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Evaluation of Trihalomethanes Formation Using Combined Process Coagulation/flocculation/membranes in Water Treatment

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The objective of this study was to evaluate the efficiency of the combined process coagulation/flocculation followed by ultrafiltration and a chlorination step using natural coagulant Moringa oleifera Lam in replacement to conventional treatment on the THM formation. Assays were carried out using Pirapó River Basin raw water, low turbidity (50 NTU). Coagulation/flocculation assays were initially performed in Jar-test using saline solution (NaCl – 1M) from M. oleifera seeds (MO) as coagulant with dosages range from 10 to 60 mg.L^{-1} and polyaluminum chloride (PAC) as chemical coagulant with dosages range from 7 to 12 mg.L^{-1}. In membrane filtration step, ultrafiltration polyethersulfone membrane was used with 1bar operation pressure. Chlorination step was performed with sodium hypochlorite (1.5 mg.L^{-1}) during 30 min to 8 h. The parameters analyzed were color, turbidity, UV_{254nm}, dissolved organic carbon (DOC), total trihalomethanes (TTHM) and free chlorine. TTHM produced was evaluated by gas chromatography. The optimal dosage for PAC and MO coagulants were 9.5 and 50 mg.L^{-1}. After the chlorination step, TTHM residual increased lower using MO (9.3 μg.L^{-1}) than PAC (32 μg.L^{-1}), remaining in according to legislation (100 μg.L^{-1}). The coagulation/flocculation pre-treatment for both coagulants minimized the fouling formation in UF process.

1. Introduction

Despite the benefits derived from the disinfection, the use of chlorine and other compounds have received attention from the scientific community, due to their reactions with natural organic matter (NOM) from the surface waters, which can form disinfection byproducts undesirable human health as trihalomethanes (chloroform, bromodichloromethane, dibromochloromethane and bromoform), haloacetic acids (HAA), and haloacetonitrilas (HAN), among others (Hong et al., 2007).

With high carcinogenic and mutagenic potential (Gopal et al., 2007), studies are needed of alternative water treatments capable of removing/reducing the amount of total trihalomethanes (TTHM) formed. The best alternative considering the facility of working with the same disinfectant and high costs in removing THM after formed is the removal of NOM before application of chlorine (Chen et al., 2008).

Despite the performance and cost-effectiveness proven of chemical coagulants, mainly aluminum sulphate and polialuminum chloride (PAC), there is still a degree of residual aluminum content after treatment, which has been linked to Alzheimer's disease. Moreover, aluminum is not biodegradable and can cause problems of disposal and treatment of the large amount of sludge generated (Teh and Wu, 2014). Due to many problems created by using the synthetic coagulants, there is a high demand to find an alternative coagulant which is preferable to be natural. In various countries, many plants and its derivatives are used as coagulants/flocculants natural biopolymers, which some have been investigated most intensively than others, as is the case of Moringa oleifera Lam (M. oleifera) (Teh and Wu, 2014; Camacho et al., 2013).

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M. oleifera seeds can be used as a primary coagulant in drinking water clarification and wastewater treatment due to the presence of a water-soluble cationic peptides with molecular masses that range from 6 to 16 kDa that is able to reduce turbidity of the water treated (92 – 99 % of turbidity reduction) (Pritchard et al., 2010; Ndabigengesere and Narasiah, 1998). Water treated with M. oleifera seed extract produces less sludge volume compared to alum (Ndabigengesere and Narasiah, 1998). In this context, this study aims to evaluate the efficiency of the combined process coagulation/flocculation followed by ultrafiltration and a chlorination step using natural coagulant Moringa oleifera Lam in replacement to conventional treatment on the THM formation.

2. Materials and Methods

2.1 Coagulation/flocculation tests

For the tests, raw surface water was obtained from Pirapó river, in Maringá (North of Paraná State – Brazil) with characteristics of low color/turbidity and constant pH. The coagulants used for the tests was M. oleifera seeds prepared in saline solution using commercial salt NaCl (MO) and polialuminum chloride (PAC).
For MO coagulant preparation, mature seeds of M. oleifera were used, from the Federal University of Sergipe (UFS, Brazil), manually removed from the pod and shelled dry. One gram of peeled seeds were weighed and crushed with 100 mL of saline solution (NaCl - 1M) in a blender. Subsequently, the solution was stirred for 30 min and filtered under vacuum on membranes of 0.45 µm, obtaining a solution 1.0% m/v of MO seeds (Camacho et al., 2013; Nishi et al., 2011). The dosage range investigated during the tests was 10 to 60 mg.L-1. For PAC coagulant preparation, the standard solution was prepared 1.0 % v/v, adding 1 mL of PAC in 100 mL of distilled water at room temperature. The dosage range investigated during the tests was 7.0 to 12 mg.L-1, according to the recommendations of local water treatment plant, SANEPAR (Maringá/PR – Brazil).
Experimental tests were conducted in jar-test equipment Nova Ética - Model 218 LDB with six samples of raw water (500 mL) simultaneously in order to evaluate the optimal coagulant dosage. The conditions used in each test jar-coagulant are shown in Table 1.

Table 1 – Operational conditions of jar-test

Parameters	PAC	MO
Rapid mixing velocity(rpm)	110	100
Rapid mixing time (min)	1.0	3.0
Slow mixing velocity (rpm)	45	15
Slow mixing time (min)	15	15
Settling time (min)	15	60

The parameters apparent color, turbidity and compounds with absorption in UV-254 nm have been measured according to Standard Methods (APHA, 2005). Each experiment was performed in duplicate. The data obtained of the tests were analysed by statistical analysis (ANOVA and Tukey's test) using the software Statistica 6.0 to determine the optimal dosage of each coagulant.

2.2 Ultrafiltration and disinfection tests

In the membranes filtration step, it was used ultrafiltration membrane modules of poly(ether sulfone) with hydrophilic character and 50 kDa molecular weight cut-off, hollow-fibers form produced by the company PAM-Selective Membranes (COPPE-UFRJ, Rio de Janeiro-RJ). The operating pressure used was 1 bar. To measure NOM removal, the parameters apparent colour, turbidity, compounds with absorption in UV-254 nm and dissolved organic carbon (DOC) of the filtrate has been measured (APHA, 2005). Each experiment was performed in duplicate. These tests were conducted to verify the influence of pretreatment coagulation/flocculation using different coagulants (PAC and MO) in membrane performance (NOM removal, TTHM formation and fouling). The data was analysed by statistical analysis (ANOVA and Tukey's test) using software Statistica 6.0.
The disinfection of filtered water was performed with sodium hypochlorite in flasks of 500 mL with aluminum foil involved with finality to absence the light and minimizing the degradation of chlorine. It was applied a concentration of 1.5 $mg.L^{-1}$, which is used by local water treatment station (SANEPAR) in contact times of 30 min, 1, 2, 4 and 8 h. To cease the reaction of residual chlorine with organic matter in each contact time, the samples were supplemented with sequestrant free residual chlorine (sodium thiosulfate solution ($Na_2S_2O_3$)).

3. Results

The characterization of the raw water in terms of parameters related to the organic matter is shown in Table 2.

Table 2 – Raw water characterization

Parameters	Mean Values
Turbidity (NTU)	56.4 ± 5.6
Apparent colour (uH)	262 ± 22.4
pH	7.5 ± 0.71
UV_{254nm} (cm^{-1})	0.230 ± 0.08
DOC ($mg.L^{-1}$)	4.455 ± 0.77
TTHM ($\mu g.L^{-1}$)	4.31 ± 0.49

Due to the fact that most of the raw water from the treatment plant site was characterized to be of low colour/turbidity, due to long periods of drought and difficult to treat, water with these characteristics was used in this study.

3.1 Determination of optimal dosage of PAC coagulant dosage in coagulation/flocculation tests

The mean of the residual and removal efficiencies for parameters colour, turbidity and UV_{254nm} with PAC coagulant tests are shown in Table 3.

Table 3 - Means of residual and removal efficiencies to determine the optimal dosage of PAC coagulant

Dosage ($mg.L^{-1}$)	Residual Turbidity (NTU)	Turbidity Removal (%)	Residual Colour (uH)	Colour Removal (%)	Residual UV_{254nm} (cm^{-1})	UV_{254nm} Removal (%)
7.0	7.55 ± 0.19	87 (a)	60 ± 4.24	81 (a)	0.074 ± 0.004	67 (a)
8.0	6.46 ± 0.04	89 (a)	53 ± 2.83	83 (a)	0.058 ± 0.005	75 (b)
9.0	5.13 ± 0.33	91 (b)	48 ± 0.00	87 (b)	0.050 ± 0.003	78 (bc)
9.5	3.94 ± 0.19	93 (c)	41 ± 7.78	90 (bc)	0.039 ± 0.002	83 (c)
10.0	3.59 ± 0.54	94 (cd)	36 ± 1.41	91 (cd)	0.040 ± 0.005	82 (c)
11.0	2.72 ± 0.35	95 (d)	32 ± 5.66	92 (cd)	0.040 ± 0.001	82 (c)
12.0	2.75 ± 0.07	95 (d)	30 ± 0.00	94 (d)	0.038 ± 0.003	83 (c)

The letters (a, b, c and d) in each column of the table identifies different statistics groups for each parameter analysed (Tukey test, $p < 0.05$), and the results of residual represented by mean ± standard deviation.

In general, analysing each parameter, high removals of turbidity (> 90 %), colour (> 90 %) and UV_{254nm} (> 80 %) were observed using dosages above 9.0 $mg.L^{-1}$, obtaining residuals lower than 5.0 NTU for turbidity, 40 uH for colour and 0.090 cm^{-1} for UV_{254nm}, with the turbidity and colour within the current legislation (Brazil, 2011). Regarding parameters removal, statistical analysis indicates that the best dosages were in the range 10 - 12 mg L^{-1} for colour and turbidity removals and 9.5 to 12 $mg.L^{-1}$ for UV_{254nm} removal. According local water treatment plant (SANEPAR), to obtain a long filtration career and a filtrate of good quality, the turbidity after coagulation/flocculation step must be below 5.0 NTU. The residuals of turbidity, colour and UV_{254nm} parameters showed that when using a dosage of 9.5 mg L^{-1}, turbidity residual was below 5.0 NTU, choosing this value the optimum dosage for PAC coagulant. When compared to the results of Yarahmadi et al. (2009), which also evaluating the effectiveness of the PAC coagulant in the coagulation/flocculation process with raw water turbidity of 50 NTU, an optimal dosage of 20 $mg.L^{-1}$ reduced the turbidity of 50 NTU to 4.5 with turbidity removal of 89 %. Thus there was obtained a much higher dosage than obtained in this study (9.5 mg L^{-1}), since the raw water used by the authors was synthetic prepared with kaolin and different jar-test operating conditions were used.

3.2 Determination of optimal dosage of MO coagulant dosage in coagulation/flocculation tests

The mean of the residual and removal efficiencies for parameters color, turbidity and UV_{254nm} with MO coagulant tests are shown in Table 4. According to statistical analysis, the interaction between variables is clear, not only from the table data analyses, but also because of the ANOVA report which gives a p-value for interaction lower than 0.05 in each case. There was no statistical difference in dosages 50 - 60 $mg.L^{-1}$ for turbidity and UV_{254nm} and 50 $mg.L^{-1}$ for colour parameter, choosing 50 $mg.L^{-1}$ as the optimum dosage for MO coagulant. Many studies with M.oleifera as primary coagulant have demonstrated high removal efficiency

(Camacho et al., 2013; Nishi et al., 2011). Mangale Sapana et al. (2012) have also obtained an optimal dosage of 50 $mg.L^{-1}$ for M. oleifera powder using a low turbidity water (<50 NTU) resulting in a final turbidity lower than 5 NTU.

Table 4 - Means of residual and removal efficiencies to determine the optimal dosage of MO coagulant

Dosage ($mg.L^{-1}$)	Residual Turbidity (NTU)	Turbidity Removal (%)	Residual Colour (uH)	Colour Removal (%)	Residual UV_{254nm} (cm^{-1})	UV_{254nm} Removal (%)
10	20.6 ± 0.85	64 (a)	119 ± 2.12	62 (a)	0.107 ± 0.001	52 (a)
20	14.2 ± 0.49	75 (b)	113 ± 3.54	64 (a)	0.096 ± 0.006	57 (ab)
30	11.4 ± 0.41	80 (c)	94 ± 2.83	70 (b)	0.107 ± 0.004	52 (a)
40	9.58 ± 0.18	83 (d)	84 ± 0.00	73 (c)	0.097 ±0.006	56 (a)
50	8.06 ± 0.28	86 (e)	35 ± 1.41	89 (e)	0.073 ± 0.004	67 (c)
60	8.88 ± 0.32	85 (de)	74 ± 2.83	76 (d)	0.084 ± 0.004	62 (bc)

The letters (a, b, c, d and e) in each column of the table identifies different statistics groups for each parameter analysed (Tukey test, $p < 0.05$), and the results of residual represented by mean ± standard deviation.

Several studies have shown that the use of salt water is more efficient as solvent than e.g. distilled water in Moringa oleifera coagulant preparation. Okuda et al. (1999) showed for example that the crude coagulation capacity was up to 7.4 times higher when the active agents were extracted with salty water as compared to distilled water. The reason for this is assumed to be that the coagulating protein is more soluble in water with high concentration of ions (Okuda et al., 2001). This improves efficiency by salt extraction was described by a salting-out mechanism, which increased the ionic strength and the solubility of the active components (Okuda et al., 2001; Ndabigengesere and Narasiah, 1998).

3.3 Ultrafiltration tests

With the optimal dosage of PAC and MO coagulants determined in the previous step (9.5 $mg.L^{-1}$ for PAC and 50 $mg.L^{-1}$ for MO), the coagulation/flocculation with subsequent UF in order to compare the efficiency of NOM removal was performed. The removal of colour parameters, turbidity, DOC and UV_{254nm} these tests are shown in Table 5.

Table 5 - Means of removal efficiencies of coagulation/flocculation/UF process

Sample	Colour Removal (%)	Turbidity Removal (%)	UV_{254nm} Removal (%)	DOC Removal (%)	Fouling (%)
Control*	100 (a)	99.5 (a)	86.3 (a)	64.2 (a)	24.7 (a)
PAC	100 (a)	99.6 (a)	93.0 (c)	69.4 (b)	13.5 (b)
MO	100 (a)	99.6 (a)	88.5 (b)	74.6 (c)	9.2 (c)

*raw water without pretreatment (coagulation/flocculation)

Statistical analysis reveals that use of the coagulation/ flocculation as UF pre-treatment increased the NOM removal, especially when UV_{254nm} and DOC parameters were evaluated. Despite NOM removal efficiencies in the UF process with and without pretreatment were similar, the fouling formed without pretreatment (control - 24.7 %) was much higher when compared with coagulation/flocculation as pretreatment with PAC (13.5 %) and MO (9.2 %), i.e. coagulation/flocculation was able to remove the hydrophobic fraction that is mainly responsible for the formation of fouling. This small fouling of UF membrane may be due to the hydrophobicity of the membrane. As hydrophilic, i.e., has a negative charge on its surface, can have a greater removal NOM and less fouling due to cake formation, which not only increases the retention capacity of the membrane, but also prevents the particles that cause obstruction reach membrane pores (Jung et al., 2006).

Although it contains greater amount of NOM in feed, the coagulant which showed the lower fouling was MO coagulant. This is probably due to the fact that with smaller pores, UF membrane do not suffered internal pore clogging by particles in the water or, if suffered was a lesser extent when compared to PAC coagulant. Franco Silva and Paterniani (2012), evaluating membranes filtration with and without pretreatment (coagulation/flocculation using as a coagulant M. oleifera), observed that the filtration with pretreatment had higher removal efficiency (89 % - turbidity and 86 % - colour) compared with the filtration without pretreatment (62 % - colour and turbidity).

3.4 Post-chlorination and THM formation

After chlorination process of filtrated water with UF process, it was measured the TTHM formation and residual chlorine in water treated, which is presented in Figure 1.

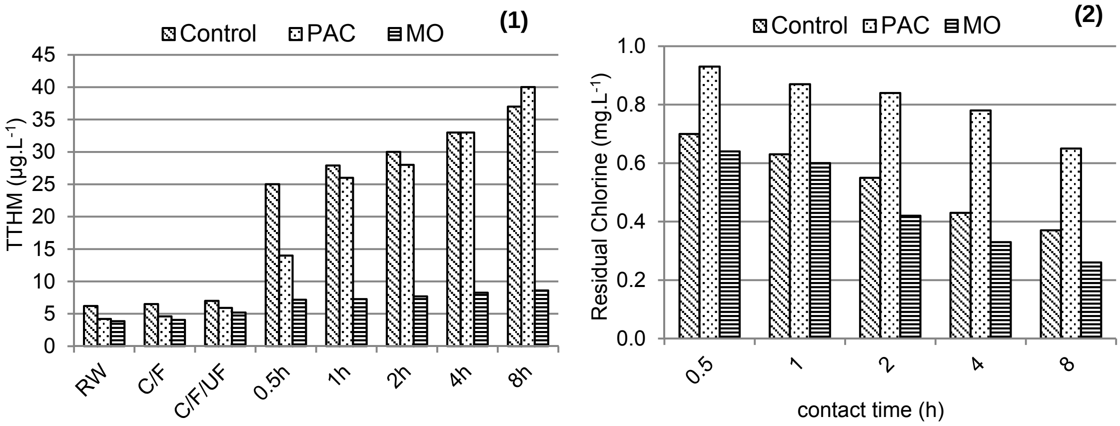

Figure 1: (1) TTHM and (2) residual chlorine for coagulation/flocculation/filtration process using PAC and MO as coagulants before (RW – raw water; C/F – coagulation/flocculation; C/F/UF – coagulation/flocculation/ultrafiltration) and after chlorination in contact times of 0.5, 1.0, 2.0, 4.0 and 8.0 h. Control: raw water without pretreatment (coagulation/flocculation)

According Ordinance nº 2914/2011 (Brazil, 2011), it is required to maintain at least 0.2 mg.L^{-1} of free chlorine residual entire length of the distribution system (reservoir and network), as can be observed after contact time of 8 h in Figure 1(2). However, it can be observed that the amount of TTHM increased with increasing contact time, with values in accordance with Ordinance nº 2914/2011 (100 µg.L^{-1}) (Brazil, 2011). The TTHM concentrations after post-chlorination were similar to that observed by Rojas et al. (2011).
Even if the values of TTHM are within the current legislation Ordinance nº 2914/2011 (Brazil, 2011) that is 100 µ.L^{-1}, it should be noted that in other countries, such as Italy, legislation is more stringent as the maximum concentration of trihalomethanes in tap water, with a maximum of 30 µ.L^{-1}. If it is considered this lower limit of TTHM, the combined process of C/F/UF was in accordance with laws more vigorous. Reductions in the amount of TTHM formed were proportional to reductions in DOC and UV_{254nm}, especially when MO coagulant was used, as observed by Lee et al. (2005). Andreola et al. (2010) evaluated the formation of TTHM in the water treatment station of Maringá/Brazil (SANEPAR), through the distribution network and terminating in household containers and related to the amount of TTHM formed with the quantities of organic matter throughout the process using conventional filtration. From the results obtained, the formation of TTHM in water treatment station occurs progressively over the sampling points, and in the raw water, as there is no chlorine in contact, there is no formation of TTHM. The values of residual chlorine (> 0.76 mg.L^{-1}) and TTHM (>50 µg.L^{-1}) output of treated water after contact tanks were in accordance with those established by Ordinance nº 2914/2011 (Brazil, 2011), which TTHM was higher than obtained in this study. Therefore, in order to provide higher NOM removal and minimization of TTHM formation, UF membranes must be used, since conventional filtration cannot eliminate part of NOM hydrophilic (low molecular weight), even using coagulation/flocculation as pretreatment (Chen et al., 2008).

4. Conclusions

Among the results, statistical analysis indicates that different dosages for PAC and MO coagulant are statistically different with optimum dosages of 9.5 and 50 mg.L^{-1}. After the chlorination step, TTHM residual increased lower using MO (9.3 µg.L^{-1}) than PAC (32 µg.L^{-1}), remaining in according to legislation (100 µg.L^{-1}). The coagulation/flocculation pre-treatment for both coagulants minimized the fouling formation in UF process, mainly when MO is used as coagulant (9.2 %). Due to the fact that Moringa oleifera is natural (produce less sludge, doesn't alter the pH, etc) compared to PAC is an alternative of utmost importance, mainly when used with the combined system coagulation/flocculation/UF which facilitates the elimination of NOM from water and reduce TTHM formation. At the same time, the clogging of the membrane is avoided since the cake formed on the membrane can be easily dispersed through backwash.

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