8] using a bench-top pH meter and probe (Cornig Model-430; Cat No. 476436). Calibrating the instrument everyday with freshly prepared buffer solution ensured the accuracy of the pH measurements [9]. Conducting duplicates with 10 percent of the time ensured precision. The absolute range was determined to be 0.1 pH units.

Bromide was measured according to procedure 'SM 4110B' of 'Standard Methods 1998' [8]. Bromide samples were pre-filtered with a 0.2-μm filter papers and then were analyzed by an ion chromatography system, i.e., Dionex, Series-4500i, with an AS14A column and with the facility for chemical suppression of eluent conductivity. The reported detection limit was 5 μg/L. For determination of HAAs, GC-MS was used. The detailed analytical protocol for analysis of HAAs was given elsewhere [10]. Sum of the six haloacetic acids' mass concentrations was reported as total six haloacetic acids (HAAs-6) in micrograms per liter (μg/L). The operation procedures and running conditions were discussed elsewhere [10].

2.4 Statistical Data Analysis and Model Development

HAAs data were generated from real water treatment plant reservoir and were analyzed using SPSS, version 16.0. Statistical models were then developed for predicting HAAs formation. Principles for choosing independent variables were to choose the best matching between the dependent variables and the independent variables. This determined the best probability for the coefficient of the independent variables and the weak relationships within the independent variables. Otherwise, independent variables were removed from the models.

Multiple regression analysis was used because of the multi-dimensional nature of the data. The objective of analy-

Fig. 1 Location map of
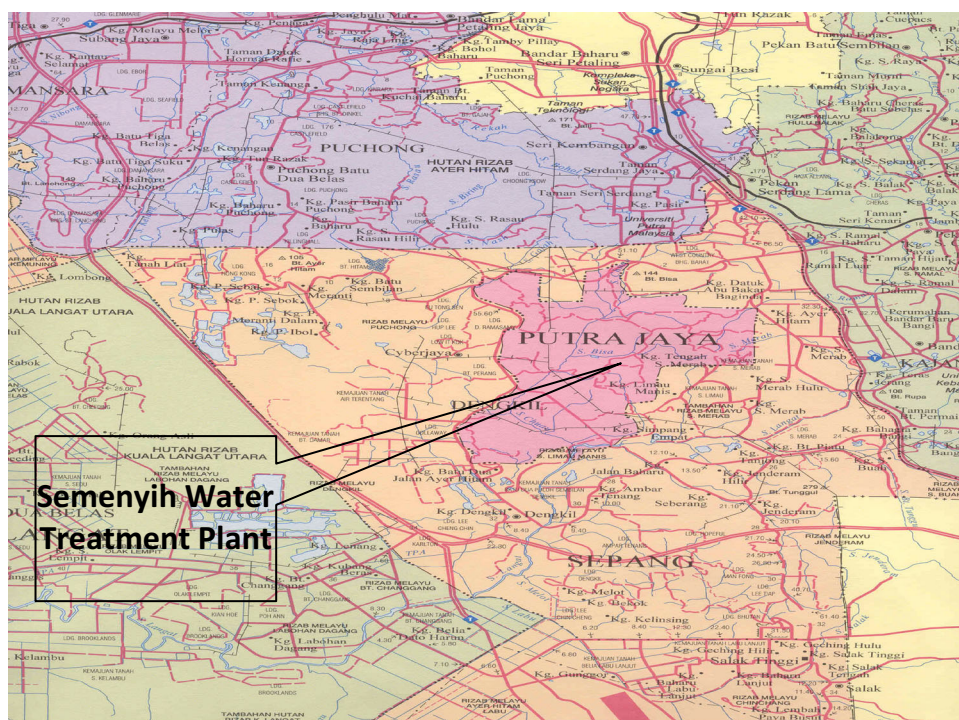Semenyih water treatment plant
(SWTP), Malaysia

Table 1 Raw water quality parameters at Semenyih water treatment plant, Malaysia

Water quality parameters	Mean	Range
pH	6.0	4.7–6.3
Temperature (°C)	25.9	22.2–27.7
Contact time (min)	30	30–30
TOC (mg/L)	3.1	1.0–9.6
Chlorine dose (mg/L)	2.7	2.6–3.8
Br$^-$ (mg/L)	0.02	0.01–0.10
HAAs (mg/L)	0.022	0.009–0.090

sis was to check which variables had the greatest impact on the HAAs presence using multiple regression analysis. Then, those combinations that were promising (large R^2, large F) were used to develop all the possible three-variable and two-variable models [11]. The best-fit model from those considered had the largest R^2 for which removal of variable significantly degraded the quality of the fit.

The real disinfected raw water (Table 1) was used for model development of HAAs presence in the Semenyih water treatment plant (SWTP), while for raw water, pH, temperature, bromide ion concentration, and TOC values, operating setting of contact time, and chlorine dose were used. Multiple regression analysis was performed to evaluate the statistically significant variables of the system, at significance level of 0.05. The inclusion of each variable in the proposed model was based on the t criterion [11,12]. Methodological details about the model development were extensively discussed in the past studies [10].

3 Results and Discussion

The raw water quality characteristics of the Semenyih water treatment plant (SWTP) during sampling are given in Table 1. The reported six HAAs species were monochloroacetic acid, MCAA, dichloroacetic acids, DCAA, trichloroacetic acid, TCAA, bromochloroacetic acid, BCAA, dibromoacetic acid, DBAA, and dichlorobromoacetic acid, DCBAA [13]. Mathematical evaluation of the data indicated that the factors like chlorine dose, TOC and bromine levels were critical for the occurrence of HAAs in this treatment plant.

3.1 HAAs Formation in Semenyih Water Treatment Plant

HAAs formation status in SWTP is given in Fig. 2. The high HAAs value formed were 10.3 µg/L for DCAA, 6.6 µg/L for TCAA, 1.6 µg/L for BCAA, 1.6 µg/L for DBAA, 0.24 µg/L for MCAA and 0.44 µg/L for DCBA. DCAA with 70 % and TCAA with 60 % of total HAAs were formed during disinfection of river water of Semenyih water treatment plant (SWTP). On the other hand, 1.6 µg/L DBAA (>10 % of total HAAs) and DCBAA (1 % of total HAAs) was formed because of moderate limit of Br$^-$ (0.10 µg/L) in SWTP. In the previous research [10], the brominated species were found to be more carcinogenic than the chlorinated species. Here,
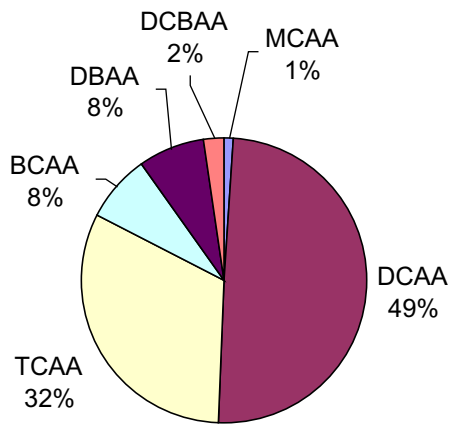
Fig. 2 HAAs formation statuses in SWTP, Malaysia

the levels of brominated species were still safe, but for future safety, SWTP administration needs to investigate the bromine source in the Semenyih river water.

3.2 Factors Affecting the HAAs Formation

Generally, the natural organic matters (NOM) levels are measured as total organic carbon (TOC). Increasing the NOM level affects HAAs formation in two ways. First, increasing the NOM level will increase the level of HAAs precursors, which ultimately increases HAAs formation. Second, increasing the NOM level will increase the concentration of disinfectants. The remaining disinfectant in distribution system could be maintained using proper dosage of Cl_2. This high concentration of disinfectant dosage causes more production of HAAs. HAAs level would be increased with increasing the TOC level and chlorine dosage. In this study at SWTP, the contact time was not changed during chlorination, so it does not really affect the HAAs levels. pH affects the formation of DBPs in many ways. In general, the high pH results in a high level of trihalomethanes (THMs), but a lower level of HAAs formation.

The effect of pH on HAAs formation is quite complex because the rate of formation and hydrolysis vary for each individual species. Uyak and Toroz [11] studied the formation of various species of HAAs with varying the pH, ranged from 4 to 10. According to previous study [10], highest level of monochloroacetic acid (MCAA) was reached at pH 7. However, for bromochloroacetic acid (BCAA), the pH has not big impacts on its formation. Furthermore, from high pH value (pH = 8), a dichloroacetic acid (DCAA) is favored. But, the reverse is true for trichloroacetic acid (TCAA). TCAA formation was also decreased under solutions of high pH, and the rate was increased until pH 8 is reached. After this point, TCAA was decreased gradually [14]. Concentration of dibromoacetic acid (DBAA) increases at higher pH due to medium bromide ion concentration. In the present study of

SWTPR, the pH was ranged from 4 to 7 and in that pH, HAAs would be appeared in dissociated form. At pH 2, this compound would be protonated. So, at pH 4–6 (slightly acidic and neutral), it favors into dissociated form. So here, pH was not significant variable for the formation of HAAs. Temperature range found in the SWTPR was around 22.2–27.7 °C, and it also do not affect the formation of HAAs.

In SWTP, medium level of bromide ion was found around 0.01–0.10 mg/L. Bromide, an inorganic ion, does not react with NOM directly. Bromide can be oxidized by chlorine to hypobromous acids depending on the pH [15]. Due to having almost neutral pH, tri-halogenated specie of HAAs was detected in the treated water, i.e., DCBAA. Otherwise, tri-halogenated species undergo hydrolysis reactions to form THMs at higher pH [16]. The finding of this study indicates that the mean value for chlorine dose, bromide ion concentration and pH was seemed to be controlled in SWTP for formation of haloacetic acid model. Except the mean, the value for total organic carbon was bit high. This may be due to the waste deposal into the river water or might be due to heavy rain water runoff in the river. The detected six major compounds such as MCAA, DCAA, TCAA, BCAA, DBAA and DCBAA could be quantified in all samples. MBAA, DBCAA and TBAA were not detected for all samples under this study [17]. A plot of residual versus predicted values of HAAs for linear and nonlinear model is given in Fig. 3.

The presently discussed multiple linear and nonlinear models of HAAs for SWTP are as follow:
For linear model,

$$HAAs = -0.027 + \ TOC \ (0.003)$$
$$+ \ Cl_2 \ (0.014) + \ Br \ (0.215) \tag{1}$$
$$R^2 = 0.80 \tag{2}$$

For nonlinear model,

$$HAAs \ = 10^{-2.604} \ (TOC)^{0.490} \ (Cl_2)^{1.585} \tag{3}$$
$$R^2 = 0.50 \tag{4}$$

Simple regression analysis was used to examine the correlation of HAAs with respect to pH at the sampling points of Semenyih water treatment plant. The dependent variables of model should follow normal distribution for the accurate fitting. K–S tests indicated that at significance level of 0.05, all dependent variables should fall within the normal distribution [10].

Many attempts were made to determine chlorination dosage effect on the production of HAAs in the SWTPR. Using Pearson's correlation, a moderate relationship ($r = 0.657$) was obtained between HAAs formation and chlorine dose for linear model. But for nonlinear model, medium level of correlation was obtained ($r = 0.510$). Norwood et al. [14] reported that higher chlorine to carbon ratios favor the formation of chlorinated acids. Miller and Uden [17] observed that
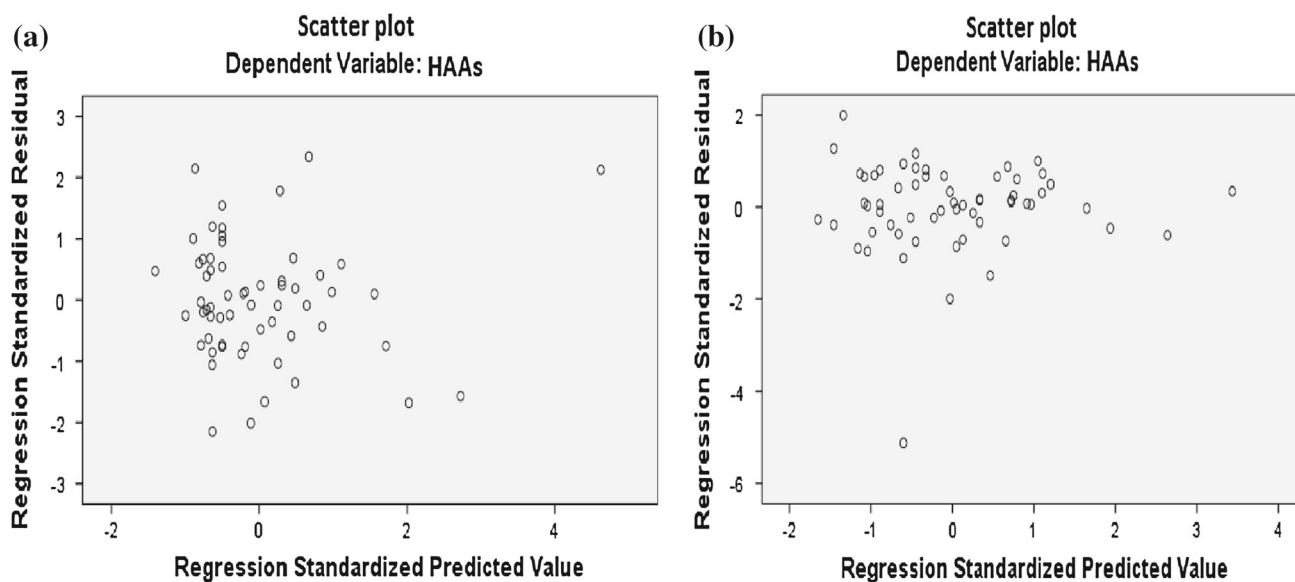
Fig. 3 Plot of residual versus predicted values of HAAs **a** linear, **b** nonlinear model

Table 2 Pearson's correlation coefficients of HAAs linear without log transformation and linear with log transformation models

	TOC	Cl⁻	Br⁻	Temperature	Contact time	pH	HAAs
TOC	1.000						
Cl⁻	0.657	1.000					
Br⁻	0.681	0.626	1.000				
Temperature	0.129	0.229	0.207	1.000			
Contact time	0.000	0.000	0.000	0.000	1.000		
pH	0.133	−0.309	0.141	−0.059	0.000	1.000	
HAAs	0.816	0.657	0.681	0.180	0.000	−0.237	1.000
log TOC	1.000						
log Cl⁻	0.546	1.000					
log Br⁻	0.576	0.523	1.000				
log Temp	0.051	0.772	0.764	1.000			
log Contact time	0.000	0.000	0.000	0.000	1.000		
log pH	−0.167	−0.323	−0.233	−0.056	0.000	1.000	
log HAAs	0.669	0.510	0.550	0.017	0.000	−0.264	1.000

DCAA was formed before the other DBPs, and that TCAA formation required more rigorous oxidation by chlorine than DCAA.

Pearson's correlation coefficients of HAAs linear without log transformation and linear with log transformation models are given in Table 2.

Many studies [10,14,16,18] reported that there is a linear relationship between pH and THM formation but in case of HAAs, inverse relationship ($r = -0.237$) was obtained. For nonlinear model, inverse relationship was obtained ($r = -0.264$) as well. Past studies also support these findings [10]. This might be because of the fact that the oxidation potential is decreased when the pH is increased. So the oxidation prod-

uct, i.e., TCAA, subordinates, while the substitution product, i.e., chloroform, dominates. Summers et al. [15] found that HAAs aromatization decreased with increasing pH. This occurs because at a lower pH, HOCl is the main specie. HOCl concentration is decreased when the pH is increased because of the shift in equilibrium which causes a lower TCAA formation.

Moreover, for linear model, Pearson's correlation matrix illustrates good correlation ($r = 0.816$) between TOC and HAAs. On the contrary, for nonlinear model, moderate relationship ($r = 0.669$) was observed. Chlorine consumption by hydrophilic extracts was found to correlate with aromatic and phenolic contents of TOC as well as the subsequent HAAs

Table 3 Statistical evaluation of linear and nonlinear model

Model	R	R^2	Adjusted R^2	F value	Standard error of the estimate	Durbin–Watson estimate
Linear	0.895	0.801	0.790	75.166	0.0060639	2.0
Nonlinear	0.712	0.507	0.489	30.844	0.17311	2.2
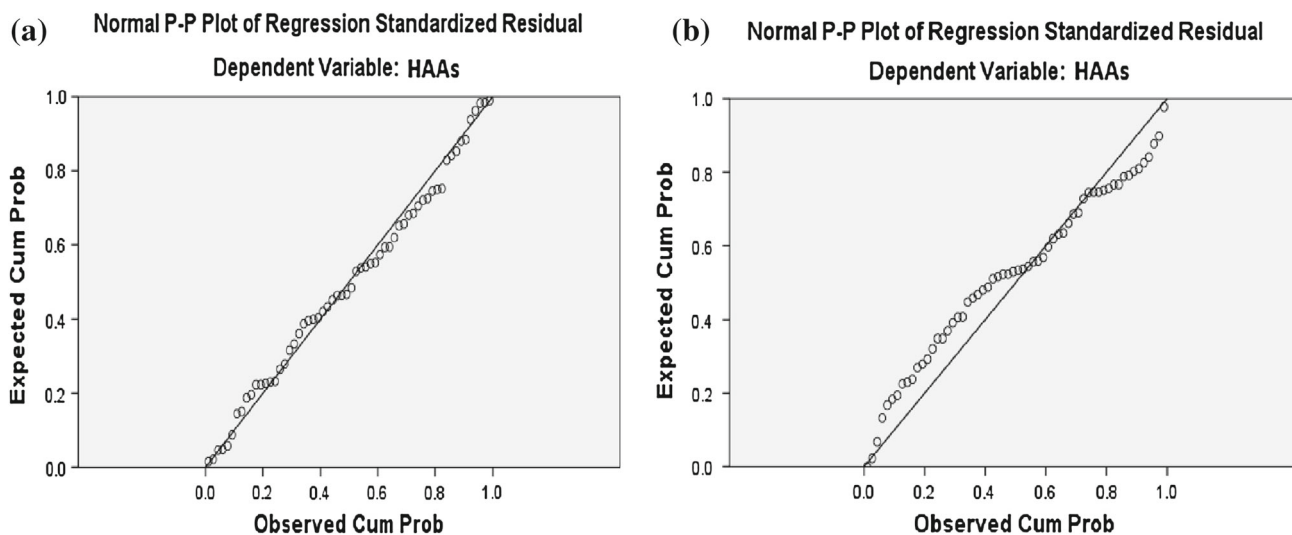
Fig. 4 Comparison between predicted and observed values for **a** linear and **b** nonlinear model

formation [16]. It means that the phenolic and aromatic content of the TOC influenced the incorporation of chlorine and may be associated with precursors to HAAs. The rate of formation of HAAs is equal to that of the TOC consumption. Humic acids have been observed to be more reactive than fulvic acid with the chlorine dose. Reckhow and Singer [19] hypothesized that molecules of a higher degree of conjugation will preferentially lead to the formation of TCAA. The fulvic acid that has a higher content of the methyl-ketone species than the humic acids showed higher TCAA yield than the humic acids. On the other hand, a moderate correlation ($r = 0.681$) was obtained between bromine and HAAs for this linear model. For nonlinear model, medium level of relationship ($r = 0.550$) was obtained. There was no established relationship between contact time and HAAs for the linear model and nonlinear model. This may be due to the same contact time during the chlorination process.

A poor relationship ($r = 0.180$, $r = 0.017$) was obtained between temperature and HAAs for linear and nonlinear model, respectively. This would be due to the fact that there was no wide change in temperature on equatorial climate region and all the samples under studies had constant values of temperature about 22.2–27.7 °C [10]. The mean value of the residual should be zero; otherwise, it should be suspected a calculation error or that additional variable should be added to the regression model [20–23].

Since linear model has higher R^2 value than the nonlinear model and both residuals and predicted values for linear model of HAAs was more close to zero line than nonlinear model of HAAs, the linear model is in good agreement with the predicted and observed values. It means that this multiple linear regression model of HAAs is useful for predicting the level of HAAs in disinfected water distribution. However, as shown in Table 3, the nonlinear model shows comparatively low R^2 value. In this case, predictive capability of linear model is also better than the nonlinear model.

3.3 Model Validation

Developed predictive models for HAAs was validated for an individual dataset of HAAs estimation in water sample received from another sampling period. Results of validation analysis for HAAs are shown in Figs. 4 and 5.

The validation of linear model of HAAs shows very acceptable predictions with R^2 value of 0.74, while validation of nonlinear model of HAAs shows a relatively better prediction with R^2 value of 0.662. These raw water quality variables and operating conditions show the variations of TOC, chlorine dose and bromide ion concentration. Traditional regression procedures are also suitable for the presence of HAAs in river water. Regression analysis coefficients for linear and nonlinear models are given in Table 4.
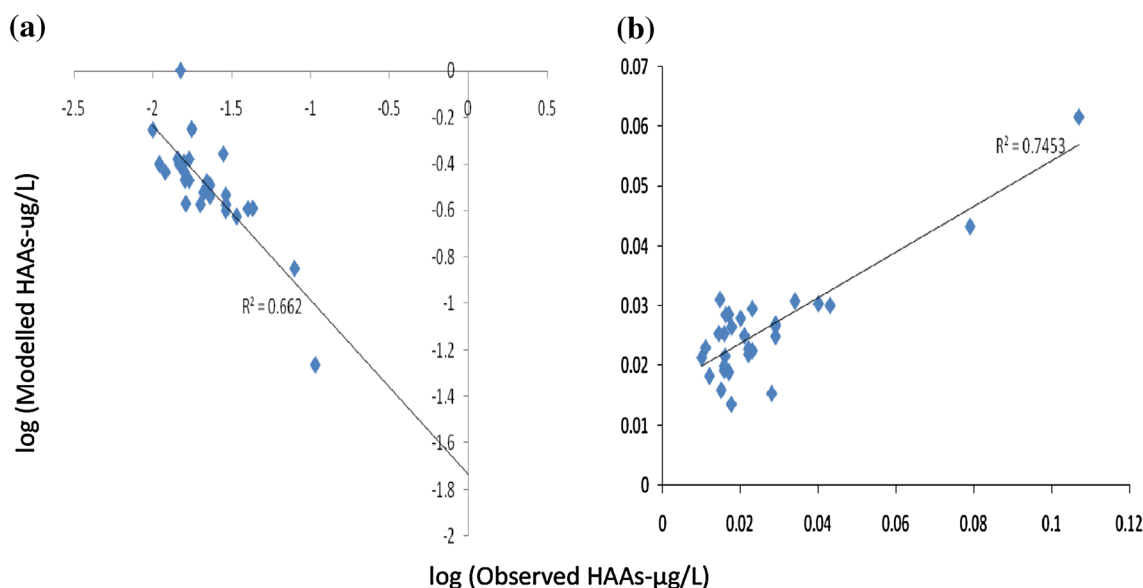
Fig. 5 Comparison between observed and modelled value, **a** nonlinear, **b** linear model

Table 4 Regression analysis coefficient for linear and nonlinear model

	*β	Standard errors	t value	p level
HAAs				
Constant	−0.027	0.009	−3.115	0.003
TOC	0.003	0.001	4.606	0.000
Cl^-	0.014	0.004	3.922	0.000
Br^-	0.215	0.066	3.253	0.002
log HAAs				
Constant	−2.604	0.246	−10.525	0.000
log TOC	0.490	0.105	4.544	0.000
log Cl^-	1.585	0.608	2.610	0.012

*β = parameter estimator or variable estimator or coefficient

Based on the validation results, linear model is considered better than nonlinear model. Mean square error of prediction (MSEP) was tested in order to investigate the predictive capability of these models. Analyses were done to determine the MSEP [23]. It is defined as the average difference between independent observation and prediction from the fitted equation for the corresponding values of the independent variables [20–23].

Using another set of results taken from SWTP and finding of the calculation of error in prediction and absolute percentage error (APE), it was observed that linear model has lower value of prediction error and mean absolute percentage error (0.00017, 38.72) compared with the nonlinear model (1.53, 69.75). Based on these prediction results, it was concluded that linear model is better than nonlinear model.

Modelling results indicate that, with this linear model and nonlinear model, estimation of HAAs levels in chlorinated raw water of SWTP with reliable level of prediction can be done. This could be beneficial for evaluating the human interaction to HAAs in water having data at specific sites. These predictive models can be used to guide the decision making for the operational control during the treatment process. However, it must be observed that the diversity and complexity of HAAs formation reactions make it difficult to develop universally applicable models.

4 Conclusion

Halogenated organic contamination originates due to reaction of chlorine with organic compounds such as propane, ketones, aldehydes and trichloroacetate. The major HAAs species formed during chlorination of raw water of SWTP were DCAA with 70 % and TCAA with 60 % of total HAA. On the other hand, due to medium level of bromide ion (0.10 μg/L) in SWTP, 1.6 μg/L DBAA (>10 % of total HAAs) and DCBAA (1 % of total HAAs) was formed. Using developed models, HAAs formation potentials can be predicted in Semenyih drinking water reservoir. Explanation of observed variability of HAAs for Malaysian water supplies is at optimum level. Based on the obtained validated results, the linear model appeared to be better than the nonlinear model in terms of percentage error of prediction.

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