

Reduction of Natural Organic Matter and Disinfection By-Product Precursors by Coagulation and Adsorption

E.-E. Chang¹; Pen-Chi Chiang²; Su-Huei Chao³; and Chung-Huei Liang⁴

Abstract: Source water from King-Men Reservoir water in Taiwan was treated by alum and polyaluminum chloride coagulation, and the corresponding natural organic matter was fractionated by ultrafiltration membranes for determining its molecular weight (MW) distribution to provide the baseline information for further investigation. Due to the characteristics of the source-water nature, a lower alum dosage could reduce the total organic carbon (TOC) concentration by a great amount (25%), while the difference in TOC removal efficiency was found to be insignificant at a higher dosage (60–90 mg/L) and even operating in a lower pH range. The results show that the larger MW coagulant polyaluminum chloride, having higher-charge neutralization and bridging capacities, exhibits better performance than the lower MW alum. In addition, the granular-activated carbon-adsorption process provides a relatively lower THMFP in the treated water at the same level of trihalomethanes and to lower the coagulant doses demand as well.

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Introduction

Natural organic matter (NOM) is a term used to describe the complex matrix of organic material present in natural waters, and A relationship exists between its characteristics and treatabilities (Owen et al. 1995). Molecular weight (MW) distribution of NOM is one of the important parameters in characterizing NOM (Amy et al. 1992; Newcombe et al. 1997). In water-treatment processes, each unit process may exhibit different removal efficiencies for a specific MW fraction of NOM (Jacangelo et al. 1995). For example, some investigators have suggested that coagulation is suitable to treat a higher MW fraction of NOM.

In addition, ultraviolet (UV) absorption, a useful surrogate measurement of selected organic constituents in fresh water, may exhibit strong correlation with organic-carbon content, color, and precursors of disinfection by-product (DBP). (Najm 1994). Furthermore, our previous studies found that the parameters including the nature of NOM, chlorine dosage (or demand), UV absorbance, and total organic carbon (TOC) should be used to estimate the DBP formation potential (DBFP) (Chang et al. 2001a,b) and found that there was no reduction of UV₂₅₄ dissolved organic carbon (DOC) in the coagulation, filtration, and granular-activated carbon (GAC) for the preozonation processes; in fact

the UV₂₅₄/DOC increases after the GAC process. This evidence strongly suggests that both pre and postozonation can effectively lower the UV₂₅₄/DOC values, thus reducing the DBFP through converting precursors to nonprecursors (Chiang et al. 2002). TOC is a more convenient and direct expression of TOC and can be used to evaluate the effects of DBP control strategies. Singer (1994) suggested that chlorine consumption was a reasonably good indicator of DBP formation. Edzwald et al. (1985) and Najm et al. (2000) reported that the DOC rather than TOC is a more appropriate indicator of DBFP in water-treatment plants.

The reduction of NOM by various methods including coagulation, oxidation, adsorption, and membrane filtration has been revealed in several studies (Semmens and Field 1980; Chadik and Amy 1983; Reckhow and Singer 1984; Hubel and Edzwald 1987; Krasner and Amy 1995). Kavanaugh (1978) and Babcock and Singer (1979) demonstrated that coagulation with alum and ferric salts was effective in removing turbidity and reducing organic DBP precursors. In the coagulation process, the effects of high dosage up to the overdose range are readily apparent because of the formation of hydrolyzed species. Edzwald (1993) found a better DOC removal by coagulants in water with higher specific UV absorbance. The U.S. EPA's 1998 Stage 1 disinfection by-product rule (DBPR) provides an NOM removal strategy called enhanced coagulation to limit the formation of DBP. TOC removal requirements based on the TOC-alkalinity matrix on source-water characteristics are shown in Table 1 (U.S. EPA 1998). White et al. (1997) reported that water sources with high TOC and low alkalinity would need the proposed Step 1 TOC requirements, while those source waters with low TOC were required to employ the alternative process to meet the drinking-water standards. The benefit to using this enhanced coagulation practice is to avoid forcing a unit to introduce a high dosage of coagulants to reduce the pH to between 5 and 6, which is, in general, the optimal pH range for the coagulation process. However, the coagulation process is only effective in removing large organic precursors, so the conventional treatment methods have limited efficiency in eliminating small precursors, which have high formation potential of DBP (Chang et al. 2001a,b).

¹Professor, Dept. of Biochemistry, Taipei Medical Univ., 250 Wu-shin St., Taipei 110, Taiwan.

²Professor, Graduate Institute of Environmental Engrg., National Taiwan Univ., 71 Chou-shan Rd., Taipei 106, Taiwan.

³Student, Dept. of Biochemistry, Taipei Medical Univ., 250 Wu-shin St., Taipei 110, Taiwan.

⁴Student, Graduate Institute of Environmental Engineering, National Taiwan Univ., 71 Chou-shan Rd., Taipei 106, Taiwan.

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Table 1. Total Organic Carbon Percent Removal Requirements for Enhanced Coagulation (Unit: %)

Total organic carbon (mg/L)	Source water alkalinity (mg/L as CaCO ₃)		
	0–60	>60–120	>120
>2.0–4.0	40	30	20
>4.0–8.0	45	35	25
>8.0	50	40	30

NOM concentrations in the source water of the King-Men Reservoir, Taiwan, are high and can not be easily reduced by the conventional-treatment process. For instance, TOC could only be reduced by 40% in the lime-coagulation process, resulting in the generation of high trihalomethane (THM) after chlorination. The purpose of this study was to evaluate NOM removal from source water by enhanced coagulation and activated carbon adsorption. The specific objectives were to (1) examine the characteristics of source water at the King-Men Reservoir; (2) assess the effect of polyaluminum chloride (PACl) and alum dosages, as well as pH, on NOM and turbidity removal; and (3) evaluate the effectiveness of activated carbon adsorption for reducing DBP precursors.

Materials and Methods

Characterization of Source Water

The source water from the King-Men Reservoir was collected from October 1998 to September 2000 for determining its molecular weight of NOM. NOM was fractionated by the UF membrane (A/G Technology), hollow-fiber modules with nominal molecular weight cut-off 1, 5, 10, and 30 kilodaltons (kDa). Each membrane was composed of cellulose derivative with a surface area of 24 cm². The applied pressure through the membrane ranged from 270–350 kPa, and the filtrate was collected and analyzed for DOC and DBPFP (Thurman and Malcolm 1981).

Coagulation Tests Performed by Alum and PACl

A jar test was used to evaluate the reduction of NOM and THM by alum and PACl. Reagent-grade alum [Al₂(SO₄)·18H₂O] (15% Al₂O₃) and PACl (10% Al₂O₃) were added at 20–130 mg/L and 30–450 mg/L, respectively. Water samples were stirred by conventional jar-test apparatus with rapid mixing at 100 rpm for 2 min, slow mixing at 30 rpm for 15 min, and quiescent settling for 60 min. H₂SO₄ was used for pH adjustment to evaluate the coagulation performance. At the end of the coagulation/settling period, 600 mL of the supernatant from the jar was collected and analyzed for TOC, turbidity, trihalomethane formation potential (THMFP) and haloacetic acid formation potential (HAAFP). Coagulated aliquots were filtered through pre-washed 0.45 μm filter to determine UV₂₅₄ and DOC. In addition, the filtered samples were also prepared to perform the adsorption isotherm studies and determine the potential to form THM and haloacetic acid (HAA).

Adsorption Isotherm Studies

At first, the source-water sample was coagulated with 40 mg/L alum (pH~6). At the end of the coagulation/settling period, the supernatant was filtered for activated carbon-adsorption studies. The Darco 20–40 mesh GAC (Aldrich) was first pretreated by

boiling it with distilled water for several hours, then subsequently rinsed with distilled water, and finally dried in an electric oven at 120°C overnight. Adsorption isotherm experiments including a wide range of adsorbent/adsorbate ratio (0.6–2.4 g carbon/4.5 mg DOC) were conducted at 22±2°C. The equilibrium time was designated as 24 h throughout the entire adsorption isotherm tests. The carbon particles were separated from the liquid phase by centrifugation. Samples from the aqueous phase were analyzed for residual TOC concentration and DBP formation.

Trihalomethanes and Haloacetic Acids Formation

The appropriate chlorine doses of a water sample were determined from 3.5–20.0 mg Cl₂/L based on the amount of free residual chlorine (APHA 1995). In general, the free residual chlorine was controlled in the range of 1.1–3.5 mg/L after at 20±2°C. If the free residual chlorine was lower than 1 mg/L, an additional amount, typically 3.5–20.0 mg Cl₂/L, was added to establish a definite residual. For the THM and HAA analysis, samples were collected at 6, 24, and 168 h, respectively, after chlorine addition. The reason for sampling at 6 h was to determine the instantaneous THM (HAA) and THMFP (HAAFP), respectively, while a 24-h sampling time was arbitrarily selected for comparison with the DBP formation with respect to 168 h. The pH value of the water samples was controlled by adding 5 mL phosphate buffer solution throughout these chlorination experiments. These treated samples were immediately sealed with a TFE-lined screw cap and then stored in the dark room. THMs were analyzed by high-pressure (HP) 6890 gas chromatography (GC)/ED. A microextraction procedure using *methyl-tert-butyl* ether [high-pressure liquid chromatography (HPLC) grade, Baker] and esterified with diazomethane, which was prepared in the laboratory following the procedures suggested by standard methods (APHA 1995), used to analyze HAA by the same GC. Standard solutions (Supelco) were prepared for calibrating four THM compounds (Cat. No. 48140-U) and five HAA compounds (Cat. No. 48047). In this research, according to the U.S. EPA's Stage 1 DBPR, five species of HAAs were taken into consideration (monobromoacetic acid (MBAA); dichloroacetic acid (DCAA); trichloroacetic acid (TCAA); bromochloroacetic acid (BCAA); and dibromoacetic acid (DBAA)). All the detailed quality assurance/quality control (QA/QC) requirements including detection limits, internal standard, surrogate standard, and preservative agents for DBP followed the procedures suggested by the standard methods (APHA 1995).

Analytical Methods

DOC, TOC, chlorine demand, DBPFP, UV₂₅₄, turbidity, pH, and alkalinity analyses were measured in raw-water and treated-water samples. All analyses, unless otherwise noted, were performed according to the 19th edition of the standard methods (APHA 1995). Water samples for DOC and UV₂₅₄ analyses were first filtered through a prewashed 0.45 μm filter and then determined by a TOC instrument (O. I. Corporation model 700) and UV spectroscopy (Hitachi U-2000), respectively.

Results and Discussion

Characterization of Raw Water

Table 2 presents the results of source-water analyses at the King-Men Reservoir, indicating a high TOC (>8 mg/L) and low alka-

Table 2. Characteristics of Source Water

Parameters	Concentration			Sample numbers
	Average	Range	Standard deviation	
pH	Not available	6.5–7.5	Not available	7
Alkalinity—mg/L as CaCO ₃	52.6	36.6–71.8	17.8	7
Turbidity—NTU	14.0	7.7–25.0	6.4	7
UV ₂₅₄	0.158	0.108–0.218	0.042	7
Total organic carbon—mg/L	9.6	7.0–12.4	1.9	7
Chlorine demand—mg/L as Cl ₂				
6 h	11.3	6.1–18.5	4.8	7
24 h	12.6	6.4–21.2	5.7	7
168 h	35.5	17.0–68.7	21.9	7
THM formation potential—μg/L				
6 h	186	118–226	41	6
24 h	361	168–487	109	6
168 h	703	367–1,427	344	7
HAA formation potential—μg/L				
168 h	471	236–655	156	7
Molecular size—%				
>1 k	41	33–47	7	3
1 k~5 k	11	6–18	6	3
5 k~10 k	5	2–7	3	3
10 k~30 k	1	1–2	1	3
>30 k	42	32–57	13	3

linity (<60 mg/L as CaCO₃). This source water has the dominant DOC distribution with MW less than 1 kDa (41%) and greater than 30 kDa (42%). Previous studies (Chiang et al. 2002) reported that MW distributions of reservoir-water samples were relatively even. These results are quite different from other studies (Amy et al. 1992; Newcombe et al. 1997), which suggests that the MW characteristics are site specific. The MW fraction affecting the performance of different treatment processes is discussed later.

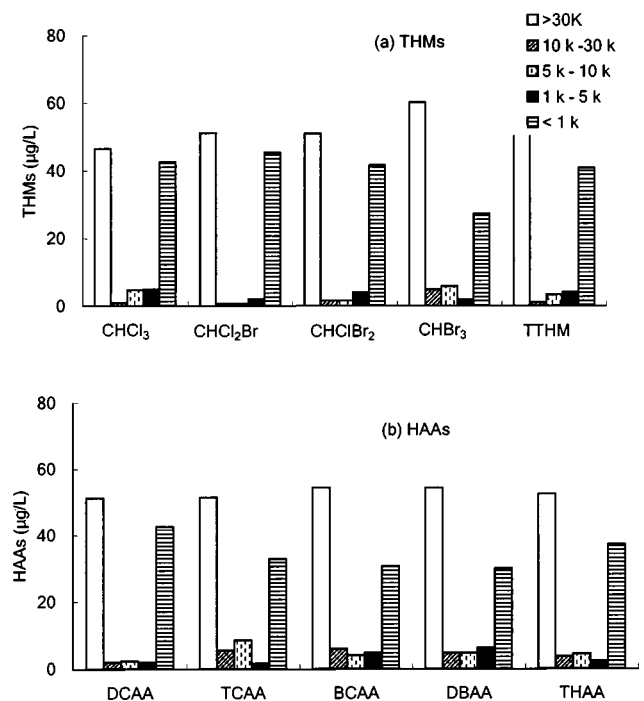
In addition, the percentages of THMs and HAAs at various MW ranges of organic matter of the source water are shown in Fig. 1. It is clear that King-Men source water contains higher concentrations of DOC in the MW fractions >30 kDa and <1 kDa. Accordingly, the highest distributions of four species of THMs are in the MW fractions >30 kDa, and the ratios range from 46–67%. On the other hand, the same as the distribution of THMs precursor, the HAAs precursor distributions of the source water constitute most of MW organics >30 kDa (55%) and <1 kDa (40%). Thus, removal of these two major fractions in water-treatment processes is the key to reduce the level of DBPs in the treated water.

In Table 2, it is also observed that the THM and HAA concentrations in the finished water at the King-Men water-treatment plant may not meet the future stringent drinking-water standards, i.e., 80 and 60 μg/L, respectively. Consequently, an alternative treatment method of ozonation, enhanced coagulation, adsorption, and/or nanofiltration would be required for reducing NOM. In this investigation, there were only two unit processes, coagulation and adsorption, selected for further evaluation.

The U.S. EPA proposed the following equation for modeling the concentration of THM in the course of drinking-water disinfection (Singer 1994):

$$\begin{aligned}
 \text{TTHM} = & 0.00309[(\text{TOC})(\text{UV}-254)]^{0.440} \\
 & \times (\text{Cl}_2)^{0.409}(t)^{0.265}(T)^{1.06}(\text{pH}-2.6)^{0.715} \times (\text{Br}+1)^{0.036}
 \end{aligned}
 \quad (1)$$

where TTHM=total trihalomethane concentration (μmol/L); *t*=time (h); *T*=temperature (°C); Br=bromide ion concentration (mg/L); and Cl₂=chlorine dose (mg/L). In Eq. (1) the pH index, pH-2.6, can be regarded as an important factor affecting THM formation. However, this index can be varied with the characterization of NOM in the different location. This hypothesis is also clearly demonstrated in Fig. 2, which indicates the pH index appears as pH-3.7, a proportional factor indicating its effect on the formation of THMs and HAAs.

**Fig. 1.** Molecular weight characteristics for source water (a) THMs and (b) HAAs.

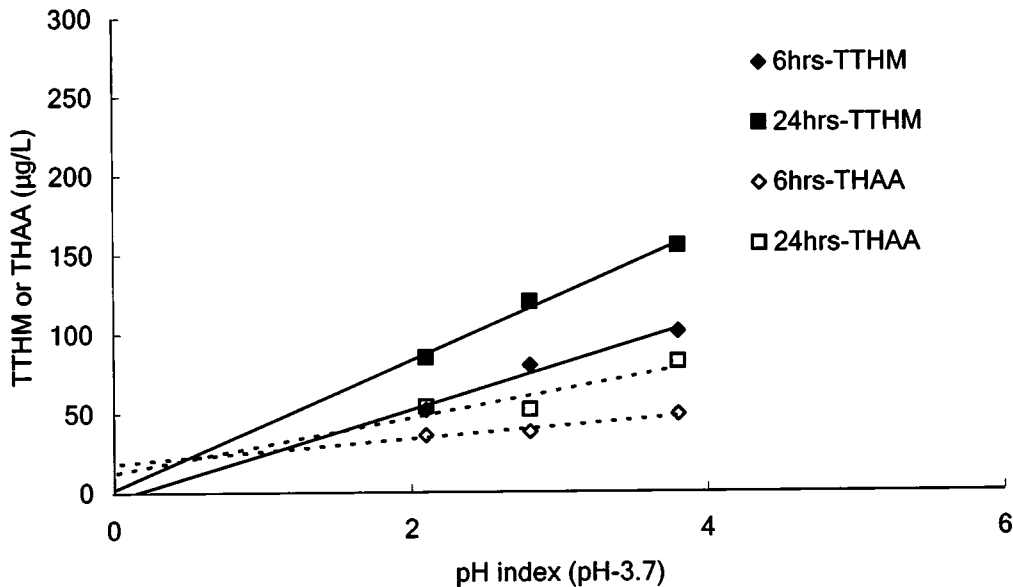


Fig. 2. Results of correlation between pH index

Reduction of Natural Organic Matter and Disinfection By-Products by Coagulation

An alum dose of 20 mg/L reduced the TOC concentration by 25%, as shown in Fig. 3. As the alum dosage increased further, the reduction in TOC and turbidity also increased gradually. As suggested by the U.S. EPA (1998), an alum dose of approximately 40 mg/L should be introduced to meet the Step 1 TOC removal requirement (50%) for this high-TOC and low-alkalinity water. For the present study, a 40 mg/L alum dosage decreased turbidity to below 2 NTU and was at the point of diminishing returns for TOC reduction. For example, as alum dosage increased from 60–90 mg/L, the difference in TOC removal efficiency was found to be insignificant (0.23 mg TOC/10 mg alum) even operating in the lower pH range (5.8), which is fairly consistent with the findings suggested by the U.S. EPA enhanced coagulation requirement. On the other hand, it is noted that restabilization may have occurred, thereby increasing the residual TOC and turbidity at the PACl dosage 450 mg/L at 10% Al_2O_3 [Fig. 3(b)]. This hypothesis could be further confirmed by the evidences shown later in Figs. 5(b) and 6(b), which exhibit relatively lower removal efficiencies at the PACl dosage 450 mg/L.

It is believed that the coagulation process had poor capability to eliminate small MW organics, which occupies the second high fractions of organic matters and DBP precursors in King-Men source water (Table 2 and Fig. 1). This observation is also consistent with our previous study evaluating the performance of a full-scale coagulation process, which indicated that the coagulation is able to remove large MW fractions (>30 kDa) with about 59% DOC reduction, as compared to 7% elimination for the MW fraction <1 kDa. (Chiang et al. 2002). Due to the characteristics of the source-water nature, the coagulation process has its limitation to reduce organics efficiently even at high-coagulant dose.

In Figs. 4 and 5, it is observed that $CHCl_3$ and $CHBrCl_2$ decrease rapidly at 60 mg/L dose (alum and PACl) and then decrease gradually with increased chemical dosage. Small quantities of $CHBr_2Cl$, however, do not change significantly. Observationally, $CHCl_3$ is the predominant species in the total THM formation due to the nature of the chlorination process as well as water characteristics, e.g., the incorporation factor and Cl_2/Br^- molar ratio (Chang et al. 2001a,b).

Fig. 6 presents the 6-h results of HAA formation in water samples treated by alum and PACl. TCAA is the major component of HAA. The patterns of the five HAA species at different levels of alum and PACl are as follows: (1) TCAA concentrations decreased gradually with increased chemical dosage; (2) MBAA and DCAA declined significantly; (3) BCAA decreased initially

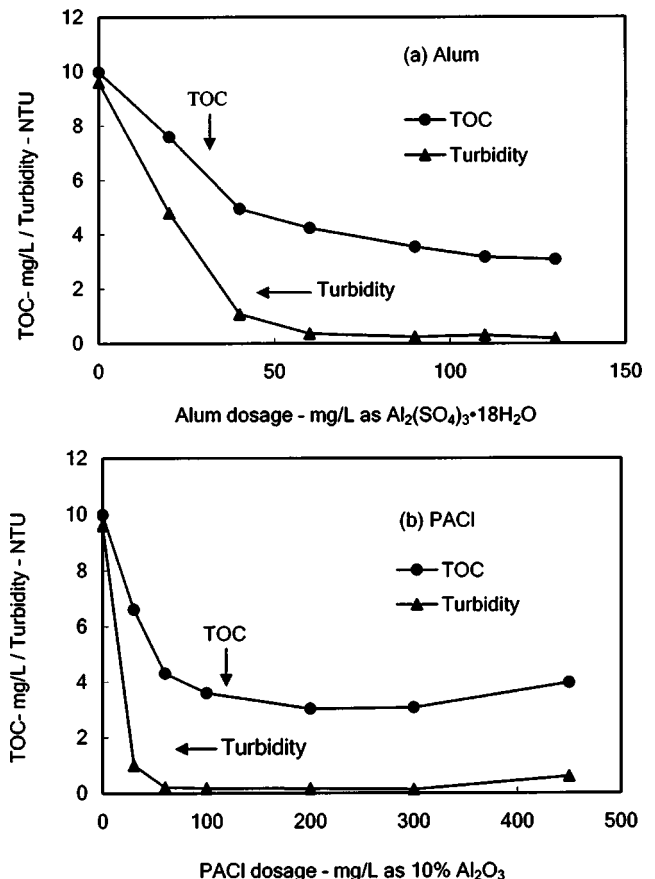


Fig. 3. Residual total organic carbon (TOC) and turbidity levels treated by various levels of (a) alum and (b) PACl coagulant dose

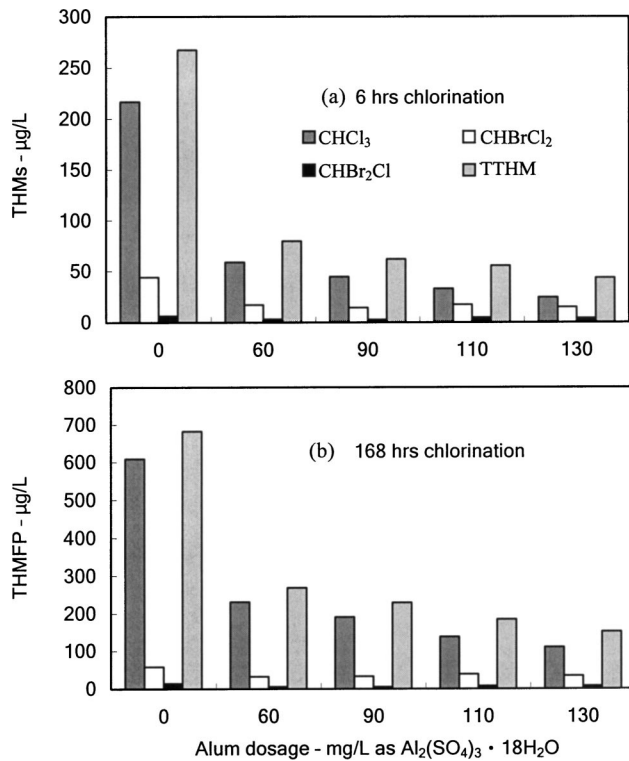


Fig. 4. (a) THMs and (b) THMFP in source water treated by alum

and then increased slowly in the alum coagulation case; and (4) DBAA did not change appreciably. In general, the alum and PACl coagulants can be successfully used in reducing HAA precursors except for the occurrence of restabilization at higher PACl dosages.

Figs. 7 and 8 show the THM and HAA formation in water samples treated by various levels of alum and PACl, respectively. For comparison purposes, chemical dosages are expressed as Al₂O₃. The organic precursors in the source water were reduced significantly by alum at doses higher than 10 mg/L as Al₂O₃, under which the residual turbidity was below 1 NTU. The TTHM and the total haloacetic acids (THAA) concentrations in the source water treated by PACl also decreased gradually up to 30 mg/L as Al₂O₃. However, as the PACl dosage increased to 45 mg/L as Al₂O₃, slightly increased concentrations of TTHM and THAA in the treated water are observed as was TOC [Fig. 3(b)]. TTHM (THAA) approached a level equivalent to 40% (30%) of DBPFP after 6 h of contact time. It is also noted that the TTHM concentration was higher than 80 µg/L, i.e., the future drinking-water-quality standard in Taiwan, even though the residual turbidity was controlled below 0.3 NTU by using the enhanced coagulation technique (110 mg/L alum or 16.5 mg/L alum as Al₂O₃). In comparison with the performance between alum and PACl (Fig. 3), a lower dose (60 mg/L as 10% Al₂O₃) of PACl coagulant is more effective in removing TOC and turbidity than the same dose of alum. The results show that the larger MW coagulant (PACl), having a higher-charge neutralization and bridging capacities, exhibits better performance than the lower MW alum. However, the benefit of using PACl chemical was intangible at a higher dosage of coagulant.

In addition, pH in the source water would be changed during the coagulation process. For instance, the pH value decreases from 7.0–5.8 after the coagulation process. It is necessary to re-adjust the pH value in the finished water to meet the drinking-water standard in Taiwan. Furthermore, the THMFP varied with

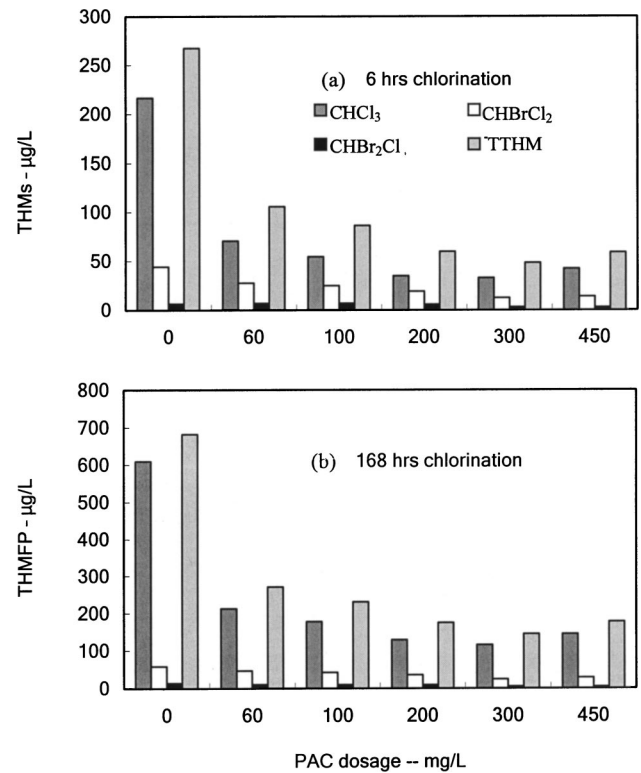


Fig. 5. (a) THMs and (b) THMFP in source water treated by PACl

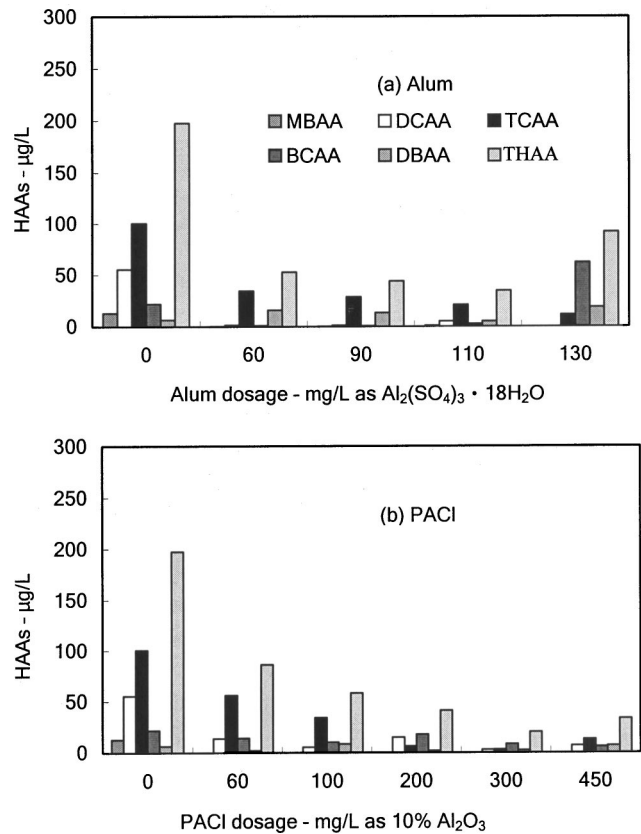


Fig. 6. HAA formation (6 h) in source water treated by (a) alum and (b) PACl

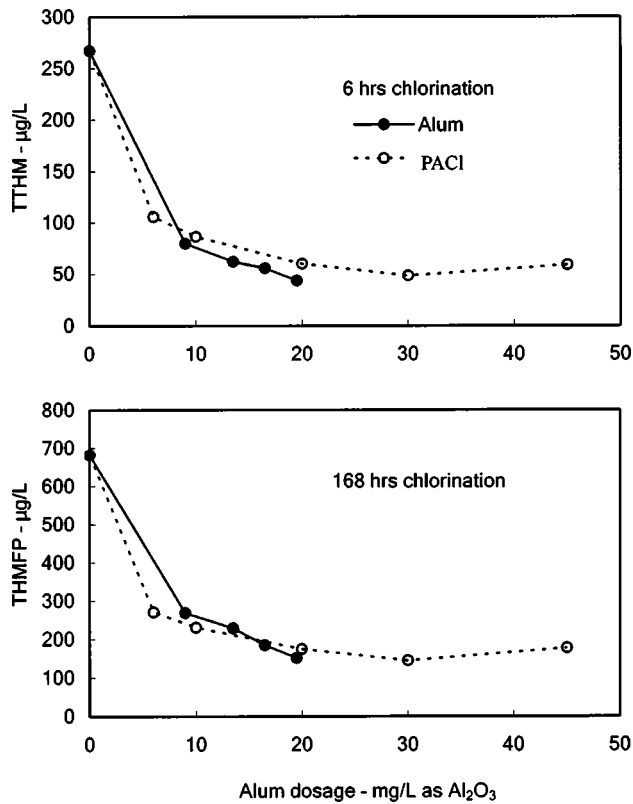


Fig. 7. Comparisons of TTHM and THMFP between alum and PACI

different acidic or alkaline conditions of the finished water as shown in Fig. 9. For HAA, it is observed that the pH at 5.8 and 6.5 did not make a significant difference in the formation of HAA. However, the pH value at 7.5 did increase 20–40% of the HAA formation as compared with the pH value at 6.5. The effect of pH on the formation of THM is more noticeable than on that of HAA, e.g., the measured TTHM concentrations (6 h) at pH 6.5 and 7.5 are 84 and 104 µg/L, respectively, which are higher than the drinking-water standard. This observation provides additional evidence that the treated water by PACI or alum coagulant might promote higher DBP formation if the final pH adjustment is needed.

Reduction of Natural Organic Matter and Disinfection By-Product by Adsorption

Some researchers have indicated that coagulation with alum is effective for the removal of organic DBP precursors (Babcock and Singer 1979; White et al. 1997). On the contrary, Crozes et al. (1995) suggested that the anticipated maximum contaminant levels for HAA and THM could hardly be met by using enhanced coagulation. Recently Singer and Bilyk (2002) used alum combined with an ion-exchange resin to remove organic matters and DBP precursors from natural waters, and indicated that the combination method was more effective as compared to alum coagulation alone despite the fact that lower alum doses were used. This result implies that the precursors of THMs and HAAs are better reduced by other mechanisms such as adsorption; hence GAC was introduced to reinforce alum or PACI treatment.

NOM is adsorbed by activated carbon depending on conditions such as pH and DOC concentration. Lower pH reduces the charge density of humic and fulvic acids making them more hydrophobic (Krasner and Amy 1995). In general, activated carbon

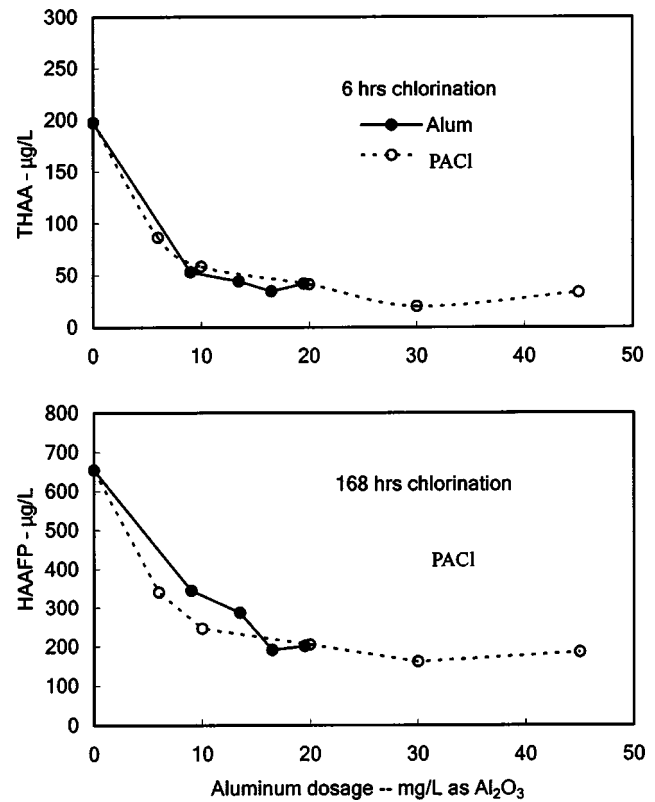


Fig. 8. Comparisons of THAA and HAAFP between alum and PACI

display has a higher adsorption capacity at a lower pH, which can be ascribed to the decrease in surface NOM electrostatic repulsions, as well as to the decrease in the solubility and in the size of the NOM molecule at pH 3 (Newcombe 1999). In addition, our colleague reported that TOC removal by adsorption increased about 10% and 30% as the pH was reduced from 8.0–6.0, and 2.0, respectively. It was noted that the pH reduction prior to GAC adsorption might be beneficial for a water-treatment unit, but the cost of pH control for adding acid to reduce the pH and adding alkali to restore a stable pH and following a GAC unit is debatable. (Chen 1996) Consequently, the adsorption-isotherm experiments in this investigation were conducted at pH 5.8–7.5 after the coagulation tests.

Because a large MW fraction of NOM can cause pore blockage of activated carbon and result in the decrease of effective

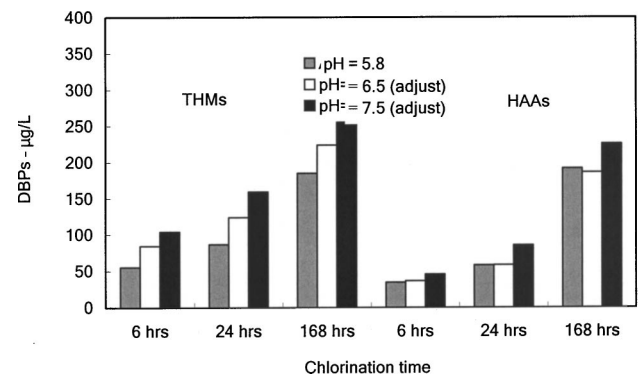


Fig. 9. Disinfection by-product (DBP) formation of source water treated by alum at different levels of pH

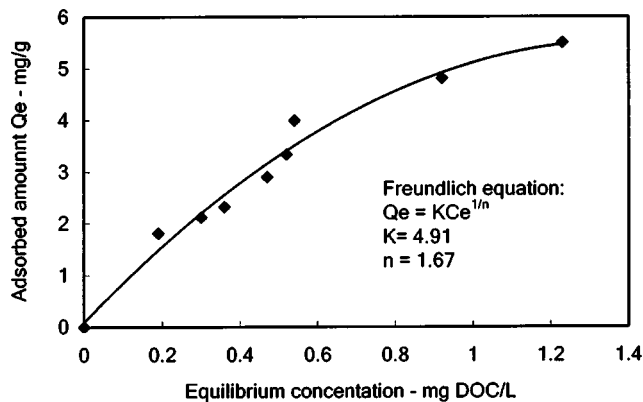


Fig. 10. Adsorption isotherm of dissolved organic compound (DOC) on Darco (Aldrich) activated carbon (initial DOC=4.5 mg/L)

adsorption sites (Pelekani and Snoeyink 1999; Ebie et al. 2001), the coagulation preceding the GAC process can prevent the GAC from pore blockage by large MW organics. Furthermore, the remaining small MW organics after coagulation/sedimentation processes can be expected to be removed by GAC adsorption. Fig. 10 presents the results of Darco carbon-adsorption isotherm studies, indicating that a favorable adsorption results in an effective reduction of DBP precursors. The Freundlich isotherm reasonably predicts the observed values. The plots in Fig. 11 reaffirm the fact that THM and HAA are greatly reduced with increased GAC dosage. For instance, the measured DBP were found to be below 20 $\mu\text{g/L}$ at GAC 1.0 g/L.

Conclusion

The characterization of NOM in the King-Men source water has the dominant TOC distribution with MW less than 1 kDa (41%) and greater than 30 kDa (42%). Accordingly, the highest distributions of THMs and HAAs are in the MW fractions >30 kDa and <1 kDa. With this every unique characterization, a lower alum dosage could reduce the TOC concentration by a great amount (25%); while at a higher dosage (60–90 mg/L), the difference in TOC removal efficiency was found to be insignificant (0.23 mg TOC/10 mg alum), even operating in a lower pH range. This is probably due to the characteristics of the source-water nature, the coagulation process has its limitation to reduce organics efficiently even at a high-coagulant dose. With the PACl dosage at a higher level, restabilization occurred, resulting in higher TOC and THM concentrations in the treated water.

In comparison with the performance between alum and PACl, a lower dose of PACl coagulant is more effective in removing TOC and turbidity than the same dose of alum. The results show that the larger MW coagulant (PACl), having a higher-charge neutralization and bridging capacities, exhibits better performance than the lower MW alum. However, the benefit of using a PACl chemical was intangible at a higher dosage of coagulant. It was also observed that the pH at 5.8 and 6.5 did not significantly affect the formation of HAA, while a pH of 7.5 did increase the HAA formation by 20–40%. The results further demonstrate that the treated water by PACl or alum coagulant might promote higher DBP formation if the final pH adjustment is needed.

The GAC adsorption process provides a relatively lower THMFP in the treated water at the same level of THM and in addition to lower the coagulant doses demand. In addition, the

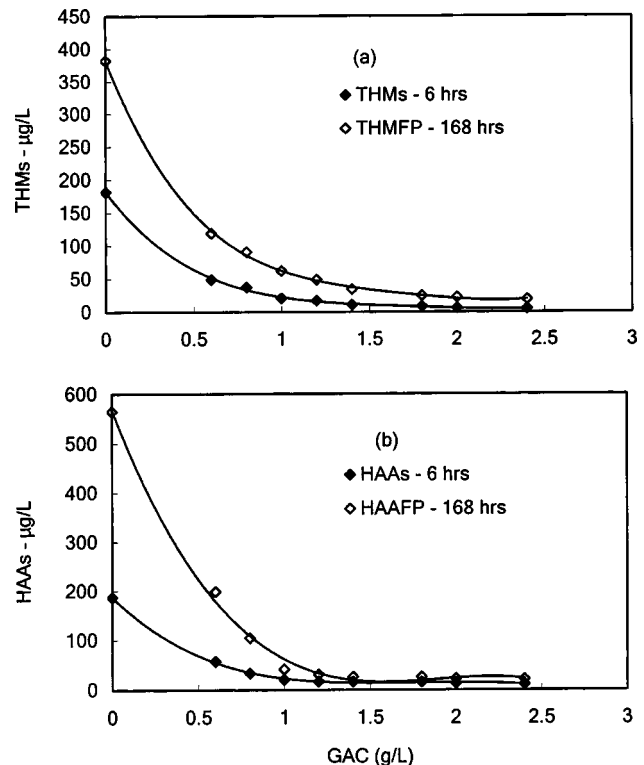


Fig. 11. Disinfected by-product formation treated by various levels of GAC (a) THMs and (b) HAAs

GAC process can effectively remove small MW organics which is difficult to remove by the coagulation process. Thus, the water treated by the alum coagulation followed by adsorption process should provide safer drinking-water quality with regard to DBP formation, and the removal mechanisms should be described quantitatively in future work.

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