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THE EFFECTS OF CATCHMENT SOILS AND LAND-USE ON THE RELATIONSHIP BETWEEN ORGANIC MATTER WITH CHLORINE DECAY AND THMFP

Hur jordart och markanvändning i avrinningsområdet påverkar sammansättningen av organiskt material, klorsönderfall och trihalometanbildningspotential

by JOHN AWAD[1,], JOHN VAN LEEUWEN[1,6,7], CHRISTOPHER CHOW[1,2,6], MARY DRIKAS[1,2,6],*
RONALD J. SMERNIK[3], DAVID J. CHITTLEBOROUGH[4], ERICK BESTLAND[5]
1 Centre for Water Management and Reuse, School of Natural and Built Environments,
University of South Australia, South Australia 5095, Australia
2 Australian Water Quality Centre, SA Water Corporation, 250 Victoria Square, Adelaide, South Australia 5000, Australia
3 School of Agriculture, Food & Wine and Waite Research Institute, The University of Adelaide,
Urrbrae, South Australia 5064, Australia
4 School of Physical Sciences, The University of Adelaide, North Terrace, South Australia 5005, Australia
5 School of the Environment, Flinders University, Bedford Park, South Australia 5042 Adelaide, Australia
6 SKLEAC, Research Centre for Eco-environmental Sciences, Chinese Academy of Sciences,
P.O. Box 2871, Beijing 100085, China
7 Barbara Hardy Institute, University of South Australia, South Australia 5095, Australia
** Corresponding Author, e-mail: John.awad@mymail.unisa.edu.au*







Abstract

With significant problems caused by organics in drinking water, comprehensive knowledge of its concentration, character and reactivity in relation to catchment features and management is important. In a study reported here, six contrasting zero order catchments of the Myponga Reservoir, South Australia were selected to determine the impacts of catchment characteristics and land management practices on THM formation potential (THMFP) of aquatic DOM and the kinetics of chlorine decay. Runoff and sub-surface waters from these catchments were standardized in DOC concentration and then treated by alum coagulation. Chlorine decay tests and DOM characterization were performed and THMFP determined on standardized samples before and after alum treatment. The results showed that runoff and subsurface waters from catchments having clay soils and with native vegetation or grass, had lower THMFPs than waters from sandy soils with these vegetative types. Subsurface waters from two catchments with *Pinus radiata* had similar THMFP formation, regardless of marked variation in surface soil texture. Under standard conditions, the residual DOM in waters of the clayey soil catchments after alum treatment showed faster chlorine bulk decay rates than of the corresponding sandy soil catchments. Organics in water from clay catchments were more difficult to remove by alum coagulation.

Key words – Chlorine bulk decay; DOM; Land-use; Soil texture; THMFP

Sammanfattning

Då organiskt material kan orsaka betydande dricksvattenkvalitetsproblem, är det viktigt att ha omfattande kunskap om dess koncentration, karaktär och reaktivitet i förhållande till egenskaper och användning av avrinningsområdet. I denna studie har sex olika avrinningsområden till Myponga Reservoir i South Australia undersökts för att bedöma hur jordtyp och markanvändning inverkar på potentialen för att bilda trihalometaner (THMFP) klorsönderfallets kinetik. Ytavrinning och grundvatten från dessa avrinningsområden standardiserades med avseende på DOC-koncentration och bereddes därefter genom koagulering med aluminiumsalt. Klorsönderfallstest och DOM-karakterisering utfördes och THMFP bestämdes för standardiserade prover före och efter beredningen. Resultaten visade att ytavrinning och grundvatten från lerjordar och från marker bevuxna med ursprunglig vegetation eller gräs hade lägre THMFP än vatten från sandjordar med dessa vegetationstyper. Grundvatten från två avrinningsområden med Montereytall (Pinus radiata) hade liknande THMFP oberoende av jordtyp. Vid standardförhållanden förbrukade vatten med organiskt material från lerjordar klorresterna snabbare än vatten med organiskt material från sandjordar. Organiskt material i vatten från lerjordar var svårare att avskilja med aluminiumkoagulering.

1. Introduction

Dissolved organic matter (DOM) present in waters from catchments is influenced by land management practice, vegetation, climate and seasonality, geomorphology and soil properties (Bryan et al., 2009; Yang et al., 2013). The concentration and character of DOM significantly influences raw and treated water quality and chemical disinfectant decay and disinfection by-product (DBP) formation in treated waters (Richardson and Postigo, 2012). For drinking water disinfection, chlorine is widely used in Australia for control of growth of pathogenic micro-organisms in supply systems with short hydraulic retention times. Chlorine decay in treated bulk water is influenced by the concentration of DOM, its character, inorganic compounds, temperature and pH (Brown et al., 2011; Fisher et al., 2011). Typically, two distinct phases of decay described as fast and slow decay rates are observed (Brown et al., 2011). The fast decay rate (FDR) involves reaction between chlorine and easily oxidizable compounds and is completed within a short time of chlorination, whereas the slow decay rate (SDR) involves more resistant compounds and continues over hours and days.

Drinking water DBPs such as trihalomethanes (THM) are formed by the reaction of chlorine-based disinfectants with naturally occurring organic matter (Richardson and Postigo, 2012). THM formation in distribution systems is a function of chlorination and quality of the water being treated (Jung and Son, 2008) especially the concentration and character of organic matter. In drinking waters with high relative abundances of hydrophobic, aromatic and humic compounds there is generally greater THM formation following chlorination than in treated waters with non-aromatic, hydrophilic compounds and/or fulvic acids (Liang and Singer, 2003; Jung and Son, 2008). In addition to organic matter, physical and chemical factors also influence the formation of THM, such as bromide ion (Br) concentration in raw waters, pH and temperature (Brown et al., 2011).

A clearer understanding of the impacts of DOM derived from drinking water catchments on conventional treatment, water quality and DBP formation is needed in order to inform catchment management for improved and optimized outcomes. The aim of this study is to determine the impacts of various catchment characteristics and land management practices on the character of DOM before and after enhanced coagulation and subsequently on THM formation potential and the kinetics of chlorine decay.

2. Material and Methods

Site description

The study sites are located in catchment of Myponga Reservoir (35°22' to 35°25'S and 138°24' to 138°28'E) and comprise part of the Mount Lofty Ranges watershed, 50 km south of Adelaide, South Australia, Australia. The Myponga catchment is primarily comprised of grassland (74 %) and native vegetation (13 %), with the remaining area used for pine plantation (*Pinus radiata*), viticulture and horticulture (Bryan et al., 2009). Six zero order catchments with contrasting soil textures (sandy and/or clayey) under native vegetation, pine or grass, were selected for this study. Zero order catchments are largely non-channelised drainages common on hillslopes and referred to as hollows or swales (Dietrich et al., 1987). Land-uses, dominant vegetation, average slopes and soil descriptions of the study sites have been previously reported in detail (Awad et al., 2015). The six sites are as follows: Site 1: native vegetation on sandy soil (NV-S); Site 2: pine on sand (P-S); Site 3: grass on sand over sandy clay (G-S/SC); Site 4: native vegetation on sandy clay loam over sandy clay (NV-SCL/SC); Site 5: pine on sandy clay loam over sandy loam (P-SCL/SL); Site 6: grass on sandy clay loam over sandy clay (G-SCL/SC).

Site instrumentation

Catchments were either instrumented with surface flow barriers and auto-samplers to sample surface runoff through a Replogle-Bos-Clemmens flume at lower slope sites, as described by Fleming and Cox (2001), or with barrier sheeting (~30 m length and 12 cm height). The latter diverts surface water captured to a central location (lowest point) where the water is discharged through a polyethylene pipe (19 mm) and collected in polyethylene terephthalate (PET) bottles.

Suction cup tension lysimeters were used for sub-surface water collection in the six catchments. The lysimeters were installed at depths of approximately 60 cm to capture sub-surface flows at the B/C horizon and at various slope heights (upper, middle, and lower sites). Water collection was through application of a vacuum (~60 psi) within the lysimeter. Sub-surface water collection was also conducted using capped 90 mm diameter PVC plastic pipes (piezometers).

Water quality analyses

Water samples were collected between June and November 2013 from each catchment and from Myponga River and Reservoir within 24 hours of rainfall events (rainfall ≥ 20 mm) or about every two weeks. Samples were stored at ~4°C until analyzed.

Analyses of DOC concentrations and UV-Vis absorbances of waters

Samples for DOC and UV absorbance measurements were pre-filtered through 0.45 µm pre-rinsed sterile cellulose membrane filters. DOC concentration was determined using a TOC analyzer. UV-Vis absorption spectroscopy was used to measure DOM indirectly and as chromophoric or coloured DOM. Color was determined from the absorbance at 456 nm using 5 cm glass cuvette using a platinum/cobalt standard (50 HU). Specific UV absorbance (SUVA) values, the ratio of absorbance at 254 nm/m to DOC concentration and Specific color (SpCol), the ratio of color in HU to DOC concentration were determined to examine DOM character before and after treatment.

Fluorescence excitation-emission matrix (F-EEM)

F-EEM spectra were acquired to assess the character of DOM in terms of humic (HA), fulvic (FA), Protein1 (PI), Protein2 (PII) and soluble microbial protein (SMP) -like constituents. Emission spectra from 280–600 nm with data acquisition at 0.5 nm increments were obtained from excitation wavelengths of 200–500 nm at 5 nm increments. High purity Milli-Q water spectra subtracted from the measured sampled spectra. The method of Chen et al. (2003) was used to calculate five different EEM regions representing the five biopolymers, as detailed above.

HPSEC-UV detection

Separation of organic compounds based on apparent molecular weight (AMW) distributions was estimated using HPSEC with detection at 260 nm, as described by Chow et al. (2008). Column size, type, flow rate and other information of the HPSEC-UV detection method has been previously reported (Chow et al., 2008). Water samples for HPSEC analysis were filtered through 0.22 µm sterile cellulose membrane prior to the analysis. The weight-averaged molecular weight ($MW = \sum n^*MW / \sum n$) was calculated as described by Chow et al. (2008).

Treatment with Alum

Organics from these catchment were treated by alum, DOC concentrations in waters as collected were standardized to 8.0±1.0 mg/L by addition of high purity Milli-Q water. Standard jar tests were performed at ambient temperature and at pH 6 ±0.1 (Chow et al., 2009) using aluminium sulphate (alum) as $Al_2(SO_4)_3.18H_2O$. Very high alum dose (VHD, > 2 times the predicted enhanced dose by mEnCo@ software (van Leeuwen et al., 2005, 2009a)) was used for maximizing DOC removal. The DOC concentration and the DOM character prior to and after treatment with alum for catchment runoff and sub-surface waters are presented in Table 1.

THMFP and Chlorine decay

Trihalomethane formation potential (THMFP) was determined for standardized water samples before and after alum treatment as described by Ng et al. (2013) using a headspace GC/ECD. For testing the effect of DOM character on THM formation, Specific THMFP values, the ratio of THMFP to DOC concentration (µg THMFP/mg DOC), were determined. Bromide concentration was measured using Standard Method 4110 (Eaton et al., 1998).

Chlorine decay tests were performed on water samples before and after alum treatment under standard conditions of pH, 7.1±0.1; 23°C; DOC, 2±0.1 mg/L and chlorine dose, standardized to 4 mg/L. Total and free residual chlorine concentrations were measured at predetermined time intervals from dosing, over 10 days using Standard Method 4500 F (Eaton et al., 1998). Chlorine bulk decay coefficients (for fast and slow reacting compounds, defined based on the parallel first order decay function, Equation 1) were determined using Tablecurve© 2D Curve Fitting Software as described by van Leeuwen et al. (2009b).

$$C_t = C_0\,[f\,e^{-K_{fast}t} + (1-f)\,e^{-K_{slow}t}] \qquad \text{Eq.1}$$

Where C_t is the chlorine concentration (mg/L) at time t (h); C_0 is the initial chlorine dose (4.0 mg/L); f is the fraction of chlorine demand attributed by fast reactions; K_{fast} is fast reaction rate co-efficient (h^{-1}); and K_{slow} is slow reaction rate co-efficient (h^{-1}).

Table 1. *Average DOC concentration and DOM character prior to and after treatment by alum.*

Raw water		DOC (mg/L)	Br* (mg/L)	SUVA[†] (L/mg.m)	SpCol[†] (HU. L/mg)	MW[‡] (Da)	PI[§] (%)	PII[§] (%)	SMP[§] (%)	FA[§] (%)	HA[§] (%)
Reservoir		15.1	0.31	3.3	4.6	1310	2	14	9	46	29
River		16.2	0.38	3.6	6.5	1435	2	12	7	51	29
NV-S, S1	Runoff	81.0	0.21	4.6	8.6	2105	2	8	5	53	32
	Subsurface	118	0.18	4.6	11.4	2230	3	8	5	48	35
P-S, S2	Subsurface	96.0	0.02	3.3	4.7	1276	14	17	9	38	22
G-S/SC, S3	Runoff	55.0	0.00	4.1	11.9	2120	4	13	10	44	29
	Subsurface	61.1	0.00	4.1	8.5	1684	4	12	7	47	30
NV-SCL/ SC, S4	Runoff	67.0	0.00	3.9	11.4	2015	3	11	6	51	29
	Subsurface	29.1	0.02	3.1	4.3	1652	13	27	10	36	16
P-SCL/SL, S5	Subsurface	112	0.06	3.4	5.6	1266	2	10	7	49	32
G-SCL/SC, S6	Runoff	13.0	0.43	3.0	5.7	1275	2	12	8	45	33
	Subsurface	9.1	0.23	2.2	4.4	981	4	17	10	46	24

Treated water		DOC (mg/L)	SUVA[†] (L/mg.m)	SpCol[†] (HU.L/mg)	MW[‡] (Da)	PI[§] (%)	PII[§] (%)	SMP[§] (%)	FA[§] (%)	HA[§] (%)
Reservoir		7.9	1.9	2.0	808	9	24	12	38	17
River		7.3	1.5	2.3	796	10	24	12	38	15
NV-S, S1	Runoff	20.1	1.6	0.6	687	16	27	13	32	12
	Subsurface	28.8	1.4	3.3	774	18	24	17	32	9
P-S, S2	Subsurface	52.4	0.9	3.3	711	21	27	11	32	9
G-S/SC, S3	Runoff	22.0	1.2	2.1	656	14	28	13	35	10
	Subsurface	25.9	1.3	3.4	700	19	23	11	36	11
NV-SCL/ SC, S4	Runoff	21.4	1.5	1.2	643	12	21	12	39	16
	Subsurface	23.0	0.9	1.0	750	16	24	14	33	14
P-SCL/SL, S5	Subsurface	46.9	1.4	1.5	784	15	27	14	32	13
G-SCL/SC, S6	Runoff	4.9	2.1	2.5	754	11	21	11	39	18
	Subsurface	6.9	2.1	2.9	758	8	22	11	42	18

* Average bromide ion concentration in raw waters

[†] Average SUVA, specific UV absorbance; SpCol, Specific color

[‡] Average of Weight-Averaged Molecular Weight identified by HPSEC-UV detection

[§] Average percentage abundances of organic components identified by F-EEM

3. Results and Discussion

Specific THMFP prior to and after alum treatment

Specific THMFP (µg/mg DOC) prior to and after treatment by enhanced coagulation are presented in Figure 1. Runoff water samples collected from native vegetation and grass sites had DOM of higher Specific THMFP (average: +20.5±11 %) than samples from reservoir water (Figure 1a). DOM present in reservoirs can comprise organics from two different sources, allochthonous DOM from soil and catchment sources and autochthonous DOM from microbial activities in the water body. Autochthonous compounds are likely to produce relatively less THMs levels than allochthonous compounds (Hong et al., 2013).

Raw waters from zero catchments with native vegetation on sandy soil (NV-S, S1) had DOM of higher Specific THMFP (+39 %) than organics of waters from clay soils (NV-SCL/SC, S4). The significantly higher Specific THMFP in waters of S1 corresponds with the high relative abundance of organics of HMW, aromatic and humic-like compounds (Table 1). Grass catchments showed the same trend, with Specific THMFP concentrations considerably higher in water from S3: G-S/SC (~47.4±5.9 µg/mg DOC) compared to S6: G-SCL/SC (~38.1±4.7 µg/mg DOC). The G-SCL/SC site has soil with higher clay content (mean 22±6.1 %) compared with the G-S/SC site (mean 15±13 %).

For the two *Pinus radiata* sites, despite the P-SCL/SL site (S5), having a higher percentage of clay compared with P-S (S2), (i.e. P-S: 5.5±2.3 % vs P-SCL/SL: 19.7±2.5 %), the Specific THMFP from the P-SCL/SL site (mean 36.4 µg/mg DOC) was similar to that from P-S (mean 39.1 µg/mg DOC), Figure 1a.

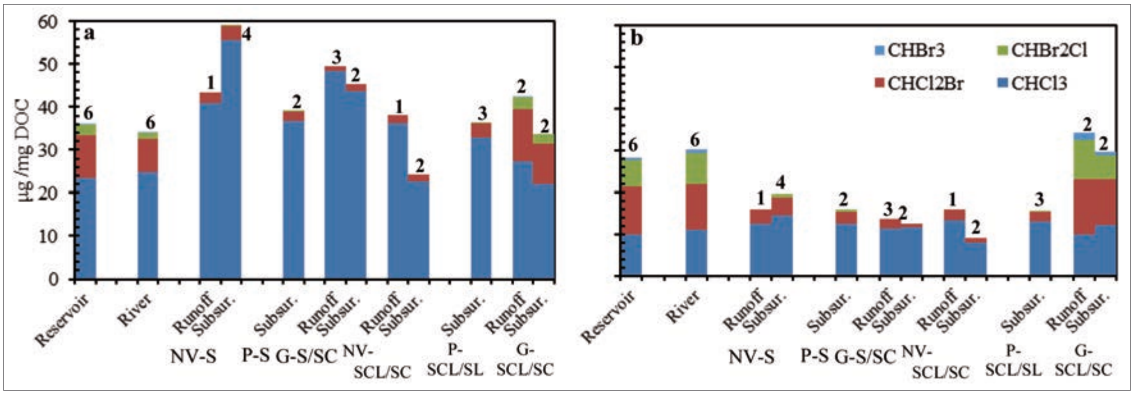

Figure 1. *Average Specific THMFP for (a) raw water and (b) treated waters. Values above bars represent the number of samples analysed from each site/depth.*

Following coagulation under standardized jar test conditions, residual DOM present in treated reservoir water had a higher Specific THMFP than DOM in waters from all of the investigated catchment sites except the G-SCL/SC site (for which Specific THMFP was similar to that from reservoir waters, Figure 1b). Specific THMFP levels in the flows under NV-S (S1) were similar to those of NV-SCL/SC (S4). Also, for the two *Pinus radiata* sites, the Specific THMFP from the P-SCL/SL (S5) site was very similar to P-S (S2) site, Figure 1b. In contrast, there were marked differences in the Specific THMFP concentrations between the two grassed sites with the clayey site values being much higher than that of the sandy site (Figure 1b).

The G-SCL/SC site, had the highest Br concentrations (average; 0.33±0.1 mg/L) and the highest remaining relative abundance of aromatic and humic-like organic compounds (SUVA: 2.1±0.04, SpCol: 2.7±0.6, HA-like: 18±3 % and FA-like: 41±2 %) after treatment by enhanced coagulation. These factors led to high levels of Specific THMFP produced from organics present in waters from this catchment.

Comparison of before and after standardized jar tests indicated that aromatic, HMW and humic-like DOM, resulted in higher Specific THMFP formation than is the case for LMW and protein-like DOM. The relative abundances of THM bromide species were different between raw and treated water with significantly lower potential in raw water than treated water (Figure 1). The relative abundances of chloroform ($CHCl_3$) for raw and treated waters were 86±13 % and 68±22 % respectively. Bromide concentration had a lower effect on THMFP levels in raw waters than in treated waters.

Chlorine decay coefficients prior to and after alum treatment

Chlorine bulk decay coefficients for fast and slow reacting compounds, prior to and after treatment by enhanced coagulation are presented in Figure 2. Runoff

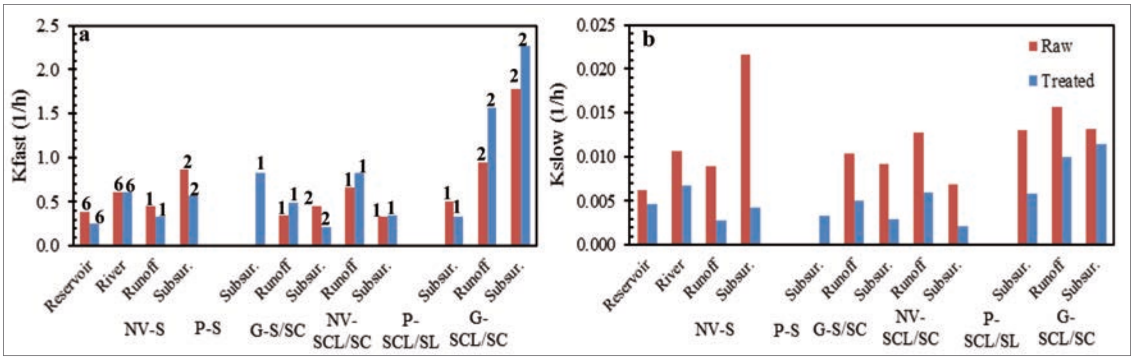

Figure 2. *Average chlorine bulk decay (1/h) under the standard condition, a) Fast decay and b) Slow decay. Values above bars represent the number of samples analysed from each site/depth.*

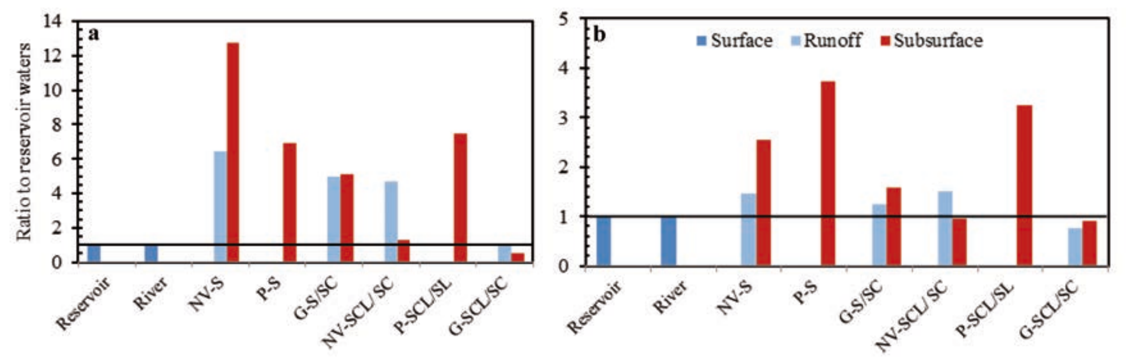

Figure 3. *The ratios of THMFP concentrations (a) prior to and (b) after treatment with alum of catchment runoff and sub-surface waters, to reservoir water.*

water samples collected from catchments with native vegetation and grass and from the river were found to have higher initial chlorine decay rates (K_{fast}; average: +57.2±53 %) than samples from reservoir waters (Figure 2a). Consistent with that, the slow reaction (>2 h, K_{slow}) coefficients for DOM from runoff and river water samples were higher than of the DOM from reservoirs water (for river water, +73 %; for sandy catchments, +56 %; and for clayey catchments, +130 %).

For native vegetation catchments, the K_{slow} value for DOM from catchments with sandy soil (NV-S, S1) were much higher than that for DOM from clay soils (NV-SCL/SC, S4), shown in Figure 2b. The highest K_{slow} chlorine decay rate was of DOM in raw water from the native vegetation-sand site (having the highest relative abundance of aromatic, humic-like and HMW organics). In contrast, for grass catchments the K_{slow} was considerably higher for G-SCL/SC (~0.0145 h^{-1}) than G-S/SC (~0.01 h^{-1}). This may be attributed to marked differences in the K_{fast} between the two catchments where the K_{fast} for G-SCL/SC (S6) site (~1.36 h^{-1}) was much higher than that for the G-S/SC (S3) site (~0.40 h^{-1}). The initial fast reaction (in the first 2 h of chlorination) effects the chlorine concentration (residual chlorine after the first 2 h) for the slow reaction and consequently, on the slow bulk decay coefficient for the slow reaction. This is similar to that found by Jabari Kohpaei and Sathasivan (2011) who reported that the residual chlorine after the fast reaction affects the slow chlorine decay process.

After treatment by alum, the K_{slow} of DOM from waters of the sandy soil catchments were lower than of the corresponding clayey soil catchments, as shown in Figure 2b. Differences in K_{slow} were found to be greater for DOM from grass catchments (–63 %) compared with other catchments (–44 % for pine and –14 % for native vegetation). In contrast, as with the Specific THMFP concentrations for two *Pinus radiata* sites (P-S, S2 and P-SCL/SL, S5), the K_{slow} was found to be greater for DOM from P-SCL/SL, S5 (0.006 h^{-1}) than from P-S, S2 (0.003 h^{-1}).

For treated water, the lowest average K_{fast} (~0.52±0.2 h^{-1}) and K_{slow} (~ 0.004±0.002 h^{-1}) values were recorded for the native vegetation catchments. This was followed by the *Pinus radiata* (mean ~0.58± 0.24 h^{-1} for K_{fast} and ~0.005±0.001 h^{-1} for K_{slow}) and the grass catchments (mean ~1.14±0.8 h^{-1} for K_{fast} and ~0.007±0.004 h^{-1} for K_{slow}), see Figure 2.

Comparison of Specific K_{slow} data of DOM in waters before and after standardized jar tests indicated that (similarly as Specific THMFP) DOM of HMW, aromatic and humic-like compounds had higher chlorine decay than LMW, protein-like DOM.

THMFP prior to and after alum treatment

For raw waters, the THMFPs of catchment runoff flows ranged from 550 µg/L to 3510 µg/L and for sub-surface flows, from 305 µg/L to 6930 µg/L. After treatment by enhanced alum coagulation, the THMFPs of catchment runoff waters ranged from 170 µg/L to 340 µg/L and for sub-surface samples, from 205 µg/L to 835 µg/L. The average THMFPs of reservoir water prior to and after treatment with alum (542±21 µg/L and 224±7 µg/L, respectively) were used as baseline values to compare against those of the various catchment sites (Figure 3).

For raw waters from sandy soil catchments, the highest THMFP relative to reservoir water was from the native vegetation catchment (NV-S: 6.5:1 for runoff and 12.5:1 for subsurface flow), followed by *Pinus radiata* (P-S: 6.9:1 for subsurface flow), and then the grass site (G-S/SC: 5.0:1 for runoff and 5.1:1 for subsurface flow). For clay soil catchments, the highest ratio was from *Pinus radiata* (P-SCL/SL: 7.5:1 for subsurface flow), followed by native vegetation (NV-SCL/SC: 4.7:1 for runoff and 1.3:1 for subsurface flow) and then grass site (G-SCL/SC: 1.0:1 for runoff and 0.6:1 for subsurface flow).

For treated waters, there were marked differences in the THMFP concentrations between the two grass sites with the sandy site values being much higher than that of the clayey site. Native vegetation catchments showed the same trend with the THMFPs considerably higher for NV-S:S1 compared to NV-SCL/SC:S4. In contrast, no distinct differences were found between THMFPs of the two pine sites. This appears to be due to a lack of overall impact of clay soil on *Pinus radiata* sites, in the presence of very high loadings of litter layers (P-S: 5.1±2.1 cm; P-SCL/SL : 7.4±3.1 cm average measurements). It is suggested that the high litter layer loadings lead to high levels of DOM being released that exceed the adsorption capacity of the clay soils. Similarly, Hassink (1997) reported that there was a limit in the amount of organic carbon and nitrogen that could be absorbed by clay or silt particles of grassland and arable soils. Recent studies (Saidy et al., 2012, 2015) have reported the effect of different clay minerals on the susceptibility of plant-derived organic carbon to microbial degradation. According to these studies, when the organics loading exceeded the sorption capacity of minerals, the minerals had very low influence on the microbial degradation of organic materials studied (DOM of *Medicago truncatula cv Praggio*). However, when the amount of DOM was less than the sorption capacity of minerals, the DOM was more resistant to microbial degradation, presumably through organo-mineral formations that protected the organic compounds.

The grass on clay (G-SCL/SC) site generally had flow with the lowest DOM loading (Table 1) and consequently showed the lowest THMFP (Figure 3b), (the average ratios of THMFPs to reservoir water after treatment with alum, of runoff and subsurface waters were 0.76:1 and 0.92:1 respectively). The data presented shows that both soil texture and vegetation loading and type have major impacts on the quality of source waters used for drinking water supply. Highest quality was of waters from clay soil catchments vegetated with grasses, while the lowest quality was from catchments under pine plantation.

4. Conclusion

Catchment properties, including vegetation type and soil texture, have a significant influence on the quality of waters for drinking water supply. In determination of the THMFP from DOM present in water samples from the catchments studied, potentials were highest in waters from pine, followed by native vegetation and then grass catchments. Clay soils influenced the character and concentration of organics present in surface runoff and sub-surface waters from catchments under native vegetation and grass. Residual organics in the water phase from clay soil sites result in lower THMs than that from sandy soil sites. Loadings of organic compounds in sub-surface waters under pine catchments were minimally influenced by the soil textures studied and consequently were found to not influence specific THMFP.

Based on the character of DOM remaining in treated waters after enhanced coagulation, chlorine bulk decay rates from DOM in waters of the clayey soil catchments were faster than of the corresponding sandy soil catchments. Organics from grassland catchments have relatively fastest chlorine bulk decay rates.

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