

Effects of mixing on chloramination process

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ABSTRACT

According to a recent study more than 90 percent of water treatment plants utilizing chloramination for distribution system residuals indicate a certain level of dissatisfaction toward the process performance. One factor that may lead to such dissatisfaction is the inadequacy of mixing when ammonia is added to chlorinated water. If mixing is not instantaneous and uniform, the actual chlorine to ammonia nitrogen molar ratio will become variable at a micro-level, even though the overall ratio at the macro-level is close to the desired 1:1 ratio. Because of the non-uniform mixing, certain portions of the mixture might have a molar ratio exceeding the stoichiometric ratio of 1:1. In such instances, certain unintended reactions (e.g. breakpoint type of chlorine chemistry) can occur. This will lead to the resultant monochloramine concentration being significantly less than the stoichiometric concentration, based upon the calculation using the overall molar ratio. Other factors, such as pH variation in the micro environment, could also affect the final chemical composition of the chloramination process. In this study, the effect of mixing was studied by conducting breakpoint chlorination experiments under different levels of mixing, represented by the average velocity gradient, G in s^{-1} . A unique way of plotting breakpoint chlorination curve was utilized to analyze the data, which allowed a clear delineation if the monochloramine formation was according to the stoichiometry. A quantitative comparison between experimental data and stoichiometry can clearly indicate the impact of non-uniform mixing. The experimental data showed that as the G value increased from 35 to 500 s^{-1} , the monochloramine formation increased from 75 to 87 percent of the stoichiometric value. The location of the breakpoint, correspondingly, increased from a molar ratio of 1.25 to 1.75. Comparison of 40 s^{-1} (50 rpm) and 300 s^{-1} (200 rpm) experimental data was conducted and a breakpoint curve was plotted imposing one over the other. It has been observed from previous literature that in ideal conditions, breakpoint occurs at chlorine to ammonia nitrogen molar ratio of 1.5:1, and the peak of monochloramine is expected at a molar ratio of 1:1. Hence, breakpoint curve was plotted at mixing speed of 50 and 200 rpm, indicating free chlorine, monochloramine, dichloramine, trichloramine, and total chlorine concentration at contact time of 45 minutes. Few studies were found in literature on mixing effects in chloramination. Data from a previous study was re-analyzed and compared with the current study, and a similar trend was observed. In another case study, the design G value for a modern water treatment plant in metropolitan Boston was found to be 800 s^{-1} , which was higher than the maximum G value used in this study (500 s^{-1}), and is likely to be more than sufficient. In conclusion, when chlorine and ammonia are combined to produce monochloramine, the degree of mixing indeed has significant impact on the performance of the chloramination process, and therefore must be a critical consideration in its design and operation.

Key words | chloramination, DBPs, mixing, monochloramine, velocity gradient

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INTRODUCTION

When disinfection by-products (DBPs) are a concern or the free chlorine residual is difficult to maintain, water treatment plants use chloramines to provide stable disinfectant residuals in distribution systems. Chloramine (NH_2Cl , NHCl_2 , and NCl_3) is formed by mixing free chlorine with ammonia in treated water prior to discharge into the distribution system. Recently, there has been growing concern about the biological instability in the drinking water distribution system for treatment plant utilizing chloramines. Researchers have shown that excess ammonia released in the distribution system from incomplete mixing or chloramine decay can not only promote the growth of ammonia-oxidizing bacteria (AOB), but also contribute to an increase in organic material. The presence of AOB can lead to the production of nitrite and nitrate and increased organic material exerts an additional chloramine demand. Failure to achieve good mixing in chloramination could lead to treatment utilities' incompliance with the Surface Water Treatment Rule and the Total Coliform Rule (Liu & Ducoste 2006).

Another study was performed using controlled bench-scale mixing experiments to investigate the impact of mixing at the point of ammonia and chlorine addition on DBP formation (Symons *et al.* 1998). Three different mixing intensities, G value of 60 sec^{-1} , 500 sec^{-1} and $1,000 \text{ sec}^{-1}$, were studied to span the spectrum of practical approach. From their experiments, they indicated that mixing conditions did not significantly affect the disinfectant speciation at the contact time characteristic of distribution systems and for contact time characteristic of rapid mix basins, mixing might have a substantial effect on speciation.

Yamamoto *et al.* (1990) studied the effects of different mixing speeds ranging from 12 to 240 rpm on residual chlorine during chlorination of seawater containing ammonia nitrogen. It was reported that mixing affect the concentration and proportion of residual chlorine during chlorination of seawater containing ammonia nitrogen. They found that at lower speed the peak of the breakpoint curve occurred at lower chlorine dosage as compared to the higher speed. Also, at high mixing speed, the increase in the residual chlorine level was directly proportional to the dose of chlorine. At low mixing speed, the residual

chlorine level began to decline before the peak of the breakpoint curve, which was flatter than the one obtained with high mixing speed. Similar trend was seen in the experiments conducted in this study.

The design report of a recent major water treatment plant put into operation since 2005 was studied to compare with the results obtained from this study (CDM 2002). Chloramination treatment unit design was the main area of focus. Post ammoniation is used as a part of their treatment process and sodium hypochlorite is the source of chlorine. Sodium hypochlorite with a concentration of 2 to 3 mg/L and aqua ammonia with a concentration of 0.5 to 0.75 mg/L are injected just before the dry feed mixing chamber (rapid mixing) to get adequate mixing. There are four rapid mix chambers each having a velocity gradient of 800 s^{-1} , which corresponds to 56 rpm. Plant flow in this chamber is 270 mgd on average and 405 mgd maximum with the retention time of 9.5 sec at 270 mgd flow. Following this chamber, there is a dry feed reaction chamber with baffles installed.

It is clear from these studies that adequate degree of mixing is important to maintain the chlorine residual in the process of chloramination. In this study, an *optimum value of G* for appropriate mixing in the process of chloramination under pre-set conditions is evaluated. Also, the best approach to utilize the results of this study in the real world, full-scale plant application is studied.

CHLORAMINATION OPERATING PARAMETERS

From a survey done by the American Water Works Association (AWWA) and AWWA Research Foundation (AwwaRF), the most important parameters to consider in designing, operating, and in optimizing a chloramination process were studied and the key parameters are listed below (Kirmeyer *et al.* 2004).

Chemical application points

This includes considering where chemicals are added in the treatment process, the order in which they are injected,

and how chemical feed rates are controlled. For flexibility, multiple ammonia and chlorine injection points may be provided. Only one ammonia injection point is normally used at any one time.

pH and temperature

The optimum pH range generally is 8.0 to 9.0 and the ideal temperature is 10 to 15°C.

Chloramine residual

The desirable chloramines residual depends on the water source and conditions within the distribution system. A range of chloramine residuals from 0.5 to 6.8 mg/L has been noted in this survey. Typical finished water chloramines levels are 2 to 3 mg/L in finished water. U.S. Environmental Protection Agency (EPA) regulations require a detectable disinfectant residual or HPC (heterotrophic plate count) less than 500 cfu/mL at monitoring points in the distribution system and limit the chloramines residual to a maximum of 4.0 mg/L.

Chlorine: ammonia-nitrogen ratio

The $\text{Cl}_2:\text{NH}_3\text{-N}$ weight ratio should generally be maintained between 4:1 and 5:1 at the point of application to enhance formation of monochloramine and minimize the excess of free ammonia. Further, to reduce the potential for nitrification, a free ammonia target of 0.10 mg/L as N or less (e.g. 0.05 mg/L as N) should be maintained in the distribution system.

Mixing and reaction time

It is best to provide for immediate and thorough mixing of treatment chemicals within the process stream, a range of velocity gradient of 300 to 1,000 s^{-1} is suggested. Mixing in the pipe is most common, but mechanical or static mixing may be employed.

Periodic switch to free chlorine

It may be advantageous to switch to free chlorine periodically for short periods of time to rid the system of

chloramine-resistant bacteria and to reduce the potential of nitrification. This is a significant undertaking and may not be feasible in large distribution systems.

MIXING

Intensity of mixing is an important design parameter in most of the treatment units. Ideal mixing is considered as one of the factors for the formation of chloramines species and proper breakpoint chlorination in chloramination process. Intensity of mixing is usually measured in terms of velocity gradient (G value). From previous studies and surveys it has been observed and reported that intensity of mixing affects the process of chloramination and is an important parameter (Yamamoto *et al.* 1990). Literature which gives an optimum value of velocity gradient for appropriate formation of chloramine species in the chloramination process is not available. However a Japanese group studied the mixing effect using seawater (Yamamoto *et al.* 1990).

VELOCITY GRADIENT

Steady state turbulence generation requires continuous power input to the system. The relationship between power dissipation and velocity gradient as elucidated by Camp & Stein (1943) has been widely adopted in practice. T. R. Camp (1955) developed a rational approach to design and evaluate mixing chambers. According to Camp, the intensity of mixing required in the rapid mix is generally measured by velocity gradient (G). The equation for velocity gradient with no vertical baffles is

$$G = \sqrt{\frac{W}{\mu}} = \sqrt{\frac{P}{\mu V}}$$

where, G = velocity gradient, fps/ft, meter/sec/meter or sec^{-1} ; W = power imparted to the water per unit volume of the chamber, ft-lb/sec-ft³, kg/sec-m²; P = power imparted to the water, ft-lb/sec, watt; V = chamber volume, ft³, m³; μ = Absolute viscosity of the water, lb-sec/ft², kg/s-m.

The value of total power imparted to the liquid by various impellers in a baffled tank is determined by using

relationships developed by Rushton, given by the following equation:

$$P = \phi \rho n^3 D_i^5$$

where, P = power imparted to the water, ft-lb/sec, watt; ϕ = impeller constant for turbulent flow also known as power number; n = rotational speed, rps; D_i = impeller diameter, ft, m; ρ = density of liquid, $\rho = \gamma/g_c$; γ = specific weight of the liquid, lb/ft³, kg/m³; g_c = acceleration due to gravity, 32.17 ft/sec², m/s².

The power number (ϕ) is a function of mixing chamber geometry, impeller geometry and the Reynolds number (R) where

$$R = \frac{D_i^2 n \rho}{\mu}$$

Calculating the power number for impellers is a complex process. The manufacturers of the impellers usually provide these numbers.

The rate of particulate collisions is proportional to the velocity gradient, G ; therefore, the gradient must be sufficient to furnish the desired rate of particulate collisions (Reynolds & Richards 1996). In the rapid mixing process for monochloramine formation, it is critical that both chlorine and ammonia interact with each other as uniformly as possible, therefore the level of G must be adequate and serves as the key operational parameter for optimal chloramination. Monochloramine formation is instantaneous and rapid mixing is used. Unlike flocculation, mixing intensity and time (Gt) is not relevant for monochloramine formation.

EXPERIMENTAL DESIGN

The controlled bench-scale experiments primarily investigated the impact of different degrees of mixing on the chloramination process where chlorine solution is dosed with a specified delay in dosing ammonia, which is also termed as post-ammoniation. Also, there are some other important variables which need to be considered for optimization of the chloramination process. These variables include ammonia and chlorine application points, pH,

temperature, chlorine to ammonia-nitrogen ratio, mixing, and reaction time for chloramines formation. All these variables were taken into consideration while designing the experiment.

Experiments were conducted in a jar test apparatus at various known values of G (mean velocity gradient). The main experimental variables were the mixing speed (in rpm) and chlorine to ammonia nitrogen molar ratio.

Temperature was set to 10°C, considering the applications at the utilities. pH was adjusted to a range of 7 to 9 in accordance with the chloramines chemistry. The molar ratios of chlorine to ammonia nitrogen were selected based on the breakpoint pattern. Mixing intensities were selected as per the jar test device and further adjusted looking at the experimental data available. Reaction time of 15 minutes and 45 minutes were also selected, based on the chloramines chemistry.

The experiments were conducted in 2-liter reactors (B-KER² from PHIPPS & BIRD). Impeller speed was fixed. The calibration curve of Laboratory G curve for flat paddle in the gator jar (Manual of Water Supply Practices 1991) was used to evaluate G as a function of impeller speed. Chlorine stock solutions were delivered to the reactors. Sodium bicarbonate was added to adjust the pH. The solution was then mixed for 1 minute at the fixed impeller speed. Ammonia stock solution was then added in the reactors and then samples were mixed continuously for 15 minutes after the completion of dosing at 10°C.

A standard jar test apparatus allows six reactors to be run simultaneously, with the same mixing intensity in each sample (see Figure 1). One sample was used as a control and was dosed only with chlorine stock solution. Chlorine to ammonia nitrogen molar ratio of 0.5, 0.75, 1.0, 1.25, 1.5, 1.75, 2.0 and 4.0 was used for the experiments. Each of the molar ratios was studied for a reaction time of 15 minutes and 45 minutes. Three different mixing intensities with impeller speeds of 50 rpm, 150 rpm and 200 rpm were studied to span the spectrum of practical applications.

BREAKPOINT CHLORINATION

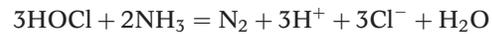
When chlorine is added to waters containing ammonia, the breakpoint phenomenon becomes significant in the pH



Figure 1 | Jar test set up.

range of 6 to 9. The breakpoint is that dosage of chlorine that produces the first detectable amount of free chlorine residual. Chloramine formation and oxidation of ammonia by chlorine combine to create a unique curve for the addition of chlorine to ammonia-containing solutions (Figure 2). At a fixed ammonia concentration, as the chlorine dose increases, the chlorine residual at first increases to a maximum at a $\text{Cl}_2:\text{NH}_3\text{-N}$ molar ratio of 1:1. As the chlorine dose is increased further, the chlorine residual falls to a value close to zero. The chlorine dose

corresponding to this minimum is called the breakpoint, and it occurs at $\text{Cl}_2:\text{NH}_3\text{-N}$ molar ratio of 1.5, under common solution conditions. The primary overall reaction that causes the residual chlorine concentration to decrease and thus to form a breakpoint is the breakpoint reaction, which can be represented as:



During breakpoint chlorination, the amount and type of chlorine residual changes according to Figure 2. The nature of the chlorine residual present prior to the breakpoint reveals that it is composed almost entirely of chloramines. These are referred to as “combined chlorine residuals” as opposed to Cl_2 , OCl^- and HOCl , which are known as “free chlorine residuals.” As the chlorine to ammonia nitrogen ratio increases, the ammonia molecule becomes progressively more chlorinated, i.e. NH_3 (ammonia) to NH_2Cl to NHCl_2 to NCl_3 . Prior to the breakpoint, primarily monochloramine and some dichloramine are produced. Following the breakpoint, free chlorine (HOCl and OCl^-) predominate.

As the dose of chlorine is increased, the residual reaches a maximum at a molar ratio of 1:1 and then starts

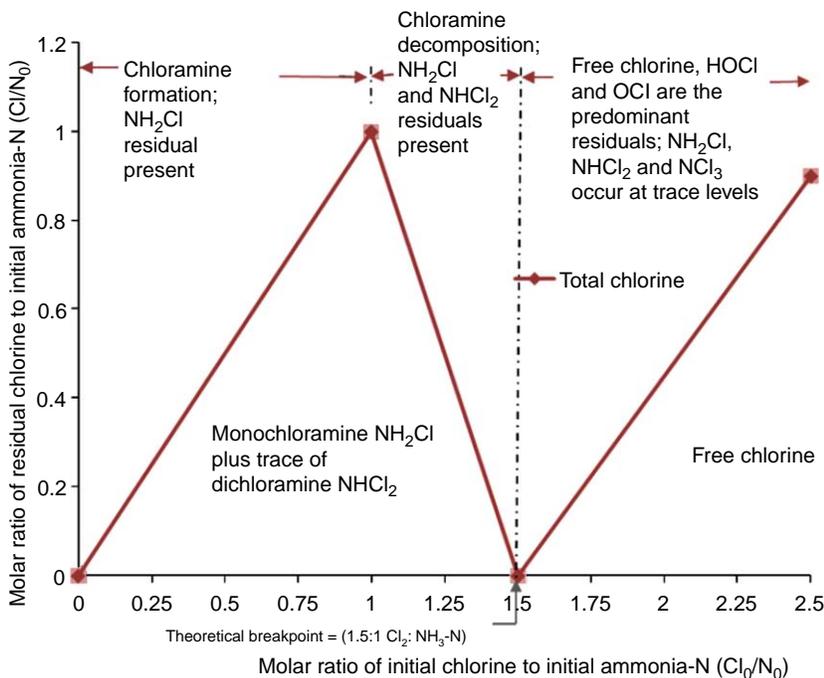


Figure 2 | Theoretical breakpoint curve.

decreasing. At a $\text{Cl}_2/\text{NH}_3\text{-N}$ molar ratio of 1.5:1, all of the available chlorine residuals are greatly reduced or eliminated. The residual would drop almost to zero as the molar ratio approached 1.5 to 2.0 (breakpoint), depending on the conditions of pH and temperature. After the breakpoint, any further increase in chlorine dose will increase the residual in the form of free chlorine. As such, any utility considering chloramination should target the $\text{Cl}_2:\text{NH}_3\text{-N}$ molar ratio at which the most monochloramine occurs (theoretically 1:1).

In the reaction mechanism, each step has its own rate of reaction that depends on the pH, temperature, and the initial concentrations of reactants. These conditions will subsequently affect the rate of the overall breakpoint reaction as well as the products formed. Lower pH and temperature favors the formation of NCl_3 , as does higher initial dose molar ratio. This aspect is important in the determination and control of undesirable end products. The rates of the formation and subsequent decay of dichloramine determine the speed of the overall breakpoint reaction. Water pH can significantly affect the kinetics of breakpoint chlorination; the optimal pH range is 7–8. In the previous studies, it was found that the ratio of chlorine to ammonia needed for the reaction decreases with decreasing temperature and with increasing initial ammonia concentration (Kirmeyer *et al.* 2004).

RESULTS AND DISCUSSIONS

An effective way of plotting the breakpoint curve is shown in Figure 2, marking Y axis ordinate as the molar ratio of residual chlorine to initial ammonia nitrogen concentration ($\text{Cl}_2/\text{NH}_3\text{-N}_0$) and the X axis ordinate as the molar ratio of initial chlorine to initial ammonia nitrogen ($\text{Cl}_0/\text{NH}_3\text{-N}_0$). Breakpoint curve presented in this fashion was clearer and allowed a quantitative elucidation of the effect of mixing on chloramination process, which was difficult to achieve by using the traditional plotting. This representation of breakpoint curve did not need any specification of the initial ammonia nitrogen concentration for each plot as both the axes have the concentration of initial ammonia nitrogen as the common denominator. As we do not expect the monochloramine peak to go beyond 1.0, the amount of monochloramine formed was clearly represented in this type of plotting. Hence, the maximum value represented on the Y axis is 1.0. This type of plotting was used for all breakpoint curves in this study.

Effect of reaction time

From the breakpoint curve plotted at different reaction time, it was observed that at 15 minutes the curve's plotting of the chloramines species were not as stable as those at 45 minutes (Figure 3). Free chlorine,

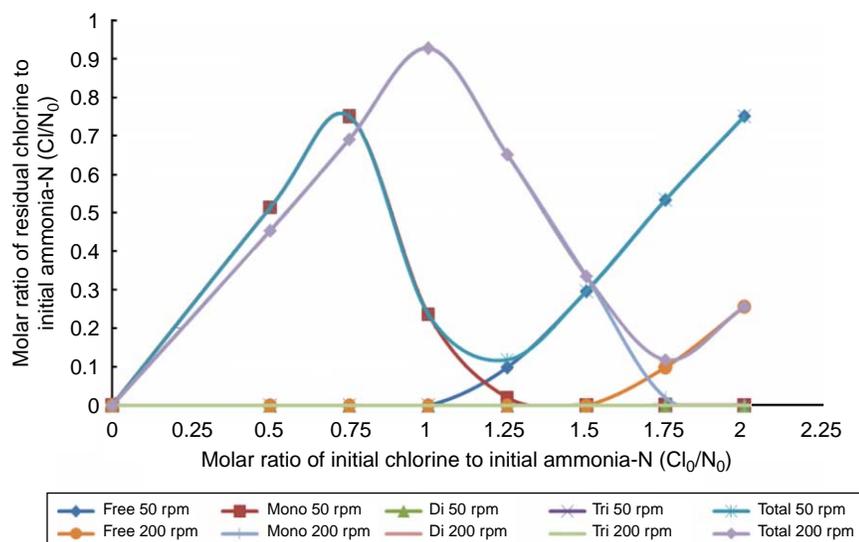


Figure 3 | Cl_0/N_0 vs. Cl_0/N_0 @ 50 & 200 rpm at 10°C after 45 minutes.

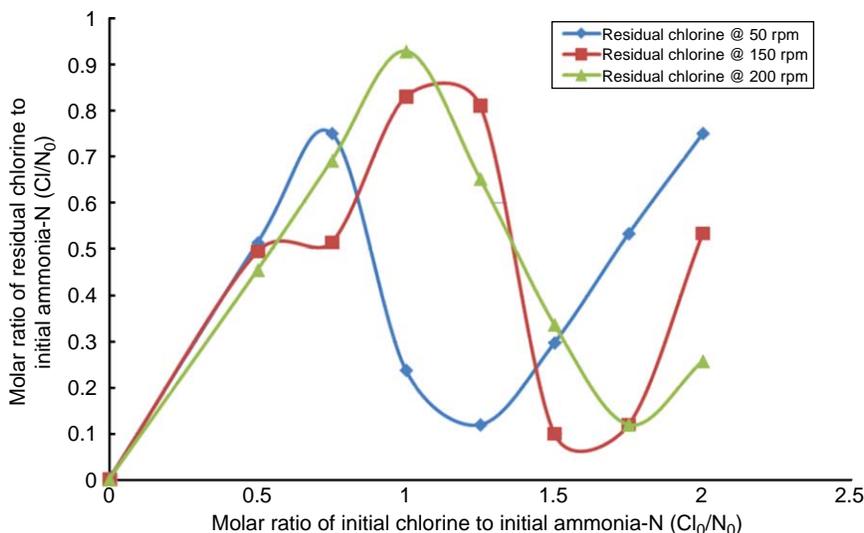


Figure 4 | Residual chlorine @ 50, 150 and 200 rpm at 10°C after 45 minutes.

monochloramine, dichloramine, trichloramine, and total chlorine were not as well defined as an ideal breakpoint curve for the reaction time of 15 minutes as compared to 45 minutes, because reactions had not reached the equilibrium at 15 minutes.

Monochloramine plays a key role in the chloramination process, hence, its pattern was observed closely. Dichloramine and trichloramine were found in negligible concentrations at a reaction time of 15 minutes as well as at 45 minutes. After breakpoint, total chlorine concentration consisted of only free chlorine.

Effect of mixing

The scope of this study focuses on the effect of mixing in the chloramination process. Figure 4 show the breakpoint curve at various degree of mixing, i.e. 50, 150, and 200 rpm at 10°C. Regardless of the stirring speed, monochloramine was the predominant species in the residual

chlorine that remained after 45 minutes, and there were no detectable formations of di- and trichloramine.

From Table 1 it can be observed that at lower rpm, monochloramine peak is lower than at higher rpm. This shows that the amount of monochloramine formed is lower at 50 rpm than at 150 or 200 rpm, especially at 45 minutes, when equilibrium is reached.

Comparison of 50 rpm and 200 rpm

Comparison of 50 and 200 rpm experimental data was done and a breakpoint curve was plotted by imposing one over the other. It has been observed from previous literature that in ideal conditions, breakpoint occurs near the chlorine to ammonia nitrogen molar ratio of 1.5:1, and the peak of monochloramine is expected at a molar ratio of 1:1. Hence, breakpoint curves were plotted at mixing speeds of 50 and 200 rpm, indicating free chlorine, monochloramine, dichloramine, trichloramine, and total chlorine concentrations at contact time of 45 minutes in Figure 3 to

Table 1 | Monochloramine peak and breakpoint at different G values (reaction time = 45 minutes)

Reaction time (minutes)	rpm	G value (sec ⁻¹)	Monochloramine peak (Cl/N ₀)	Cl ₀ /N ₀ of mono peak	Breakpoint Cl/N ₀	Cl ₀ /N ₀ of breakpoint
45	50	~40	~0.75	0.75	~0.11	1.25
45	150	200	~0.83	1.0	~0.1	~1.6
45	200	300	0.92	1.0	0.12	~1.75

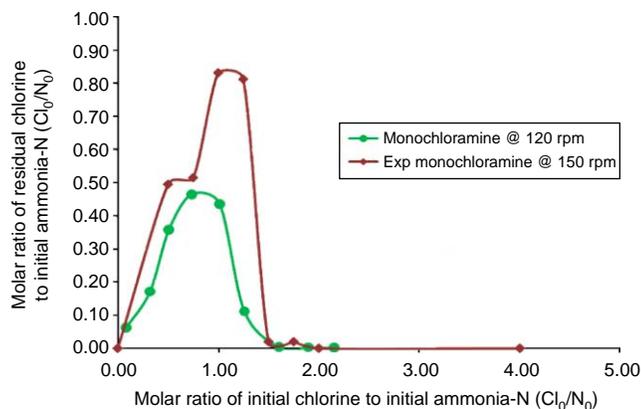


Figure 5 | Monochloramine @ 120 rpm at 20–25°C (Yamamoto *et al.* 1990) & 150 rpm (this study) at 10°C.

illustrate the effect of mixing on the breakpoint curve. Ammonia nitrogen concentration is 1 mg/L.

At 50 rpm, peak of monochloramine is located to the left of chlorine to ammonia molar ratio of 1.0 vs. 200 rpm. Also, the breakpoint is located near a molar ratio of 1.25 which is to the left of molar ratio 1.5.

At 200 rpm, monochloramine peak is observed at a molar ratio of 1:1 and breakpoint occurs at a molar ratio of 1.75:1 for a reaction time of 45 minutes, as expected theoretically at equilibrium. While at 50 rpm, the breakpoint curve is shifted to the left (see Figure 3). Monochloramine peak is seen at a molar ratio of 0.75:1 and breakpoint occurs at a molar ratio of 1.25:1. When the breakpoint graphs at 120 and 240 rpm (Figures 5 and 6) were compared in the study of the effects of different stirring speeds on residual chlorine during chlorination of seawater containing ammonia nitrogen (Yamamoto *et al.* 1990), similar trends were observed.

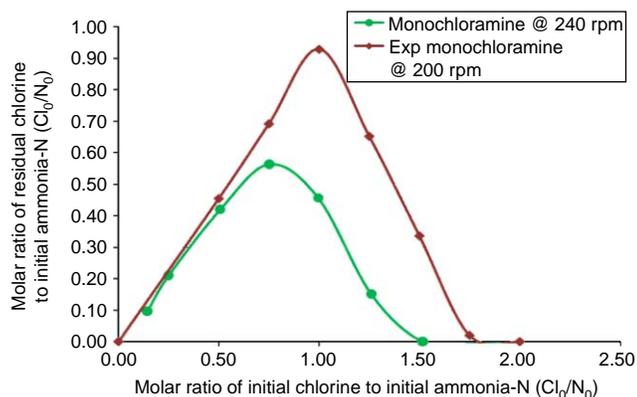


Figure 6 | Monochloramine @ 240 rpm at 20–25°C (Yamamoto *et al.* 1990) & 200 rpm (this study) at 10°C.

Comparison with other study

A comprehensive study was done of the graphs obtained from the Japanese study (Yamamoto *et al.* 1990), and a comparison was done with the results obtained from this study. Data from the Japanese study were extracted by using the *UnGraph software 2006*. A comparison was done between 120 and 150 rpm (Figure 5). It is to be noted that these rpms refer to different mixing devices used for respective studies and might not imply quantitative comparison of mixing intensity to this study.

All the graphs for both monochloramine and residual chlorine followed the same trend at different mixing speeds, except that at 120 and 240 rpm, residual chlorine loss was higher via the disproportionation of monobromamine to form dibromamine and ammonia nitrogen (Yamamoto *et al.* 1990), as their subject of study was seawater, containing other impurities e.g. bromide.

The velocity gradient (G value) range of 300 s^{-1} to $1,000\text{ s}^{-1}$ was suggested for proper mixing in the chloramination process (Kirmeyer *et al.* 2004). In this study, 200 rpm corresponds to a G value of 300 s^{-1} and 50 rpm corresponds to a G value of 35 s^{-1} at 10°C from the calibration curve of Laboratory G curve for flat paddle in the gator jar (Manual of Water Supply Practices 1991). G value for 12, 60, 120, and 240 rpm from the Japanese study (Yamamoto *et al.* 1990) is not mentioned in the article and it cannot be determined.

CONCLUSIONS

From Table 1 it can be concluded that mixing affected the concentration and proportions of residual chlorine during chloramination. At lower mixing speed, the residual chlorine level began to decline before the peak of the breakpoint curve, which was flatter than the one obtained from the higher mixing speed and the breakpoint shifts to the left of the higher mixing speed, e.g. Cl_0/N_0 of 1.25 vs. 1.75 in Figure 3. At higher mixing speed, the residual chlorine level rose in direct proportion to the dose of chlorine, and appears to approach Cl_0/N_0 of 1.0, as expected theoretically. The degree of mixing of chlorine and ammonia in the chloramination process is of importance in evaluating the residual chlorine concentrations.

For bench-scale mixing experiments, a G value of 300 s^{-1} (corresponds to 200 rpm) from the calibration curve of Laboratory G curve for flat paddle in the gator jar (Manual of Water Supply Practices 1991) is considered as a minimum mixing velocity gradient for complete mixing of ammonia and chlorine to form desirable level of monochloramine concentration. When used in real practices, scale-up is a factor which needs to be considered.

PRACTICAL APPLICATIONS

Given that small-scale mixing experiments were conducted in laboratory-sized reactors, it is appropriate to consider how these results relate to larger scale mixing occurring in practice. Unfortunately, it has been observed from the literature review that rapid mixing has not been well studied in the process of chloramination. G on the large scale should be related to the small-scale G to achieve the same degree of mixing, depending on the selected scale-up relationship. For this particular application, relationships that show G on the larger scale are equal to or greater than the small-scale G , and are probably most appropriate because chloramine-formation kinetics are relatively fast. Scale up is a factor which needs to be considered when being used in the real world applications.

The design report of an online water treatment plant was studied to compare the results obtained from this study. Chloramination treatment unit design was the main area of focus in the water treatment plant design report. Post ammoniation is used as a part of their treatment process and sodium hypochlorite is the source of chlorine. Sodium hypochlorite with a concentration of 2 to 3 mg/L and aqua ammonia with a concentration of 0.5 to 0.75 mg/L are injected just before the mixing chamber to get adequate mixing. This chamber has the rapid mixing. There are four rapid mix chambers, each providing a velocity gradient of 800 s^{-1} . Plant flow in this chamber is 270 mgd on average and 405 mgd maximum with the retention time of 9.5 sec at

270 mgd. Following this mixing chamber, there is a reaction chamber with baffles installed.

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