Impact of coagulant type and magnetic ion exchange (MIEX) pre-treatment on floc strength and structure at the Kluizen Water Treatment Plant, Belgium

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Article Info

Received date: on: September 14, 2019

Accepted in revised form: November 21, 2019

ABSTRACT

Drinking water supplied to the community should be free from Disinfection By-Products (DPB). DPB occurs when organic matter is present in surface water and reacts with chlorine. Conventional coagulation and flocculation processes have been used in water treatment plants to remove organic matters from raw water. Recently, Magnetic Ion Exchange (MIEX) followed by coagulation and flocculation has started to attract attention since it improves natural organic matter removal and decreases coagulant demand. The efficiency of NOM removal and downstream processes not only depends on the prior removal process of organic matter but also on the structure , size and strength of flocs formed in the coagulation process as well. This study aims at comparing (1) floc structures (i.e. floc sizes, floc strength, and fractal dimensions) generated from coagulation of raw water and (2) water pre-treated with magnetic ion exchange obtained from Kluizen water treatment plant (Kluizen, East-Flanders, Belgium). The floc sizes during growth, breakage and re-growth were measured using (laser) light scattering (Malvern Mastersizer 2000), coupled with jar test equipment. The strength of flocs was evaluated by empirically relating the floc size to the increased shear rate applied. The results show that pre-treated water coagulated with all the coagulants produces flocs with larger sizes compared to the flocs generated from raw water. For both water types, $FeCl_3$ gave better recoverability after breakage than PACl and $AlCl_3$. After pre-treatment, the floc size was increased by 19%, 63% and 64% for the coagulants such as PACl, $AlCl_3$, and $FeCl_3$, respectively. Coagulation process with PACl generated flocs with higher fractal dimension and lower strength constants which indicates that flocs generated is strong and compacted micro-flocs. The floc size measurement techniques and coagulation system were found to have an impact on the resulting floc sizes.

Keywords: Coagulation; floc size; jar test; magnetic ion exchange; natural organic matter; water

1. INTRODUCTION

Raw surface water often contains dissolved and Natural Organic Matter (NOM) which deteriorates water quality. The presence of NOM in water by itself is not harmful but it may have adverse effect on human health (Singer & Bilyk, 2002; Burton, 2009; Drikas *et al*., 2011). In many surface water treatment plants for potable water production, coagulation and flocculation processes are used as an essential step to get rid of NOM (Jarvis *et al*., 2004; Yukselen & Gregory, 2004; Vahedi & Gorczyca, 2011). According to Jarvis et al. (2005b) coagulation is a process of removing natural organic matter and colloids by particle destabilization, double layer compression or charge neutralization. It is the process of destabilizing organic matter by neutralizing negative charge and aggregating destabilized particles into flocs which are removed by sedimentation and/floatation (García, 2005). Better understanding of factors such as the influence of the water type (i.e., ionic balance, pH, temperature, etc.) and simple fractionation of NOM into hydrophobic and hydrophilic fractions helps enhance the coagulation and flocculation process. Thus, it is important to study properties of flocs and factors affecting their strength and structure to make efficient removal of organic matter by the process of sedimentation.

In this study, floc characteristics and mechanisms affecting floc strength and structure were assessed. The research focused on the impact of coagulant type and magnetic ion exchange (MIEX) pre-treatment of raw water on floc structure, strength and re-growth potential. The raw and pre-treated water with MIEX is taken from Kluizen drinking water treatment plant, operated by De Water groep in Flanders, Belgium.

2. MATERIALS AND METHODS

2.1. Study area

The study site was situated in Flanders, Belgium at Kluizen Drinking Water Treatment plant. Besides, two water types were drawn from the site to run the experiment. All the experiments were carried out in the laboratory at a temperature 20±2°C.

2.2. Sample collection

The first sample was raw water taken directly from the reservoir of Kluizen Drinking Water Treatment plant; however, the second sample was the same raw water but pre-treated with Magnetic ion exchange resin. The MIEX pre-treatment was carried out in a pilot plant at the Kluizen site. The pilot plant was started in September 2013 by adding 625 liter of virgin resin (Purolite) to the reactor. Purolite is strong base anion exchange with a porous structure which contributes to the absorption of organic matter. The water contacted with a resin for 1.5 minutes at a flow rate of 25m3/hr through the reactor. The reactor was made to generate 25 liter of resin with 10 % of NaCl-solution every 3 to 4 days. After treatment of 1000 m^3 of raw water, 2 liters of new virgin resin was added to the reactor to replace the exhausted part of the resin.

Table 1: Physico-chemical characteristics of raw water and pre-treated water

Parameter	**Raw water**	**Pre-treated water**
pH	8.16	7.57
UVabsorbanceat254nm(m^{-1})	28.34	11.51
Turbidity (NTU)	2.98	3.06
Conductivity(µS/cm)	647.29	663.93
Color (mg/l Pt-Co)	63.64	38.29
DOC(mg/l)	11.21	6.34
SUVA(m^{-1}mg/l)	2.53	1.81

Note: SUVA= Specific ultra-violet absorbance (L m-1 mg-1) which is the ratio of UV absorbance at 254 nm (m-1) to DOC (mg/l)

2.3. Analytical Techniques

The experiments were carried out using liquid coagulants obtained from Kluizen water treatment plant. The following coagulants were tested: Aluminum chloride ($AlCl_3$), Polyaluminum chloride (PACl) and Ferric chloride ($FeCl_3$). All coagulation experiments were conducted at corrected pH of 6.8 adjusted by adding one molar (M) of sulphuric acid (H_2SO_4). After pH regulation, coagulation experiments were performed with 1L measuring beakers filled with 0.5 liter of the sample water. To find the optimum coagulant dose for each treatment option, doses between 6 mg/l and 22mg/l were investigated using a series of jar test. Coagulation experiments were carried out with a PB-900, Belgium variable speed jar tester. A rapid mixing at 260rpm for 2min was applied followed by slow stirring phase at 35rpm for 15minutes to let the flocs grow. Samples were taken for the analysis of UV_{254} after a settling of 30 min and a dose which gave the lowest UV_{254} value..UV_{254} was measured using UV-Vis spectrophotometer and Zeta potential using Zeta-sizer (Malvern Instruments, UK).

2.4. Floc size measurements

A Malvern Mastersizer 2000, coupled with the coagulation device by a peristaltic pump, was used to measure floc size as shown in Figure2. In addition, jar tester and Mastersizer were coupled to measure the floc size as in Jarvis *et al.* (2005b). . The water sample was pumped from the jar tester through the measurement cell of Mastersizer by a peristaltic pump at a rate of 6 liter per hour (l/hr). Floc size measurements were performed every minute for all stages of growth, breakage and re-growth. The median floc size (d_{50}) was read from the instrument and logged on for further analysis.

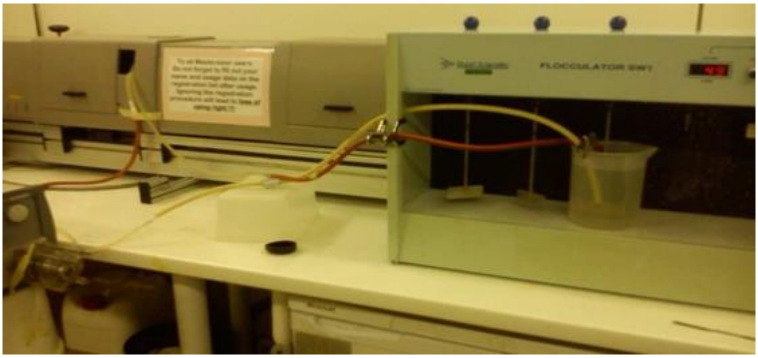

Figure 1: Experimental set up for floc size measurements (Mastersizer coupled with jar tester)

Following the flocculation phase, a variation of different higher shear rate was applied to break the floc at slow mixing rate. For the flocs coagulated jar tester, a breakage phase of 5 minutes was carried out after flocculation at a mixing speeds of 50 rpm, 75rpm, 100 rpm, 150 rpm and 200 rpm corresponding to average velocity gradient of $33s^{-1}$, $61s^{-1}$, $94s^{-1}$,$172s^{-1}$, $265s^{-1}$ respectively. The floc size is measured every minute during all the flocculation phases. Finally, the floc was allowed to re-grow for 15 minutes after breakage phase, at stirring rate of 35 rpm and size was measured to examine the re-growth potential. The strength and recovery factor were calculated based on (Equation 1 and 2) (Yukselen & Gregory, 2004; Jarvis *et al.*, 2005b; Burton, 2009). The average floc size d_{50} as obtained from the Mastersizer was used to calculate the strength and recovery factors. As shown in Equation 1 and 2, d_1, d_2 and d_3 are the steady state average d_{50} floc sizes at the end of growth phase, breakage phase, and re-growth phase, respectively.

$$\text{Strength factor} = \frac{\mathrm{d}_2}{\mathrm{d}_1} * 100 \qquad (1)$$

$$\text{Recovery factor} = \frac{\mathrm{d}_3 - \mathrm{d}_2}{\mathrm{d}_1 - \mathrm{d}_2} \qquad (2)$$

Another way used to characterize floc strength is the empirical relationship between the applied shear force and remaining floc size in the suspension at the end of breakage phase, respectively.

$$\log_{d_2} = \log(C) - \gamma * \log(rpm) \qquad (3)$$

The slope of the equation (γ) and the intercept (log(C)) indicates , floc strength constant and floc strength co-efficient, respectively (equation 3).

2.5. Statistical Analyses

The descriptive statics such as mean, standard deviation, and standard error of the floc size measurements were analyzed by Analysis Tool pack of Microsoft Excel.

3. RESULTS AND DISCUSSION

3.1. Optimal coagulant dosage

For raw water, coagulation with $AlCl_3$ gave best removal of UV_{254} (0.073) at a dosage of 11mg /l while FeC13 gave the lowest UV_{254} (0.033) at a dosage of 9mg/l for the pretreated water, refer Table 2. The choice of best coagulant to be used depends on the type of water quality. For all the coagulants and comparable dosage, lower values of UV_{254} were obtained for the pretreated water compared to the raw water. This is mainly because UV_{254} absorbing organics were already removed by the pre-treatment of the raw water with ion exchange. This is consistent with the result of Singer & Bilyk (2002) where significant reduction of TOC was observed with the water pretreated with MIEX.

Table 2: Summary of obtained UV_{254} result for the three coagulants with different doses

Dose (mg/l)	UV absorbance at 254 nm (cm^{-1})							
	Raw + $AlCl_3$	Raw + PACl	Raw + $FeCl_3$	Pretreated + $AlCl_3$	Pretreated + PACl	Pretreated+ $FeCl_3$	Raw + $AlCl_3$	Raw + PACl
6	0.095	0.097	0.134	0.074	0.062	0.034	0.095	0.097
9	0.088	0.114	0.12	0.073	0.06	0.033	0.088	0.114
11	0.073	0.11	0.115	0.072	0.059	0.033	0.073	0.11
14	0.090	0.101	0.108	0.065	0.058	0.034	0.090	0.101
16	0.090	0.082	0.105	0.066	0.056	0.032	0.090	0.082

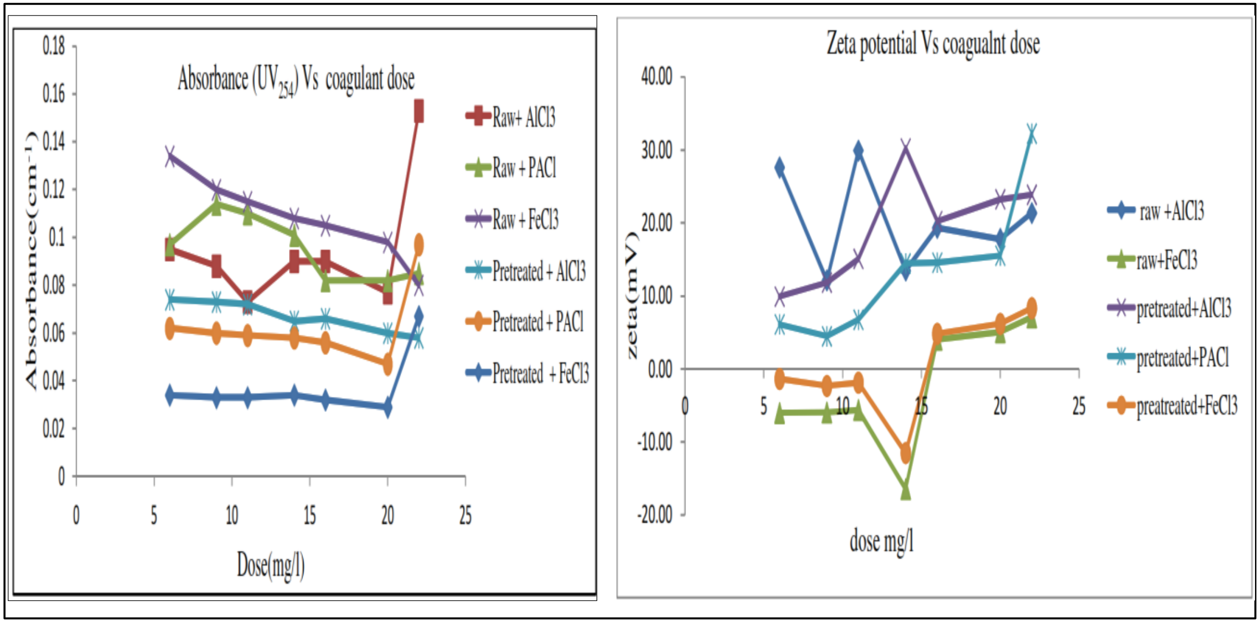

Figure 2: Absorbance and zeta potential as function of coagulant dose for all the coagulant and both water types

As shown in Figure 2, both water types coagulated with $AlCl_3$ and pretreated water coagulated with PACl have positive zeta potential for the entire dose applied. As a result, it is most likely that hydroxide precipitation and sweep coagulation are responsible for the coagulation. On the other hand, zeta potential for both water types coagulated with $FeCl_3$ changes from negative to positive at a dose close to 16 mg/l. The negative zeta potential obtained at a dose of 11 mg/l as Fe might indicate that charge neutralization is the main coagulation mechanism occurring for Fe.

3.2. Floc Size

While measuring the floc size with different coagulants and water samples, the growth, breakage and re-growth phases of the floc were investigated at a dose of 11mg/l for both raw and pretreated water. As can be seen in Table 3, the d_{50} floc size during the growth phase of flocculation for both water types was in the following order: $FeCl_3 <$ PACl $< AlCl_3$. For all the coagulants, floc sizes in the pretreated water were higher than those of the raw water. This result proves that pre-treatment of the raw water with MIEX had a significant impact on the size of the flocs formed during coagulation / flocculation with the same mixing conditions. This is in conformity with the result obtained by Jarvis *et al.* (2004) and Burton (2009) where larger and stronger flocs were observed for the pre-treated water compared to the raw water. This might be as a result of the reduction in the amount of natural organic matter such as DOC after pre-treatment with MIEX. The modified composition of the water had an impact on floc structure such as size, strength, fractal dimension, since smaller DOC to coagulant ratio had the potential to produce floc with better structure. The improved floc structure (at lower DOC concentration) was ascribed to the interaction of carbon with floc matrix. At higher DOC concentration, NOM aggregates might be covered with the organic carbons which limited further aggregation or lead to the formation of loosely structured aggregates when they were flocculated with inorganic salts. Another reason for the improved floc size after pre-treatment could be MIEX pre-treatment removed low molecular weight organics. The remaining higher molecular weight fraction polymers had improved binding capacity with flocs leading to larger flocs size

(Burton, 2009). After pre-treatment, the floc size increased by 19%, 63% and 64% for the coagulants: PACl, $AlCl_3$, and $FeCl_3$, respectively. This result indicated that pre-treatment of the raw water with MIEX and selections of coagulants had a large influence on the floc size.

Table 3: Floc size of raw water and pretreated water for the three coagulants after 1.5 minutes coagulation at 260 rpm and 15 minute flocculation time at 35 revolutions per minutes (RPM)

Coagulant	Raw water floc average size (d_{50}) during growth	Pre-treated water floc, Average size (d_{50}) during growth	Increase in size after pre-treatment
$AlCl_3$	102±13.4μm	167±13.6μm	63%
PACl	160±9.6μm	190±6.8μm	19%
$FeCl_3$	219±3.9μm	357±10.6μm	64%

The floc size during the growth, breakage and re-growth phase for the two water types with the different coagulants was shown from Figure 3 to Figure 5. The floc size was represented by the volumetric median diameter (d_{50}) as obtained from the Mastersizer. The data showed growth phase of the floc during 15 minutes flocculation at a stirring rate of 35 rpm. Floc size during the breakage phase was measured at different mixing intensities for 5 minutes while the re-growth phase was carried out for 15 minute at a reduced intensity of 35rpm.On the graphs, the first vertical line indicated the point at which increased shear (50 rpm,75 rpm, 100 rpm, 150 rpm and 200 rpm) was introduced for floc breakage (after 15 minutes of flocculation).The second vertical line indicated the point where shear was again reduced (35 rpm) for the re-growth of the flocs after 5 minutes of breakage time.

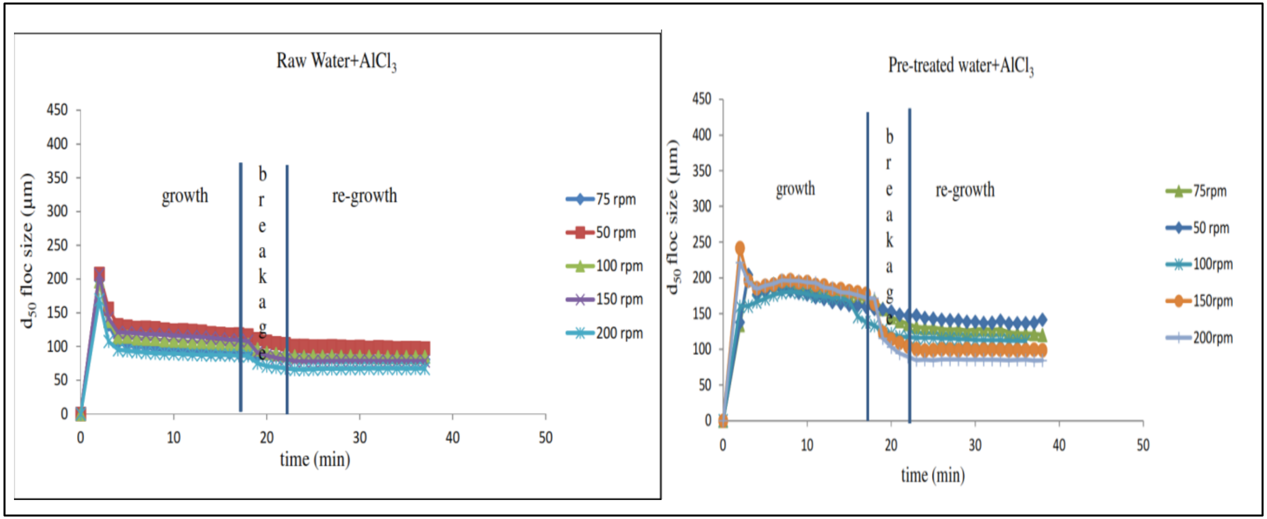

Figure 3: Floc size profile for$AlCl_3$ flocs formed from raw water and pre- treated water up on exposure to different levels of shear (at a dose of 11 mg/l as Al at pH6.8 for both water types).

From the Figures (3-5), it can be seen that the floc size during the growth phase was different for the two different water types. For all the coagulant, the pretreated water showed consistently higher floc sizes than the raw water.

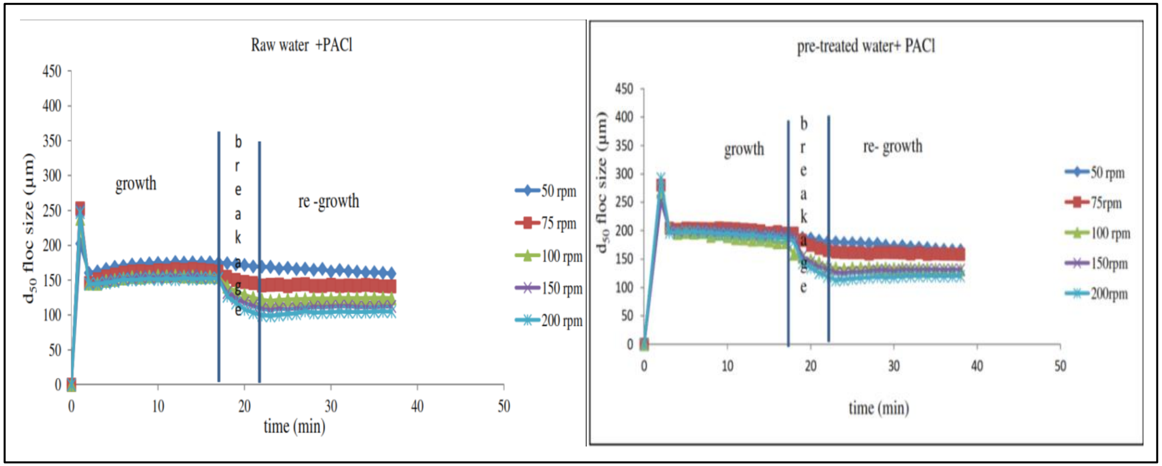

Figure 4: Floc size profile for PACl flocs formed from raw water and pre-treated water upon exposure to different levels of shear (at a dose of11 mg/l as Al at pH 6.8 for both water types).

For PACl coagulation with both water types, the evolution of the flocs size during growth, breakage, and re-growth was similar to that of $AlCl_3$. An increase in floc size was not observed during the flocculation phase after coagulation.

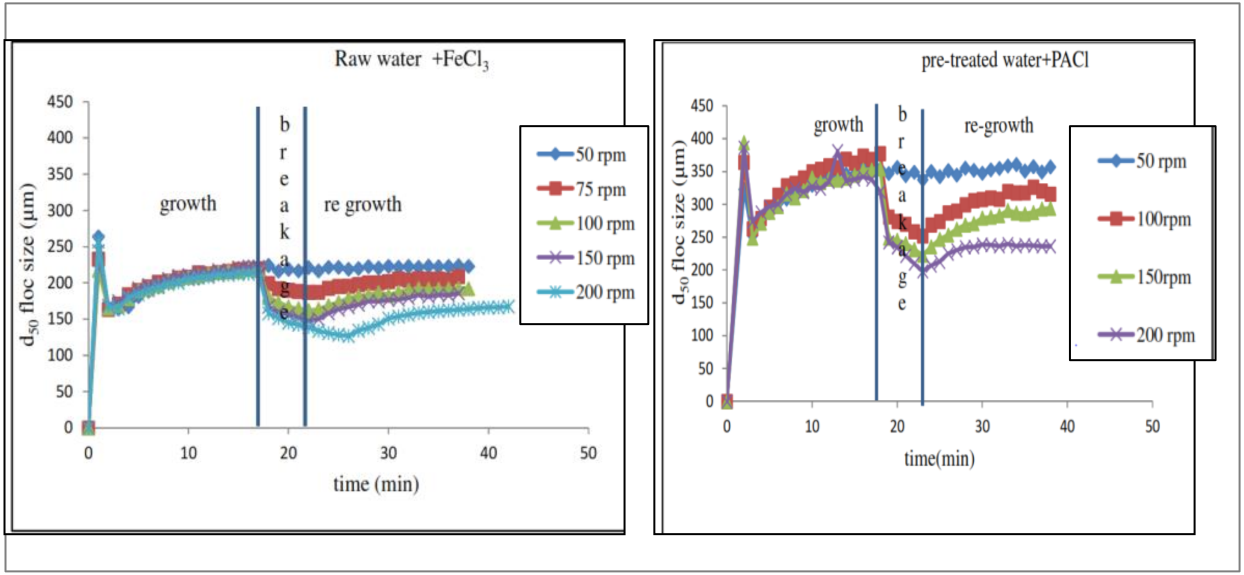

Figure 5: Floc size profile for $FeCl_3$ flocs formed from raw water and pre-treated water upon exposure to different levels of shear (at a dose of11 mg/l as Al at pH6.8 for both water types).

Statistical analysis on the floc size for both sample water types showed that there was a significant difference (Levene's Test, $p < 0.001$ at 95% confidence interval). Hence the choice of the coagulants and water types had a significant impact on the floc size obtained from the flocculation during growth phase.

It was seen from the results that, the flocculation phase for $FeCl_3$ was different from the other two coagulants. After coagulation with $FeCl_{3,}$ the floc continued to grow until the end of the growth phase for both water types. This might be explained not only by the organic matter to coagulant ratio, but also by the behavior of the coagulant itself. Larger floc formation after coagulation with $FeCl_3$ was due to the property that iron-based coagulants had preferential selectivity for organic matter than aluminum-based coagulants (Uyak & Toroz, 2007). This might explain why the pretreated water coagulated with ferric chloride had the lowest UV_{254}. This finding conforms to that of Uyak & Toroz (2007) who obtained better removal of UV_{254} by ferric coagulants than aluminum based coagulant.

It was also observed that application of high shear force during breakage resulted in a decrease in floc size. In this case, the more intense the mixing the smaller got the floc size. For example, the size decreased largely when the mixing was done at 200rpm. However, the floc size remained unchanged when the mixing was done at 50rpm. For $FeCl_3$ the breakage phase observed was similar to that the two other coagulants where broken floc size decreased as breakage intensity increased.

3.3. Floc strength and recovery

The strength of the floc to resist the high shear applied during the breakage phase is highly dependent on the intensity and duration breakage of the breakage. The breakage intensity and duration also affects the ability of flocs to re-grow in size when fewer shear is applied after breakage. Zhun, *et al*. (2012) found that the sizes of the broken and re-grown flocs were dependent not only on the applied shear conditions during breakage and re-growth but also on the conditions during the initial floc growth. Furthermore, Zhao *et al*. (2011b) carried out jar tests and investigated the strength and re-growth potential of flocs at high shear for different breakage periods (1 minute and

10 minutes). They reported that flocs had higher re-growth potential after short breakage periods (1 minute) compared to longer periods (10 minutes) at the same mixing intensity. Floc size after breakage might be inversely related to the intensity of the shear applied (Spicer *et.al.*, 1996; Burton, 2009). This is due to the fact that floc size is the result of aggregation and fragmentation of flocs where an increased shear rate can lead to breakage of more flocs (Yukselen & Gregory, 2004). For breakage at the highest intensity of 200rpm, $AlCl_3$ showed the highest strength factor (81%) followed by PACl (68%) and $FeCl_3$ (62%) for the raw water. For the pre-treated water, breakage at mixing intensity of 200rpm, $FeCl_3$ showed highest strength factor (66%) followed by PACl (64%) and $AlCl_3$ (56%). When statistically analyzed, the calculated strength factors did not show significant difference between the two water types (Levene's Test, $p=0.808$).Similarly, strength factors did not reveal any significant difference for the different coagulant types (Levene's Test =0.15). After the shear was reduced to the slow stirring at 35rpm, there was no evidence showing change in floc size of both water coagulants, $AlCl_3$ and PACl. However, $FeCl_3$ flocs obtained from both water types were able to recover after the shear was reduced to 35rpm and hence there was increase in floc size as re-growth time increased (Table 5).The recovery potential indeed depended on the intensity of the shear applied to break the floc. The floc broken with lower intensity (i.e. 75 rpm) showed larger recovery potential compared to the flocs broken with higher intensities (i.e. 200 rpm). There was no statistical significant difference observed in recovery factor between the raw and pretreated water for all the shear force applied (Levene's Test, $p=0.56$). Conversely, recovery factor showed significant difference between the coagulants used for each shear condition applied (Levene's Test, $p< 0.001$). The Post Hoc test of multiple comparisons showed that there was significant differences in recovery factor when using $FeCl_3$ compared to PACl and $AlCl_3$ (with $P=0.004$ and $P<0.001$, respectively).

Table 5: Calculated floc strength and recovery factor

Shear force (rpm)	Raw water + $AlCl_3$		Raw water + PACl		Raw water + $FeCl_3$		Pre- treated water + $AlCl_3$		Pre-treated water + PACl		Pre-treated water+ $FeCl_3$	
	SF(%)	RF (%)	SF(%)	RF (%)	SF(%)	RF(%)	SF (%)	RF(%)	SF (%)	RF(%)	SF (%)	RF(%)
50	97	-244	97	-181	98	154	92	-127	94	-113	99	161
75	90	-86	88	-25	85	67	82	-73	84	-22	-	-
100	87	-41	81	-2	75	54	71	-21	76	-13	68	48
150	78	-33	73	-2	68	50	62	-22	68	-2	65	51
200	81	-21	68	3	62	36	56	-21	64	1	66	24

Note: SF= strength factor, RF= recovery facto

Another way of characterizing the floc strength was by examining the response of d50 floc size to the increased shear rate. This was done by plotting the measured d_{50} size at the end of 5 minute of breakage phase against the rpm of the jar tester on a log- log scale (see material and method section 2.3). The R2 values for these log-log plots were high (between 0.946 and 0.996) indicating a good correlation (Figure 6).

The significant difference seen in recovery factor might be related to the charge neutralization coagulation mechanism involved with iron coagulants. This conformed to Zhao *et al*. (2011a) who found higher recoverability for flocs formed by charge neutralization coagulation mechanism. The higher selectivity of iron coagulants to NOM could be cause for the higher recoverability of iron flocs after breakage (Burton, 2009).

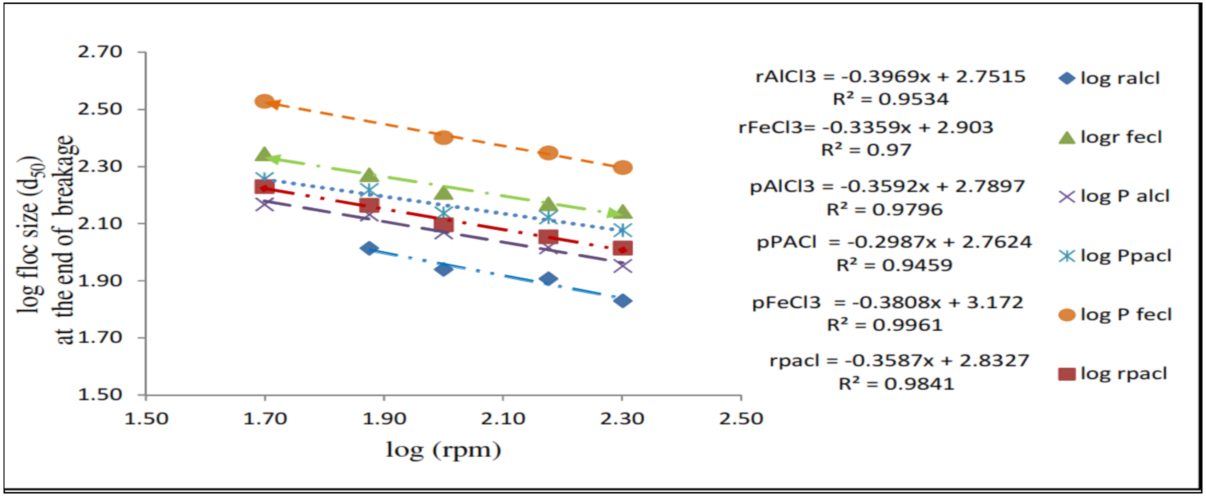

Figure 6: Response of floc sizes after breakage to an increased shear rate. Log- log relationship between floc size (d_{50}) and shear (rpm)

With pre-treated water, the coefficient of floc strength for $AlCl_3$ was found to be (0.359) whereas with raw water the coefficient changed to 0.396. The same trend was seen for PACl: 0.30 and 0.36 with pretreated water. Hence, it can be concluded that flocs formed with raw water were more prone to breakage than those with pretreated water for $AlCl_3$ and PACl. On the other hand, flocs generated with $FeCl_3$ showed higher for pretreated water (0.388) than those with raw water (0.336), and thus flocs were easier to breakdown after pre-treatment. This conformed to Burton's (2009) findings for $FeCl_3$. However, for PACl and $AlCl_3$ the trends seen here might not conform to Burton's (2009) findings. This disagreement might be attributed to differences in resin type used for ion exchange in pre-treatment of the raw water, seasonal variation in water characteristics, or differences in coagulant dose used with the two studies.

4. CONCLUSIONS AND RECOMMENDATIONS

This study examined the overall structural characteristics of flocs generated from raw surface water and water pretreated with MIEX obtained from the Kluizen drinking water treatment plant. The growth, breakage and re-growth of flocs generated from the two different water types were assessed for three different coagulants ($AlCl_3$, PACl, and $FeCl_3$). This study examined the overall structural characteristics of flocs generated from raw surface water and water pretreated with MIEX obtained from the Kluizen drinking water treatment plant. The growth, breakage and re-growth of flocs generated from the two different water types were assessed for three different coagulants ($AlCl_3$, PACl, and $FeCl_3$). Furthermore, the effect of the coagulation device and mixing intensities (velocity gradients) on the floc sizes were examined. The result obtained from the laboratory experiment showed that pre-treatment of raw water with MIEX removed (part of the) DOC and UV254 absorbing organics from the raw water which lead to improved water quality. The flocs generated using PACl as coagulant were found to be strongest based on the lower value of γ and the higher fractal dimension of the micro-flocs (floc aggregates smaller than 2.5μ m). Flocs generated using $FeCl_3$ as coagulant showed higher recovery factors (i.e., they re-grow better after breakage), which was explained by the higher selectivity of Fe to organic matter and by the charge neutralization coagulation mechanism involved with Fe coagulant. Higher floc sizes were obtained after pre-treatment with MIEX, because of the decreased concentration of lower molecular weight substances after MIEX. Higher molecular weight polymer like substances left in the water gave an increased floc sizes when coagulated. The result also confirmed that the mixing intensities during coagulation had a significant impact on the obtained floc sizes. The flocs resulted from coagulation in a jar tester (typical velocity gradient ~ $393s^{-1}$) were more compacted and lower in size than flocs generated via waterfall/weir mixing (typical velocity gradient ~ $750\ s^{-1}$) which were larger in sizes but less strong. The set-up with pump arrangement and tubes used to transfer the suspension from the beaker to the Mastersizer measurements had an impact on the floc sizes obtained.

Further research is necessary on the influence of mixing intensities/velocity gradients during coagulation on floc properties. The interaction between coagulants and different NOM constituents should be examined to better understand effects of MIEX pre-treatment on coagulation. There should also be more research to clearly understand why different coagulants behave differently for both water types. Seasonal variations on the efficiency of the coagulation/flocculation should be considered since the water quality and temperature might change with respect to the season.

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