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Research Article
Characterization of Trihalomethanes (THMs) Levels in Surface Water, Domestic and Industrial Wastewater

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Abstract
Trihalomethanes (THMs) are one of the disinfection by-products (DBPs) groups that formed due to the reaction of organic matters with inorganic DPBs precursor and disinfectant. In this study, water (tap water, laboratory, river, Alur Ilmu and lake water, Engineering Lake), domestic (sewage) and industries wastewater (pharmaceutical and pulp and paper mills) were sampled in order to characterize and compare the level of THMs with the regulated limits of wastewater effluent and drinking water. The concentrations of total THMs (TTHMs) in raw and effluent of pharmaceutical and pulp and paper mills wastewater were 2589.24 and 1565.01 μg L^{-1}, 2008.40 and 1519.63 μg L^{-1}, respectively. The concentration of TTHMs in Engineering Lake was in range of 1481.54-1520.96 μg L^{-1}, while in Alur Ilmu was around 2923.78-3724.05 μg L^{-1}. It was also unexpected results that the levels of the TTHMs in laboratory tap water was in range of 1498.64-2288.46 μg L^{-1} which was higher than regulated limits for drinking water. Thus, further treatment of THMs is required either using chemical or biological treatments.

Key words: Trihalomethane (THMs), disinfection by-products, micropollutants, disinfection, drinking water

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Data Availability: All relevant data are within the paper and its supporting information files.

INTRODUCTION

Water is a key resource for living things on earth. In addition to the need for eating and drinking, human also required water for other purpose such as washing, agriculture, industrial and transportation. Other species such as animals and plants required water for surviving. Water that suitable for human consumption is safe and clean water. Polluted water consumed by humans harmful and toxic. Example of pollutants are heavy metal, organic and inorganic compounds and micropollutants. The organic contaminants are protein, carbohydrates, fat, nucleic combination as well as Chemical Oxygen Demand (COD) and Biological Oxygen Demand (BOD) parameters, whereas inorganic contaminants are such as ammonia, phosphate, nitrate, sulphate and heavy metals. In addition, micropollutants such as pharmaceuticals compounds, personal care products (PPCPs), disinfection by-products (DBPs) and Endocrine Disrupting Compounds (EDCs). Disinfection is a vital process to inactivate pathogenic microorganisms in drinking water and wastewater. It also act as the cornerstone unit operation of water treatment process that secure drinking water safety (Crittenden *et al*., 2012; Ohar and Ostfeld, 2014) but since the 1970s it has been recognized that disinfection can produce harmful by-products and cause health concerns (Hua and Yeats, 2010). There are 600-700 types of DBPs are formed when naturally occurring organic matter interacts with halogens during treatment (Burch *et al*., 2015; Krasner, 2009; Nieuwenhuijsen *et al*., 2009).

The present of Natural Organic Matter (NOM) and Dissolved Organic Matter (DOM) in water sources are the primary precursor for the formation of DBPs in drinking water. The NOM is a complex mixture of organic compounds derived from the decay of vegetation and animal material (Hua and Yeats, 2010), while DOM consists of wide range of different compounds such as humic substances, hydrophilic acids, carbohydrates, amino acids and carboxylic acids (Thurman, 1985). Some components of the DOM react with disinfectants to form DBPs such as THMs: chloroform ($CHCl_3$), dibromochloromethane ($CHBr_2Cl$) and bromodichloromethane ($CHBrCl_2$), bromoform ($CHBr_3$) and haloacetic acids (HAA) (Pramanik *et al*., 2015) which are potentially carcinogenic (Nikolaou *et al*., 2004).

The THMs are among the most abundant and thoroughly studied DBPs (Burch *et al*., 2015) and categorised as volatile compounds (Zhang *et al*., 2015). In the most cases reported, THMs have the highest occurrence levels in the disinfected drinking waters and are currently regulated in number of countries (Wang *et al*., 2015a), bodies or organisation such as United States, Florida, Canada, Australia, European Union (EU), Japan, Netherlands, New Zealand, South Africa, Sweden, United Kingdom, Malaysia and World Health Organization (WHO). Human exposure to THMs depends not only on direct ingestion of drinking water but also occurs due to volatilization of DBPs during showering, bathing, cooking or to other behavioural or lifestyle factors (Richardson *et al*., 2007; Rivera-Nunez *et al*., 2012). Due to the impacts of severe toxicity occurs because of THMs, the compound levels should be accessed and removed from drinking water and wastewater.

The presence of THMs is not considered as a parameter in effluent of wastewater even though it has been identified as genotoxic mutagen (Zhang *et al*., 2013). According to United Stated Environmental Protection Agency, (USEPA., 1998), the Maximum Concentration Level (MCL) of total THMs (TTHMs) in drinking water is 80 µg L^{-1} that regulated based on the potential of increased risk of cancer and other health effects. On top of that, Ministry of Health Malaysia have also set quality standards of THMs for drinking water as shown in Table 1. Meanwhile, to protect surface water quality, the Florida Department of Environmental Protection (2015) have set regulatory limits of THMs for treated wastewater effluents that discharged to surface water as listed in Table 2. Hence, it is really important to remove the THMs that presence in water and wastewater.

In this study, THMs levels in tap, river and lake water, domestic and industrial wastewater were evaluated. The sample of industrial wastewater was taken from pharmaceutical and pulp and paper mill industries and sewage treatment plant before and after treatment, respectively. Meanwhile, domestic wastewater, tap, river and lake water were periodically sampled in Universiti Kebangsaan Malaysia (UKM) area.

MATERIALS AND METHODS

Sampling of water and wastewater: Industrial wastewater was sampled from pharmaceutical and pulp and paper mill industries located in Selangor, Malaysia on February, 2015. There were two type of sample i.e., raw wastewater and final effluent. Lake (Engineering Lake) and river water (Alur Ilmu) were sampled on June, 2015 and domestic wastewater (Bukit Putri pond) was sampled on September-October, 2015 in UKM area (Fig. 1). A tap water from chemical laboratory was also weekly sampled for a month from April-May, 2015. This tap water is used by many people for experiment purposes.

There were 4 sampling points of water samples at Engineering lake (Fig. 1a), (1) At the beginning of the lake

Table 1: Different national approach to the regulation of THMs in drinking water

Country	Organisation	Date set	$CHCl_3$ (µg L^{-1})	$CHBrCl_2$ (µg L^{-1})	$CHBr_2Cl$ (µg L^{-1})	$CHBr_3$ (µg L^{-1})	TTHMs (µg L^{-1})	Reference
Australia	NHMRC	2004	-	-	-	-	250	Jackson *et al.* (2008)
Canada	HC	2007	-	16	-	-	100	
European Union	EC	1998	-	-	-	-	100	
Germany	FHM	2001	-	-	-	-	50	
Japan	MHLW	2004	60	30	100	90	100	
Netherlands	VROM	2000	-	-	-	-	25	
New Zealand	MH	2005	200	60	150	100	a	
South Africa	DWAF	2005	-	-	-	-	200	
Sweden	NFA	2001	-	-	-	-	100	
UK: England/ Wales	DWI	2000	-	-	-	-	100	
UK: Northern Ireland	DWI	2007	-	-	-	-	100	
UK: Scotland	DWR	2001	-	-	-	-	100	
USA	EPA	2006	80	80	80	80	80	
World	WHO	2006	300	60	100	100	100	
Malaysia	MH	2010	200	60	100	100	a	Drinking water quality Surveillance Programme (2010)

NHMRC: National health and medical research council, HC: Health canada, EC: European commision, FHM: Federal health ministry, MHLW: Ministry of health, labour and welfare, VROM: Ministry of housing, spacial planning and the environment, MH: Ministry of health, DWAF: Department of water affair and forestry, NFA: National food administration, DWI: Drinking water inspectorate, DWR: Department of water resources, EPA: Environmental protection agency, WHO: World health organization, MH: Ministry of Health. For $CHCl_3$, $CHBrCl_2$, $CHBr_2Cl$ and $CHBr_3$, the sum of the ratio of the concentration of each to its respective guideline value should not exceed 1

Table 2: Criteria for surface water quality classifications for class I, II and III (FDEP, FAC 62-302.530)

THMs	Limits for potable water supply (µg L^{-1})
CHCl3	≤5.67
$CHBrCl_2$	≤0.27
$CHBr_2Cl$	≤0.41
$CHBr_3$	≤4.30
TTHMs	≤10.65

$CHCl_3$: Chloroform, CHB_2rCl: Dibromochloromethane, $CHBrCL_2$: Bromodichloromethane, $CHBr_3$: Bromoform, TTHMs: Total trihalomethanes

which is near the cafeteria, (2) At the middle of the lake, (3) At the drainage and (4) Discharged of the lake to river. Alur Ilmu is a 1.79 km long stretching across the main campus of UKM before flowing into the Langat river (Din *et al*., 2012). For Alur Ilmu river, there were 3 sampling points (Fig. 1b), (1) At Pusanika before cafeteria effluent discharges to Alur Ilmu (Pusanika1), (2) At Pusanika after cafeteria effluent discharges to Alur Ilmu (Pusanika2) and (3) At UKM mosque, where, it is the last point of Alur Ilmu before flowing into the Langat river. Meanwhile for Bukit Putri pond, there were 2 sampling points (Fig. 1c), (1) At the beginning of the first pond and (2) Effluent of the second pond.

Samples pre-treatments: All samples were filtered using Puradisc 25 nylon syringe filter (United Kingdom) with 0.45 µm pore size to remove all particle and suspended solid in the sample.

Liquid-liquid extraction: Liquid-liquid extraction was performed prior analysis of THMs. About 10 mL of water sample was shaken vigorously with 2 mL of n-pentane for 1 min to obtain phase separation. Then the upper phase was collected into 2 mL vials with screw cap and teflon septa. The extracted sample was preserved at 4°C before analysis.

Analysis using GC-ECD: The extracted THMs in n-pentane was measured using gas chromatography (GC, Agilent Technology 7890A, USA) equipped with an Electron Capture Detector (ECD). The DB-5 capillary column with 60 m length and 0.32 mm internal diameter was used. The carrier gas was helium with a flow rate was adjusted to 10 mL min^{-1}. Initially, the temperature was kept constant at 40°C with 4 min holding time. After that, the temperature was increased until 100°C and 150°C with the holding time of 4 and 1 min, respectively.

RESULTS AND DISCUSSION

Chromatogram of THMs detection by GC-ECD: Figure 2 shows the sharp chromatography of THMs detection for laboratory tap water sample. The retention time for $CHCl_3$, $CHBrCl_2$, $CHBr_2Cl$ and $CHBr_3$ were 4.86, 6.97, 9.18 and 11.74 min, respectively. These retention times were consistent with other types of domestic and industrial wastewater and surface water (lake and river).

Concentration of THMs in industrial wastewater: Figure 3 shown the concentration of $CHCl_3$, $CHBrCl_2$, $CHBr_2Cl$ and $CHBr_3$ in industrial wastewater as well as pharmaceutical and pulp and paper mill industries. The concentration of TTHMs for pharmaceutical industry in raw wastewater and final effluent were 2589.24 and 1565.01 µg L^{-1}, respectively. It was recorded that the concentration of $CHCl_3$ was 2462.60 and 1526.94 µg L^{-1}, $CHBrCl_2$ was 83.12 and 22.62 µg L^{-1} and $CHBr_2Cl$ was 43.52 and 15.45 µg L^{-1}, respectively in raw wastewater and final effluent, while there was no $CHBr_3$ detected in the pharmaceutical sample.

Fig. 1(a-c): Sampling point of water in UKM nearby, (a) Engineering lake, (b) Alur Ilmu and (c) Bukit Putri pond

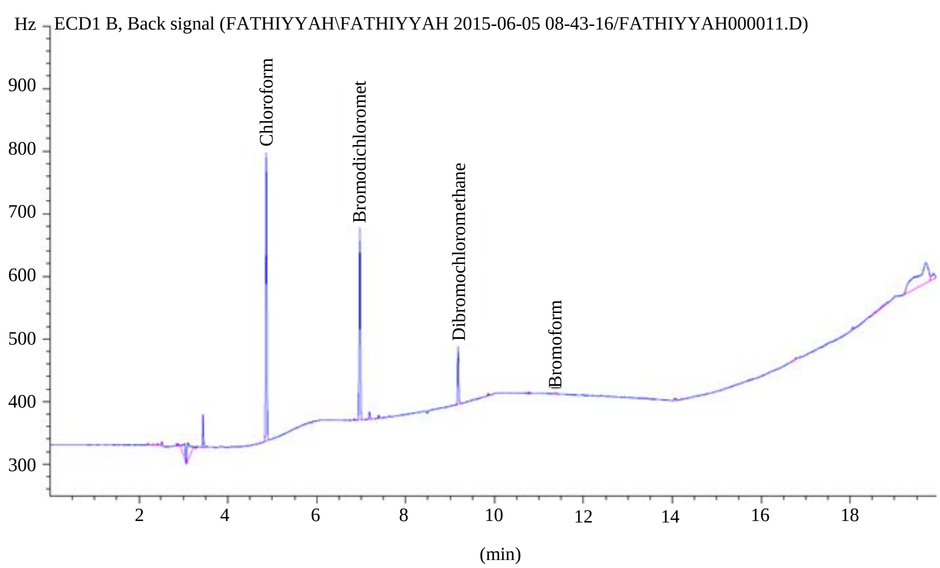

Fig. 2: Chromatogram of THMs detection on sample of tap water

The concentration of TTHMs in raw wastewater and final effluent of pulp and paper mill industry for were 2008.40 and 1519.63 µg L^{-1}, respectively. The concentrations of $CHCl_3$ were 1617.22 and 1506.81 µg L^{-1}, $CHBrCl_2$ were 208.29 and 12.82 µg L^{-1}, respectively in raw wastewater and final effluent. However, there were no $CHBr_2Cl$ and $CHBr_3$ detected in final effluent of pulp and paper mill industry. Even though, there were 39.6 and 24.3% of TTHMs reduction after treatment of pharmaceutical and pulp and paper mill industries wastewater respectively, the levels of TTHMs are still

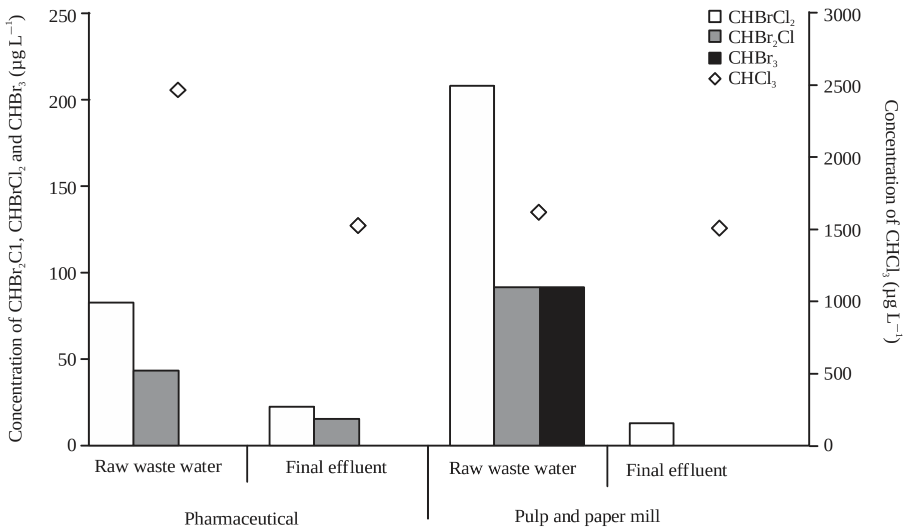

Fig. 3: Concentration of THMs in industrial wastewater

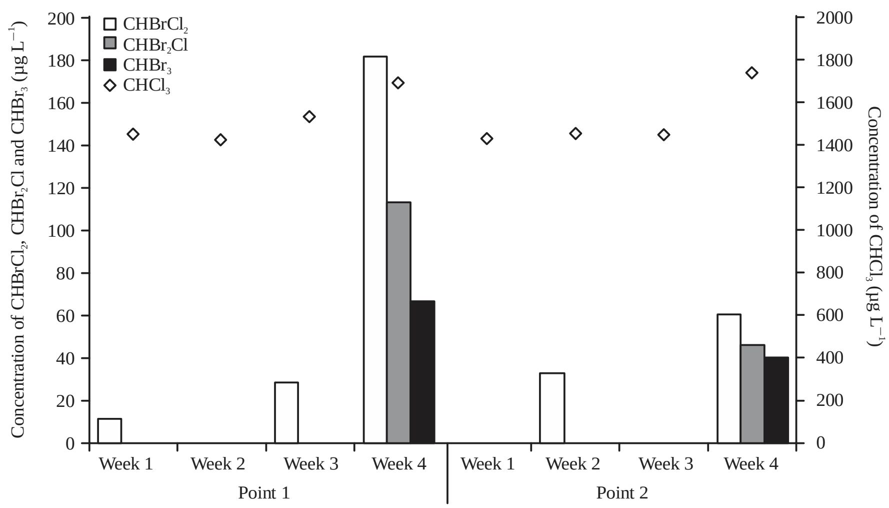

Fig. 4: Concentration of THMs in domestic wastewater

high compared to the regulated limit by Drinking Water Quality Surveillance Programme (<460 µg L^{-1}).

In pharmaceutical industry, the bacterial levels in raw wastewater is quit high that municipalities will generally perform multiple chlorination steps throughout the treatment process (Collentro, 2010). On the other hand, pulp and paper mill industry uses chlorine for bleaching of wood pulp in their production process. The reaction of organic matters that present in the pharmaceutical and pulp and paper mill wastewater with residual disinfectant produces undesirable compounds of THMs.

Concentration of THMs in domestic wastewater: Based on Fig. 4, the concentrations of TTHMs in domestic wastewater (Bukit Putri Sewage Treatment Pond) at point 1 and 2 sampled in week 1, 2 and 3 contain no $CHBr_2Cl$ and $CHBr_3$, while the level of $CHBrCl_2$ and $CHCl_3$ were detected lower than 40 µg L^{-1} and 1600 µg L^{-1}, respectively. Otherwise, in week 4, the concentration of $CHBr_2Cl$ and $CHBr_3$ were recorded higher than in week 1, 2 and 3 with the concentrations of $CHBrCl_2$ and $CHCl_3$ were higher than 60 and 1700 µg L^{-1}, respectively. The sample was taken at the same time and day for every weeks but in week 4, the sample was taken the day after rainy day.

Concentration of THMs in Engineering Lake and Alur Ilmu river, UKM: There were 4 sampling points at Engineering Lake and 3 sampling points at Alur Ilmu river. At Engineering Lake, the concentration of TTHMs for cafeteria area, middle of the lake, the end of the lake and the discharge form the lake

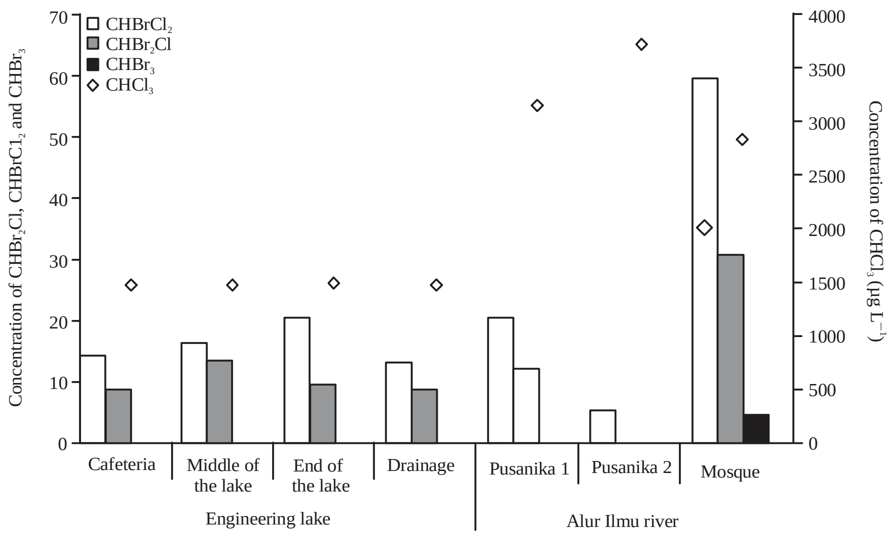

Fig. 5: Concentration of THMs in Engineering Lake and Alur Ilmu river

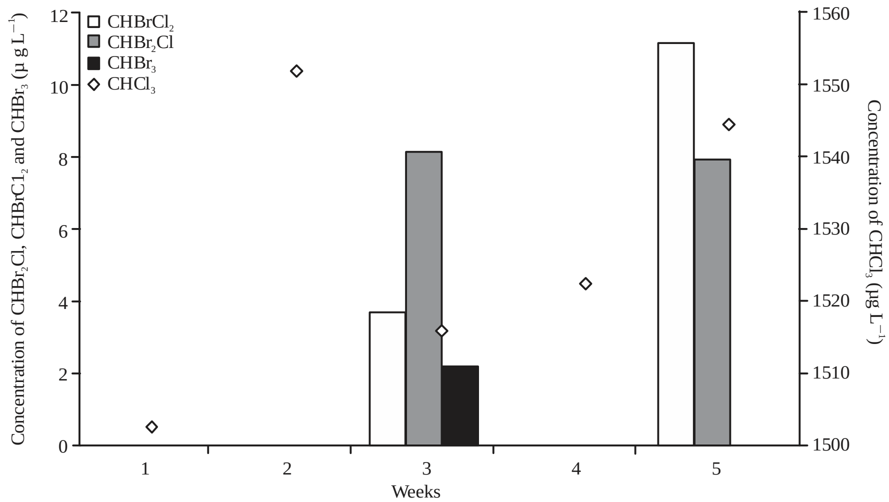

Fig. 6: Concentration of THMs in laboratory tap water for monitored every week for a month

were 1500.62, 1505.53, 1520.96 and 1481.54 µg L^{-1}, respectively (Fig. 5). The concentrations of TTHMs were significantly varied between the 4 sampling locations of Engineering Lake ($p<0.05$). At Alur Ilmu river, the concentration of TTHMs before (Pusanika 1) and after (Pusanika 2) discharge of cafeteria wastewater were 3185.01 and 3724.05 µg L^{-1}, respectively. Meanwhile the TTHMs at the last point of Alur Ilmu before entering Langat river was 2923.78 µg L^{-1}. From the results, it can be seen that the concentration of THMs in the surface water higher than regulated limits (<10.65 µg L^{-1}) (FDEP, FAC 62-302.530).

Higher concentration of THMs in Engineering Lake and Alur Ilmu river `are due to the activities of cleaning, washing and discharge of food waste by cafeteria nearby. The discharge of food waste that may contain organic matters increases the quantity of organic matters in lake and river water. Other than that, lake and river are also expose to the any dead leaf and small animal which easily fall in it consequently releases organic matters during degradation.

Concentration of THMs in laboratory tap water: Based on Fig. 6, the levels of TTHMs in laboratory tap water was monitored for a month. Throughout the period, the level of TTHMs for week 1, 2, 3, 4 and 5 were recorded as 1502.60, 1551.77, 1529.81, 1522.55 and 1563.52 µg L^{-1}, respectively. The variation of THMS concentrations between the weeks was not statistically significant difference ($p>0.05$). At week 1, 2 and 4, there were no other compound were detected present in the sample except $CHCl_3$, while in week 3,

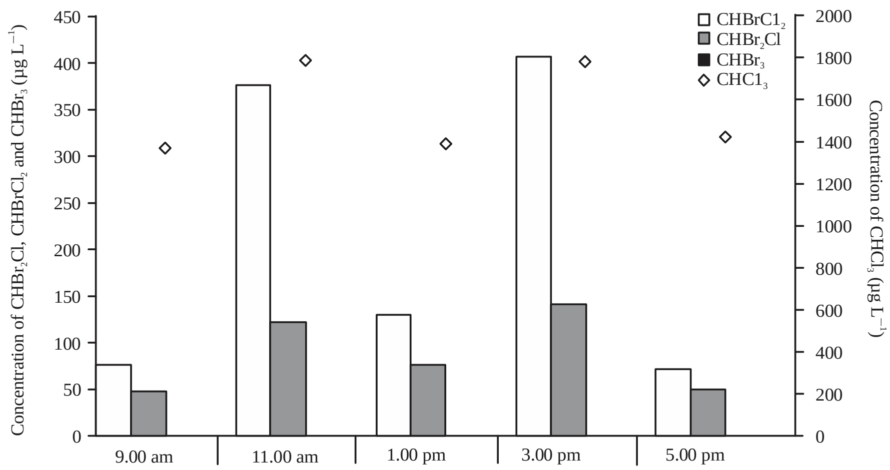

Fig. 7: Monitoring of THMs levels in laboratory tap water for every 2 h

Table 3: Comparison of TTHMs level in water and wastewater from different country

Sample type	Country	Place	TTHMs (µg L^{-1})	Reference
Industrial	Malaysia	Pharmaceutical	1565-2589	Present study
Wastewater		Pulp and paper mill	1519-2008	Present study
	China	-	940-950	Zhang *et al.* (2015)
	Thailand	-	480-491	Musikavong *et al.* (2005)
Surface water (lake, river or island)	Malaysia	Engineering Lake, UKM	1481-1521	Present study
		Alur Ilmu river, UKM	2923-3724	Present study
	Canada	St. Lawrence river	17-19	Wang *et al.* (2015b)
		Simcoe lake	3-4	Wang *et al.* (2015b)
		Capilano lake	44	Sarathy and Mohseni (2010)
		Northern arm lake	70.80	Newfoundland and Labrador (2015)
		Sandy lake	5.72	
		Beverly lake	54.15	
		Shoal Harbour river	55.57	
		Torrent river	166.25	
	Southeast spain	Segura river	91	De la Rubia *et al.* (2008)
	Taiwan	Wu-Lo river	205-262	Wang *et al.* (2015a)
	Japan	Chichijima island	165-200	
		Hahajima island	130-140	Phetrak *et al.* (2016)
				Phetrak *et al.* (2016)
	United state	Silver lake	240	
		Lansing lake	240	Siddiqui *et al.* (2000)
				Karnik *et al.* (2005)
Domestic wastewater	Malaysia	Bukit Putri pond	1423-2059	Present study
	Canada	Burdett's pond	222.15	Newfoundland and Labrador (2015)
		Northwest pond	160.25	
		Burnt pond	148.98	
		Lee's pond	191.25	
		Jack's pond	53.93	
Tap water	Malaysia	Environment laboratory, UKM	1498-2333	Present study
	United State	-	0.26-167	Burch *et al.* (2015)

the sequence concentrations of THMs groups were $CHCl_3$>$CHBr_2Cl$-$CHBrCl_2$>$CHBr_3$. Otherwise the sequence concentrations of THMs groups were in week 5 were $CHCl_3$>$CHBrCl_2$>$CHBr_2Cl$.

By monitoring the TTHMs levels in the laboratory tap water according to time, it was found that the THMs concentration varied in range of 1498.64-2333 µg L^{-1} (Fig. 7). The sequence concentrations of THMs groups for all weeks were as follow: $CHCl_3$>$CHBrCl_2$>$CHBr_2Cl$>$CHBr_3$. Even though the variation of THMs in weekly samples does not have significant difference but when the sample of laboratory tap water was strictly monitored every 2 h, the concentration of THMs significantly varies ($p<0.05$). From the TTHMs characterisation in laboratory tap water, it depicted that the tap water is not safe to be used directly either as drinking water or other daily purposes.

The standard limits of THMs sets by Ministry of Health Malaysia through Drinking Water Quality Surveillance Programme (2010) was below 460 µg L^{-1} and USEPA was below 80 µg L^{-1}. As obtained in this study, the concentrations of THMs in laboratory tap water exceeded the regulation limits. To go details, Drinking Water Quality Surveillance Programme (2010) have sets the regulation limits for each THMs groups of $CHCl_3$, $CHBrCl_2$, $CHBr_2Cl$ and $CHBr_3$ were below than 200, 60, 100 and 100 µg L^{-1}, respectively.

Comparison of THMs levels evaluated in this studies with other: In Malaysia, the level of THMs water and wastewater is not thoroughly investigated. Hence, this preliminary research determined the level of THMs as well as TTHMs in water and industrial and domestic wastewater as to evaluate whether its follow regulation limits. As summarized in Table 3, the levels of TTHMs in industrial wastewater in China (Zhang *et al*., 2015) and Thailand (Musikavong *et al*., 2005) were recorded below than 1000 µg L^{-1}. Moreover, TTHMs levels in domestic wastewater (sewage wastewater) in Canada was in range of 53.93-222.15 (Newfoundland and Labrador, 2015). These concentrations were much lower compared to the industrial and domestic wastewater (pharmaceutical and pulp and paper mill) in Malaysia. A higher level of THMs level in Malaysia domestic wastewater, might be due to the ineffectively of treatment processes for organic matters removal, thus forming THMs.

Surface water such as river and lake in Canada (Sarathy and Mohseni, 2010; Wang *et al*., 2015b) and Southeast Spain (De la Rubia *et al*., 2008) contains TTHMs lower than 100 µg L^{-1} except for Torrent river in Canada which TTHMs levels was 166.25 µg L^{-1} (Newfoundland and Labrador, 2015). The TTHMs levels in surface water for other countries such Taiwan, Japan and United State had were in the range of 130-262 µg L^{-1}. As similar with the TTHMs level in industrial and domestic wastewater, the TTHMs levels in surface water in this study area were also higher that standard limits regulated.

CONCLUSION

As a conclusion, TTHMs levels in industrial wastewater (pharmaceutical and pulp and paper mill), sewage wastewater, surface and tap water were detected higher than regulated standard limits. The $CHCl_3$ was the highest compound among others following by $CHBrCl_2$, $CHBr_2Cl$ and $CHBr_3$. Further treatment of TTHMs or preventing action should be done either using physical-chemical or biological treatment in order to ensure that the wastewater effluent, surface water and tap water are safe to be discharged and used.

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