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Prediction of *N*-nitrosodimethylamine (NDMA) formation as a disinfection by-product

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Abstract

This study investigated the possibility of a statistical model application for the prediction of *N*-nitrosodimethylamine (NDMA) formation. The NDMA formation was studied as a function of monochloramine concentration (0.001–5 mM) at fixed dimethylamine (DMA) concentrations of 0.01 mM or 0.05 mM. Excellent linear correlations were observed between the molar ratio of monochloramine to DMA and the NDMA formation on a log scale at pH 7 and 8. When a developed prediction equation was applied to a previously reported study, a good result was obtained. The statistical model appears to predict adequately NDMA concentrations if other NDMA precursors are excluded. Using the predictive tool, a simple and approximate calculation of NDMA formation can be obtained in drinking water systems.
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1. Introduction

N-Nitrosodimethylamine (NDMA), a disinfection by-product and potent carcinogen, has recently been observed in drinking water supplies. The US EPA integrated risk information system (IRIS) database lists an estimated 10^{-6} lifetime cancer risk level for NDMA in drinking water of 0.7 ng L^{-1} [1].

It has been reported that the use of chloramines or chlorine as a primary disinfectant may increase NDMA concentrations in drinking water treatments [2]. NDMA, one of nitrosamines, can be formed through a chemical reaction between monochloramine and an organic nitrogen compound. Monochloramine is used directly or formed in chlorination of drinking water in the presence of ammonia. Gerecke and Sedlak [3] demonstrated that the yield of NDMA from chloramination of dimethylamine (DMA) was about 0.6% in natural waters. DMA is one of the most frequently detected organic nitrogen compounds in surface water [4]. Also, they suggested that chloramination of surface waters with high dissolved organic carbon (DOC) concentration could result in elevated NDMA formations. In addition, Mitch

and Sedlak [5], and Choi and Valentine [6] showed that NDMA formation during chlorination could occur through unsymmetrical dimethylhydrazine (UDMH) as an intermediate. The overall rate of NDMA formation by the UDMH oxidation is very slow due to slow reactions in the preliminary step.

Mitch et al. [7] studied that the NDMA formation rate varied with pH with a maximum formation rate between pH 7 and 8. They pointed out that NDMA formation via the UDMH pathway has significant implications for disinfection in water and wastewater, because its formation is maximized at pH between 6 and 9, which are typical values during water and wastewater treatment. The rate of UDMH formation increases with pH [8]. Longer contact time during drinking water disinfection and water distribution systems may result in higher NDMA formation [9,10].

Previous investigations indicated that there would likely be relationships between NDMA formation and several parameters such as NDMA precursors and inorganic concentrations. Mitch and Sedlak [11] reported that NDMA formation rate was positively correlated with monochloramine concentration and increased linearly over time due to the very slow reaction rate. Until now, it was possible to assume NDMA formation from assumption of NDMA yield. However, there is a limitation of use in high molar ratio (monochloramine/dimethylamine) and

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presence of other NDMA precursors. Choi and Valentine [12] developed a kinetic model to predict NDMA formations. The model could not include all reaction mechanisms due to the complicated processes. It was an only proposed mechanism to predict NDMA formation.

This study investigated the use of a statistical model application for the prediction of NDMA formation as a disinfection by-product. First, a batch experiment was performed to determine NDMA formation during chloramination of DMA at different pH (7–9). Secondly, from these findings, a prediction model was developed for NDMA formation. A statistical approach was conducted to provide a better understanding of predicted reaction for the NDMA formation. In addition, an application study by the developed regression equation was conducted with a previously reported investigation.

2. Materials and methods

2.1. Materials

N-Nitrosodimethylamine (100 μg mL^{-1} in methanol) was purchased from Ultra Scientific NS-100. NDMA-d6 (1 mg mL^{-1} in methylene chloride) was obtained from Cambride Isopotes Labotratory. Sodium hypochlorite (NaOCl) solution was obtained from Fisher Scientific. Dimethylamine was obtained from Alpha Aesar.

2.2. NDMA formation

Each batch experiment was conducted in a 1-L bottle using deionized water in a dark area at 25($\pm$1) $^{\circ}$C to minimize the photolysis effect. The deionized water was produced from a Milipore water system. Monochloramine solutions were prepared daily by dissolving ammonium chloride in deionized water adjusted to pH 8.4 with 4 mM bicarbonate buffer, and followed by addition of sodium hypochlorite. A molar ratio for hypochlorite to ammonia was 1:1.2. Monochloramine and free chlorine concentrations were measured by the DPD-FAS method from APHA [13].

The mixtures of monochloramine and DMA were allowed to react in the dark for 24 h. NDMA formation was investigated through a reaction between monochloramine and DMA at different pH values (7–9). Buffer solutions used were a mixture of sodium phosphate monobasic and sodium phosphate dibasic,

and sodium bicarbonate (1 or 4 mM). The pH in sample waters was further adjusted by addition of sulfuric acid or sodium hydroxide. The concentrations of monochloramine were varied from 0.001 to 5 mM to obtain the NDMA yields at different molar ratios (monochloramine/dimethylamine). The concentrations of DMA were fixed either at 0.01 or 0.05 mM during the batch experiment.

2.3. NDMA analysis

NDMA concentrations are determined by the solid phase extraction [14]. A 500-mL water sample was added to a 1-L amber bottle with 125 mg of resins (Ambersorb 348F, Aldrich, USA). Ambersorb 572 was replaced by the Ambersorb 348F because the Ambersorb 572 was no longer available for purchase. All water samples were labeled with NDMA-d6 as an internal standard. The bottle was shaken for 70 min at 250 rpm. The water sample was filtered by vacuum filtration to get the resins, which were dried in the air for 1 h. They were transferred to a 2-mL vial with Teflon-faced septum, and 1 mL of methylene chloride was added. A 4-μL of methylene chloride extract was injected into a Varian Saturn GC/MS (2000, USA) with an autosampler (Varian 8200CX, USA) using the chemical ionization mode (positive) with methanol, or a Shimadzu GC/MS (QP5050, Japan) using the electron ionization mode equipped with a Restek Rtx-Vrx column (60 m, 032 mm I.D., 1.8 μm film thickness). The GC temperature program consisted of an initial temperature of 35 $^{\circ}$C held for 4 min, followed by an increase to 100 $^{\circ}$C at 20 $^{\circ}$C/min, and then to 200 $^{\circ}$C at 50 $^{\circ}$C/min. Temperatures of the injector and mass spectrometer ion trap were maintained at 220 and 150 $^{\circ}$C, respectively. Method detection limit (MDL) was 5 ng L^{-1}.

2.4. QA/QC

During the NDMA analysis, several quality assurance/quality controls (QA/QC) were applied. Twenty-five microlitres of 1 mg L^{-1} deuterated NDMA stock solution (NDMA-d6) in methanol solvent was added as an internal standard to each 500 mL water sample. The concentration of NDMA analysis was determined from a calibration curve on the basis of the internal standard. In particular, duplicate NDMA formation studies were conducted at a DMA concentration of 0.01 mM, and a typical summary of result is shown in Table 1. During

Table 1
A duplicate experiment for the NDMA formation at pH 8

DMA (mM)	Mono-chloramine (mM)	Experiment A NDMA formation (μg L^{-1})	Experiment B NDMA formation (μg L^{-1})	Averaged NDMA concentration (μg L^{-1})
0.01	0.05	1.9	1.0	1.5
0.01	0.1	2.4	1.4	1.9
0.01	0.2	3.7	2.9	3.3
0.01	0.5	4.4	4.5	4.5
0.01	1	6.1	6.3	6.2
0.01	2	9.7	9.2	9.4
0.01	5	18	18	18

The two experiments were conducted at 25($\pm$1) $^{\circ}$C at different day. The NDMA formation was corrected by NDMA concentration in blank.

Table 2
The NDMA presence in pure DI water and buffer solutions without monochloramine addition

Sample	NDMA concentration (ng L^{-1})	Comment
DI water	6.4	
Buffer solution (0 h)	5.5	
Buffer solution (24 h)	6.1	
Buffer solution with DMA (0 h)	24	DMA concentration = 0.05 mM
Buffer solution with DMA (24 h)	26	DMA concentration = 0.05 mM

DI water indicates deionized water. Buffer solution contained a mixture of sodium phosphate monobasic and sodium phosphate dibasic, and sodium bicarbonate.

the NDMA formation study, all of two NDMA concentrations were averaged for regression analysis. A low NDMA concentration was found in a monochloramine-free solution with DMA. Table 2 explains NDMA presence in buffer solution with DMA in the absence of monochloramine. The DMA may contain a low NDMA concentration without chloramination. The NDMA formed concentration was determined by subtracting by NDMA concentration in the monochloramine-free solution or in blank from the NDMA analysis result.

2.5. Regression analysis

In order to predict NDMA formed concentrations, a statistical model between the molar ratios (monochloramine/DMA) and NDMA formation on a log scale was developed. Before running the regression the unit of concentration was converted to a dimensionless unit. For the statistical evaluation, a simple linear regression analysis was conducted using Excel. A statistical significance ($p < 0.05$) was utilized for all of regression equations.

3. Results and discussion

3.1. NDMA formation according to pH

NDMA formation was investigated by the reaction of DMA with variable monochloramine concentrations at pH 7, 8 and 9. At an initial DMA concentration of 0.05 mM, the effect of pH on the NDMA formation in terms of ratio (monochloramine/DMA) is shown in Fig. 1. The highest NDMA formation appeared with pH 8. The lower NDMA formation was observed at pH 9 than pH 7. At pH 7 and DMA of 0.05 mM, NDMA ranged from 1.03 to 124 μg L^{-1} approximately at 1 mM of monochloramine concentration. The NDMA formation was 100 μg L^{-1}, which was 2.7 times higher, at a monochloramine concentration of 1 mM and pH 8. In addition, Fig. 2 shows NDMA formation at initial DMA of 0.01 mM. The NDMA formation was proportional to pH, and the highest NDMA was produced at pH 9.

The result showed that pH significantly varied the NDMA formation, which indicated that NDMA formation was dependent on pH value. It is likely that decomposition of monochloramine increased with increasing pH. Choi and Valentine [15] reported that pH dependence seemed to be complicated in that not only speciation of DMA but also stability of intermediates may play an important role. DMA may be dissolved well in aqueous phase
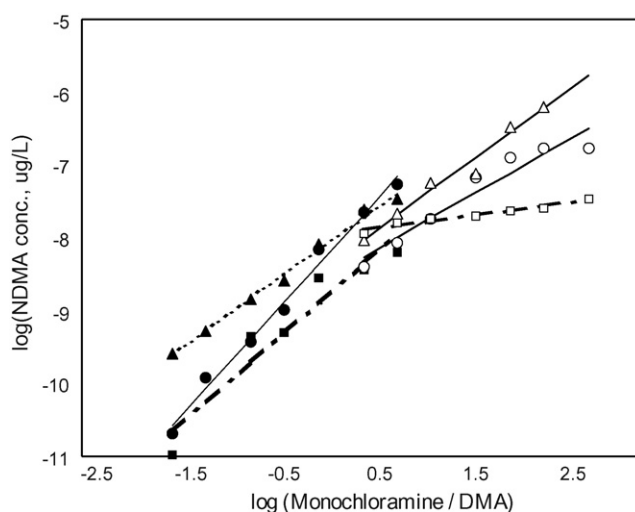

Fig. 1. NDMA formation from monochloramine to DMA ratio at a DMA concentration of 0.05 mM. For monochloramine/DMA < 1: monochloramine = 0.001–0.1 mM; (●) measured (pH 7); (▲) measured (pH 8); (■) measured (pH 9). For monochloramine/DMA > 1: monochloramine = 0.05–5 mM; (○) measured (pH 7); (△) measured (pH 8); (□) measured (pH 9); each line indicates calculated values from regression equations in Table 3. A statistical significance ($p < 0.05$) was utilized for all regression equations.

at high pH due to its high basicity, and then reacted effectively with monochloramine.

3.2. Monochloramine to DMA ratio

Figs. 1 and 2 show the NDMA formation according to molar ratio of monochloramine to DMA (or ratio) at different pH. The
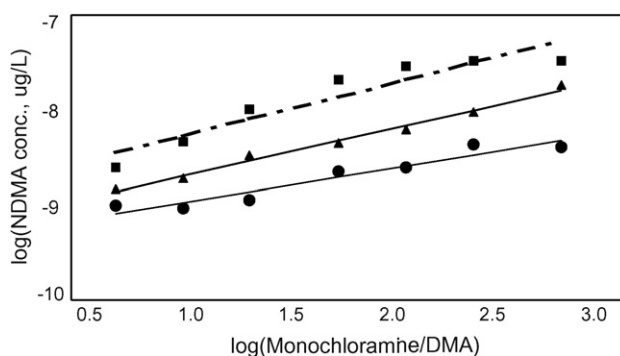

Fig. 2. NDMA formation from monochloramine to DMA ratio at a DMA concentration of 0.01 mM (monochloramine/DMA > 1). Monochloramine = 0.05–5 mM; (●) measured (pH 7); (▲) measured (pH 8); (■) measured (pH 9); each line indicates calculated values from regression equations in Table 3.

NDMA concentration indicates dimensionless in the figures. The formation of NDMA increased with increasing monochloramine concentration. The maximum yields of NDMA from DMA were 2.4% and 7.6% when the ratio is less and greater than 1 at pH 7, respectively. If the ratio becomes greater than 1, the maximum yield of NDMA formation was 28% at pH 8. When the ratio is less than 1, results of the experiment indicated the yield of NDMA ranged from 0.01% to 1.5% depending on monochloramine concentrations at pH 8. A higher NDMA formation was observed when the ratio was greater than 1 at each pH.

When the DMA to monochloramine ratio becomes greater than 1, the amount of monochloramine available to oxidize UDMH is rapidly depleted with increasing DMA due to chlorine transfer. Mitch and Sedlak [5] indicated that higher monochloramine concentrations were associated with higher formation of NDMA and dimethylcyanamide (DMC). At a 5:1 molar ratio of monochloramine to DMA, the yields of NDMA and DMC in terms of chlorine loss were 3.1% and 0.7%, respectively. Both NDMA and DMC are transformed from UDMH oxidation. Yagil and Anbar [8] reported that UDMA formation from reaction between monochloramine and DMA was base-catalyzed above pH 12. It is likely that the UDMH formation may undergo a rate limitation step in the formation of NDMA. The NDMA formation was positively correlated with monochloramine concentrations.

From these findings, when the ratio of monochloramine to DMA was greater than 1, significant NDMA formations were found. Gerecke and Sedlak [3] suggested that the yield of NDMA by chloramination of DMA was approximately 0.6% in natural waters. From their study, DMA concentration was always below 4 nM. However, in this study, higher DMA concentration (0.01 or 0.05 mM) may result in higher yield of NDMA in deionized water.

3.3. Regression analyses I

First, regression equations were developed by molar ratio of monochloramine to DMA (or ratio) in order to predict the NDMA formation from the reaction between DMA and monochloramine under different pH. As shown in Figs. 1 and 2, excellent linear correlations were observed between the molar ratios (monochloramine/DMA) and NDMA formation on a log scale at different pH values. Usually, higher correlated result was found if the ratio was less than 1 at each pH. Linearity of the formation appeared to be related to monochloramine concentrations. Table 3 shows regression equations determined by the molar ratios at pH 7–9. The NDMA formation was also dependent on molar ratio (monochloramine/DMA) and was significantly different when the ratio is less or greater than 1.

The statistical model appears to predict adequately NDMA concentrations unless other NDMA precursors are included. From the previously reported investigations, a positive correlation was found between NDMA formed concentration and monochloramine. Regression equations could provide a potential tool to predict NDMA formation for a simple and quick estimation. Although the model shows a good correlation, there are some limitations in using it due to presence of other NDMA precursors in drinking water or high monochloroamine concentrations applied. The ratios (dimensionless) of monochloramine to DMA should range from −2 to 2.5 on a log scale. Another regression equation could be required if the ratio is greater than 2.5.

3.4. Regression analyses II

Secondly, a correlation study was conducted between the NDMA concentration and a percentage composition of nitrogen in monochloramine from total (monochloramine and DMA). The percentage composition of each compound was calculated from its molecular formula. For example, nitrogen composition in monochloramine was calculated from nitrogen mass fraction multiplied by monochloramine concentration. Table 4 shows regression equations from the percentage composition of nitrogen in monochloramine at different pH values. Because the NDMA formation was associated with the nitrogen fraction, this regression analysis was attempted using the ratio of nitrogen composition in monochloramine to total nitrogen composition. As shown in Figs. 3 and 4, good correlations were observed according to pH values. Interestingly, the correlation coefficients

Table 3
A proposed regression equation for the NDMA formation at different pH and monochloramine to DMA ratio

pH	D DMA concentration (mM)	Regression equation	r^{2a}	Comment
7	0.05	$Y = 1.71x − 7.7$	0.99	Monochloramine[b]/DMA < 1
7	0.05	$Y = 0.89x − 8.3$	0.93	Monochloramine[c]/DMA > 1
7	0.01	$Y = 0.38x − 9.4$	0.92	Monochloramine[c]/DMA > 1
8	0.05	$Y = 1.11x − 7.7$	0.99	Monochloramine[b]/DMA < 1
8	0.05	$Y = 1.13x − 8.0$	0.97	Monochloramine[c]/DMA > 1
8	0.01	$Y = 0.53x − 9.2$	0.99	Monochloramine[c]/DMA > 1
9	0.05	$Y = 1.36x − 8.4$	0.90	Monochloramine[b]/DMA < 1
9	0.05	$Y = 0.21x − 7.9$	0.95	Monochloramine[c]/DMA > 1
9	0.01	$Y = 0.59x − 8.9$	0.89	Monochloramine[c]/DMA > 1

x and Y are log(molar ratio of monochloramine to DMA) and log(NDMA concentration), respectively. A statistical significance ($p < 0.05$) was utilized for all regression equations.

[a] Correlation coefficient.

[b] Monochloramine ranged from 0.001 to 0.1 mM.

[c] Monochloramine ranged from 0.05 to 5 mM.

Table 4
A proposed regression equation for the NDMA formation at different pH and nitrogen composition ratio in monochloramine

pH	DMA concentration (mM)	Monochloramine concentration range (mM)	Regression equation	r^{2a}	Monochlora-mine/DMA
7	0.05	0.001–0.1	$Y = 2.2x - 7.0$	0.98	<1
7	0.05	0.05–5	$Y = 5.7x - 6.9$	0.91	>1
7	0.01	0.05–5	$Y = 7.9x - 8.5$	0.64	>1
8	0.05	0.001–0.1	$Y = 1.4x - 7.3$	0.98	<1
8	0.05	0.05–5	$Y = 5.5x - 6.6$	0.84	>1
8	0.01	0.05–5	$Y = 11x - 8.1$	0.93	>1
9	0.05	0.001–0.1	$Y = 1.8x - 7.8$	0.95	<1
9	0.05	0.05–5	$Y = 1.3x - 7.6$	0.85	>1
9	0.01	0.05–5	$Y = 15x - 7.5$	0.93	>1

Nitrogen mass fractions in monochloramine and DMA are 0.27 and 0.31, respectively. x and Y are log(ratio of nitrogen composition in monochloramine to total nitrogen composition) and log(NDMA concentration), respectively. A statistical significance ($p < 0.05$) was utilized for all regression equations.
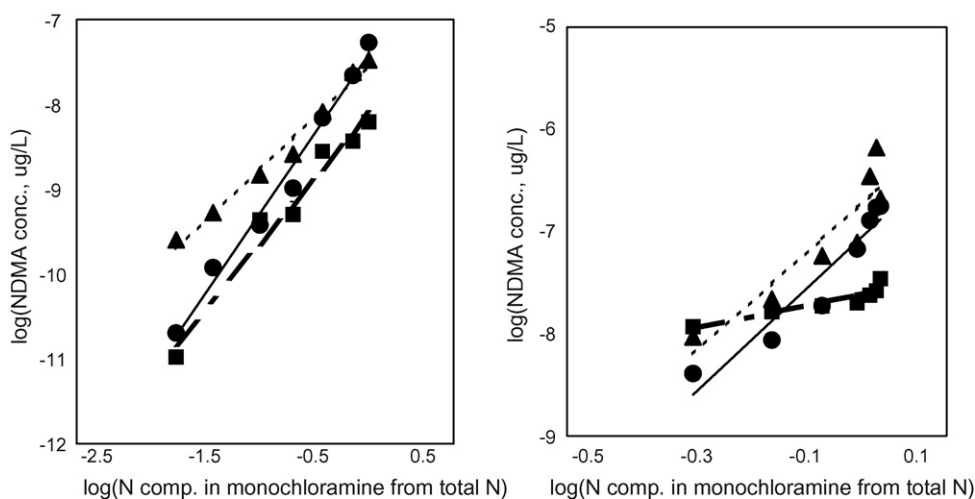^a Correlation coefficient.

Fig. 3. NDMA formation from the ratio of nitrogen composition in monochloramine to total nitrogen composition at DMA = 0.05 mM for the monochloramine/DMA < 1 (left), and the monochloramine/DMA > 1(right). (●) Measured (pH 7); (▲) measured (pH 8); (■) measured (pH 9); each line indicates calculated values from regression equations in Table 4.
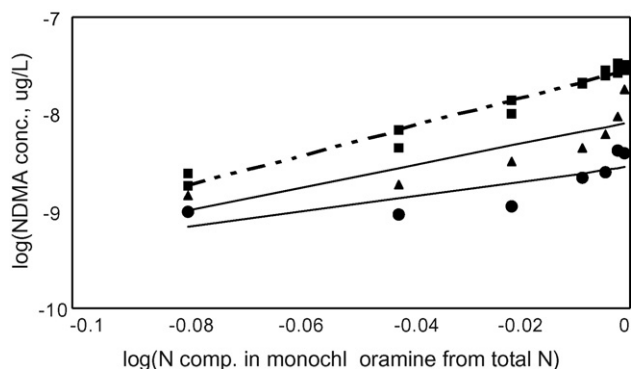

Fig. 4. NDMA formation from the ratio of nitrogen composition in monochloramine to total nitrogen composition at DMA = 0.01 mM for the monochloramine/DMA > 1. (●) Measured (pH 7); (▲) measured (pH 8); (■) measured (pH 9); each line indicates calculated values from regression equations in Table 4.

by the percentage of nitrogen composition were slightly smaller than those by the molar ratio. If the ratio was greater than 1, most data were scattered at higher nitrogen composition.

The second regression analyses were attempted to investigate the hypothesis that the nitrogen atom in NDMA could be more related to nitrogen of monochloramine. Choi and Valentine [15] had already studied that a source of one of the nitrogen atoms in the nitroso group in NDMA was from monochloramine. They developed a kinetic model in order to investigate NDMA formation during water chloramination. They proposed that NDMA formation is initiated by the formation of UDMH from the reaction of DMA with monochloramine, followed by the oxidation of UDMH by monochloramine to NDMA. They also found that the maximum NDMA occurred when the ratio of DMA to monochloramine was approximately 1.0. Because it was the first approach to estimate NDMA formation by nitrogen composition, correlation coefficients and data scattering seemed to be an important factor. If the ratio was greater than 1, most data were scattered at higher nitrogen composition. Therefore, regression equations by the molar ratio of monochlo-

Table 5
Comparison of measured and calculated NDMA concentrations

DMA (mM)	Mono-chloramine (mM)	Monochloramine/ DMA	NDMA formation (μg L^{-1}) from Choi[a]	NDMA calculated concentration[b] (μg L^{-1})	Relative error (%)
0.1	0.1	1	12	21	74
0.1	0.2	2	33	68	109
0.1	0.05	0.5	3.9	6	52
0.5	0.1	0.2	4.7	1	−79
0.2	0.1	0.5	9	6	−34
0.08	0.1	1.25	12	31	163

[a] J. Choi, mechanistic studies of *N*-nitrosodimethylamine (NDMA) in model drinking waters, Ph.D. thesis, The University of Iowa, USA, 2002.

[b] From Table 3, $Y = 1.71x - 7.7$ was used at pH 7.

ramine to DMA could be more preferable than those by analysis II.

3.5. Application study

A developed regression equation was applied to results of Choi's study [16]. The used equation for the calculated NDMA concentration was determined at pH 7 and the molar ratio <1 in order to adjust the same conditions. Fig. 5 shows the comparison of calculated NDMA values with the Choi's data. A linear relation is shown in his data. Dimensionless ratio of monochloramine to DMA of his data ranged from −1 to 0.1 on a log scale. Because only his investigation was available, unfortunately, studies on the model application were limited.

As summarized in Table 5, relative errors ranged from −79% to 163%, when the regression equation was used. Although the high relative errors may show a limitation in predicting accurately the NDMA concentrations, the equation can be used for an approximate prediction of NDMA concentration in natural water only. Usually it was not possible to calculate exact NDMA concentrations by any prediction models. Even Choi's kinetic model [12] could not show exact prediction data. The NDMA formation was dependent on molar ratio (monochloramine/DMA) and was different if the ratio was less or greater than 1. Using the predictive tool, a simple and approximate calculation for the NDMA formation can be possible in drinking water systems.

Regression equations developed could provide a potential tool to predict NDMA formation for a pre-alarm system by quick estimation.

4. Conclusion

The objectives of this study were to determine NDMA formation during chloramination of DMA and to develop a regression equation for the prediction of NDMA formation at different pH. A statistical approach was conducted to provide a better understanding of predicted reaction for the NDMA formation. This investigation showed that regression equations developed could provide a potential tool to predict NDMA formation for a pre-alarm system. However, the use of the regression equations will be limited in real water treatment facilities, where usually detect other NDMA precursors or low DMA concentrations.
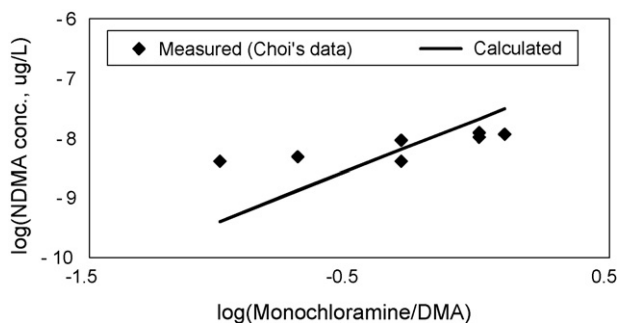

Fig. 5. Prediction of NDMA concentration from the regression equation at pH 7 (application). The Choi's data were obtained from mechanistic studies of *N*-nitrosodimethylamine (NDMA) in model drinking waters, Ph.D. thesis, The University of Iowa, USA, 2002. The calculated data were determined from Table 3, $Y = 1.71x - 7.7$ was used.

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