



# FILTRATION OF SUBMICRON PARTICLES: EFFECT OF IONIC STRENGTH AND ORGANIC SUBSTANCES

H. Prasanthi\*, S. Vigneswaran\*, T. D. Waite\*\*  
and R. Ben Aim\*\*\*

\* *School of Civil Engineering, University of Technology, Sydney, P.O. Box 123, Broadway, NSW 2007, Australia*

\*\* *School of Civil Engineering, University of New South Wales, P.O. Box 1, Kensington, NSW 2033, Australia*

\*\*\* *Université de Compiègne, Département de Génie Chimique, BP 649, 60206, Compiègne Cedex, France*

## ABSTRACT

Hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ) suspension was used in this study to investigate the effects of aggregate size and solution chemical characteristics on particle removal and headloss development in a deep bed filter. When electrolyte (KCl) concentration in the suspension was increased from 0-80 mM, the size of hematite aggregates in suspension increased from 85 to 990 nm but had no significant effect on zeta potential. It was found that at low KCl concentrations (0-10 mM), the initial removal is governed by surface chemical properties while at higher concentrations (40-80 mM), the effect of particle size (steric effect) predominates. The transient stage filter performance was found to be controlled by the favourable chemical conditions prevailing in the filtration system rather than the size of the particles.

Studies of the effect of a natural organic material (fulvic acid) on particle removal indicated that fulvic acid at low concentrations (less than 0.75 mg/L) enhanced particle aggregation and improved filtration efficiency. At concentrations higher than 2 mg/L, it was found to increase the stability of particles thus affecting filtration efficiency.

## KEYWORDS

Clean bed filter efficiency; deep bed filtration; fulvic acid; hematite particles; ionic strength; transient stage filter efficiency

## INTRODUCTION

In a typical water treatment plant, most of the suspended particles are removed by coagulation and sedimentation processes. However, a significant number of particles escape these processes and must be removed by deep bed filters. Particle removal in deep bed filtration involves two distinct steps: transport and attachment. The particles are first transported near to the filter grain by mechanisms such as diffusion, interception, sedimentation, etc., before attachment takes place. The efficiency of particle removal is influenced by a number of factors including the physical and chemical characteristics of the suspension, suspending medium and filter medium.

Particles in natural water interact with inorganic and organic ions or molecules which adsorb on to the surfaces by various mechanisms. Most particles possess an electric charge which attracts ions of opposite charge in the solution forming compact and diffuse layers. The changes in surface characteristics brought

about by a variation in solution characteristics become very important when particles interact among themselves or with filter grains during filtration. These interactions result in attractive or repulsive forces which may enhance or reduce the deposition and removal of particles in deep bed filters.

Most surface waters and reclaimed wastewaters contain organic substances in dissolved or colloidal form. The organics have a capacity to adsorb on to the particles, thus bringing about significant changes in the surface characteristics of the particles thus modifying their interactions with other particles or with any other surface they come into contact. Fulvic acid, having the lowest molecular weight among humic substances and being the most water soluble fraction, is found in natural waters in higher concentrations compared to other fractions of natural organic matter. Humic substances, in general, are negatively charged macromolecules for the pH conditions of most natural waters and impart a negative charge to particles to which they adsorb (Narkis and Rebhun, 1975).

Narkis and Rebhun (1975) also found that the presence of organic matter in solution or on the mineral clay particle surface inhibits the process of flocculation by cationic polyelectrolytes. In order that both the particle destabilisation and bridging stages of flocculation may take place, larger doses of flocculant are required to produce conditions suitable for bridging.

In the present research, hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ) was chosen as model suspension, since the stability, size, shape and chemical characteristics can be controlled with relative ease in the laboratory. Previous studies (Tipping and Higgins (1982), Amal *et al.*, (1990, 1992), Tiller and O'Melia (1993)) have shown that the physical (size) and chemical (surface charge) characteristics of these particles are affected by the adsorption of inorganic or organic species present in the solution. In these studies, KCl has been used as background electrolyte with its concentration varied to induce changes in particle size and surface chemistry of the system - factors which have significant influence on filter performance. Likewise, the International Humic Substances Society standard Suwannee river fulvic acid was used to represent the natural organics and to study its effects on filtration. The filter performance in all cases was monitored in terms of particle removal and headloss development.

## EXPERIMENTAL INVESTIGATION

Two series of experiments, one in which the ionic strength was varied and the other in which the concentration of fulvic acid was varied were carried out. In all experiments hematite suspension was used which was prepared by the method described by Penners and Koopal (1986) based on the procedure developed by Matijevic and Scheiner (1978). Positively charged particles with a mean diameter of 85 nm were prepared which were homogeneous and stable at room temperature and pH 3. Prior to conducting filtration experiments, either with different ionic strengths or with varying fulvic acid concentrations, the particles were allowed to equilibrate in the electrolyte for a period of 24 hours.

The effect of ionic strength of the suspending medium on filter performance was studied by varying KCl concentration from 0 to 80 mM ( $10^{-3}$  -  $8 \times 10^{-2}$  M ionic strength). The results of previous studies (Amal *et al.*, 1990) indicate that the rate of aggregation approaches diffusion limitation (and thus independence of KCl concentration) at KCl concentrations greater than approximately 80 mM.

Based on preliminary filtration experiments and zeta potential studies, fulvic acid concentrations of 0.25, 0.5, 0.75 and 2.0 mg/L were chosen to study the effect of the presence of fulvic acid on filtration of hematite particles. Fulvic acid concentration of 4 mg/L was also used to check the clean bed filter efficiency. In this series of experiments, the pH and KCl concentration were maintained constant at 3 and  $10^{-3}$  M respectively.

For all experiments, glass beads with a mean diameter,  $d_p$ , of 0.175 mm were used as the filter medium. The glass beads were soaked in a large volume of electrolyte solution at pH 3 and given ionic strength ( $10^{-3}$  M of KCl for all experiments with fulvic acid) for a period of 24 hours prior to filtration studies.

This procedure ensured that the glass beads surfaces were completely equilibrated with electrolyte ions. In order to measure zeta potential, glass beads were soaked in the same concentration of fulvic acid as used in filtration experiments.

A perspex filter column of 25 cm length and inner diameter of 5 cm was employed for all experiments. Glass beads were packed in the column to a height of 3 cm with a porosity of approximately 0.4. Hematite suspension at a particular concentration was fed continuously at a constant head to the filter column. Filtration was carried out for periods of 60 min. to a few hours depending upon the conditions used and the breakthrough obtained. Effluent samples of 20 mL volume were then collected every 2 minutes for the first 10 min, then the time period for sample collection was gradually increased. The headloss development was monitored at every sampling using piezometric tubes.

The mean diameter of particles and aggregates was analysed using a Malvern 4700 PCS system utilising a 15 mW, 633 nm He-Ne laser. Zeta potential of the particles and glass beads was analysed using a Coulter 400 Delsa Analyser and cross checked with mobility values obtained using a Rank Brothers Mark II Microelectrophoresis Apparatus.

The effluent quality was monitored in terms of total iron concentration which was measured using a Perkin Elmer 3100 Atomic Absorption Spectrometer. Since repetitive PCS analysis showed the average diameter of the particles at a specific ionic strength or a fulvic acid concentration to be approximately the same, it was assumed that hematite suspension has uniform size under varying chemical conditions. Thus the particle number concentration was calculated using the iron concentrations.

Table 1 summarises the relevant conditions and variables used.

TABLE 1. EXPERIMENTAL PARAMETERS

Length of the bed, cm	3
Filter medium	Glass beads*
Mean diameter of filter grains, $d_c$ , mm	0.175
Porosity of the filter bed	0.4
Filter velocity, $v$ , m/h	1.54
Filter run time, hr	1-4
Particles used	Hematite, $\alpha$ -Fe <sub>2</sub> O <sub>3</sub>
Primary particle mean diameter, $d_p$ , nm	85
Influent concentration in terms of Fe <sub>2</sub> O <sub>3</sub> , $C_o$ , mg/L	34-38
pH	3
Electrolyte; concentration, mM	KCl; 0-80
Organic matter; concentration, mg/L	Suwannee river fulvic acid®, 0-4

\* : Source: B. Braun Melsungen AG, Germany

@ : Source: International Humic Substances Society (c/o Colorado School of Mines, Colorado, USA)

## RESULTS AND DISCUSSION

Filter performance at different ionic strengths and fulvic acid concentrations was monitored in terms of particle removal and headloss development. In all experiments, the removal efficiency was significantly different in the initial and transient stages of filtration.

The effect of ionic strength and organics on clean bed filter efficiency is quantified using the initial removal efficiency,  $\eta_{exp}$  calculated using the time zero filtration experimental results:

$$\eta_{\text{exp}} = -\frac{2}{3} \frac{d_c}{(1-f)L} \ln \left( \frac{C_e}{C_o} \right)$$

where,

- $\eta_{\text{exp}}$  = experimental single collector removal efficiency;  
 $d_c$  = diameter of the filter grains;  
 $f$  = porosity of the filter bed;  
 $L$  = length of the filter bed;  
 $C_e$  = effluent concentration at time zero; and  
 $C_o$  = influent concentration.

In this paper, the performance of the filter during transient stages of filtration is assessed qualitatively from the concentration and headloss profiles. The theoretical analysis of the filter performance is given elsewhere (Prasanthi and Vigneswaran, 1993).

### Effect of ionic strength

With pH constant at 3, the aggregate size of hematite particles was found to vary at different KCl concentrations (Table 2) although their zeta potential values remained similar (Fig. 1). However, KCl concentration varies the zeta potential of glass beads (Fig. 1). In this system, the varying aggregate size at different ionic strength enabled us to study the effect of particle size (in the submicron range) on particle removal in the initial as well as during the transient stages of filtration.

*Clean bed filter efficiency.* The variations of aggregate size and  $\eta_{\text{exp}}$  obtained at different KCl concentrations, are presented in Table 2. Based on the results, the experimental runs can be divided into two categories: (i) low KCl concentrations (0-10 mM) and (ii) high KCl concentrations (40-80 mM).

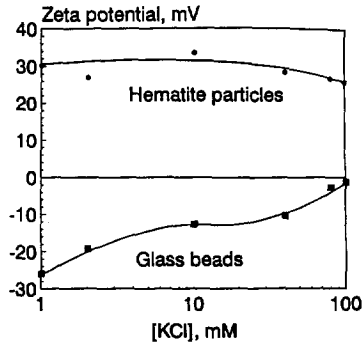


Fig. 1. Zeta potential of hematite particles and glass beads at pH 3 as a function of KCl concentration of the suspending medium

At low ionic strengths ( $\leq 10$  mM KCl), a high proportion of particles are initially removed due to the nearly equal and opposite charge on the glass beads and particles. However, a monolayer of particles rapidly adheres to the collector surface with a resultant reversal in charge of the collectors. In this case, the repulsive forces between the incoming particles and collectors do not allow for further significant removal. A slight increase in  $\eta_{\text{exp}}$  is observed when the KCl concentration is increased from 0 to 1 and then 10 mM. Mean diameters of hematite aggregates at these ionic strengths are 85, 85 and 105 nm respectively and would not seem to account for the observed increase in  $\eta_{\text{exp}}$ . Indeed, a slight decrease in removal efficiency would be expected for an increase in particle size from 85 to 105 nm (Yao *et al.*,

1971). Presumably the observed increase in  $\eta_{exp}$  under these conditions occurs as a result of lowered electrical double layer thickness of hematite particles (in suspension and on the collector) with a resultant increase in the efficiency of particle-filter grain attachment.

TABLE 2. AVERAGE AGGREGATE SIZE AND FILTER EXPERIMENTAL INITIAL REMOVAL EFFICIENCY VALUES AT DIFFERENT KCl CONCENTRATIONS

Run	KCl conc., mM	$d_p$ , nm	$C_f/C_o$	$\eta_{exp} \times 10^{-3}$
1	0	85	0.770	1.694
2	1	85	0.7283	2.055
3	10	105	0.3988	5.958
4	40	180	0.012	28.66
5	60	460	0.03376	21.96
6	80	990	0.0542	18.89

At higher KCl concentrations (40-80 mM KCl), significantly larger hematite aggregates are formed. Indeed, the expected decrease in removal efficiency with increase in colloidal aggregate size (to about 1  $\mu\text{m}$ ) (Yao et al., 1971) appears to override any increase in particle-filter grain attachment since  $\eta_{exp}$  is observed to decrease with increase in ionic strength.

The above discussions imply that for lower ionic concentrations (when the aggregate size is less than 110 nm), the initial efficiency is governed by surface chemistry of the particles and the suspending medium, but for higher ionic concentrations (when the aggregate size increases from 110-1000 nm), the steric effect takes over. However, it is difficult to generalise at this stage; more experiments with different types of particles are required to justify this qualitative explanation.

*Particle removal during transient stages of filtration.* Figure 2 presents the concentration profiles obtained at different ionic strengths. As discussed above, for low KCl concentrations, monolayer formation of particles on the filter grains at the early stages of filtration inhibits further removal of particles. Thus a quick breakthrough (deterioration of particle removal with time) is achieved with  $C_f/C_o$  (fraction of particles remaining in the effluent after filtration) values soon approaching unity.

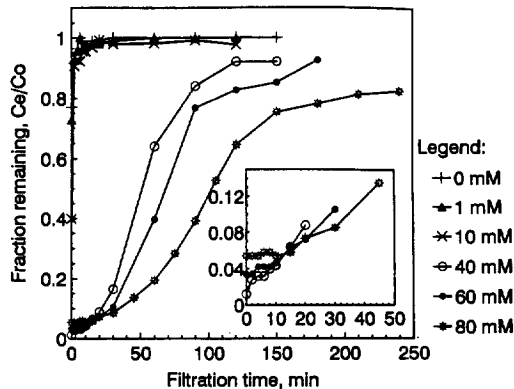


Fig. 2. Influence of different KCl concentrations on the filtration of hematite particles ( $d_c=0.175$  mm,  $L=3$  cm,  $C_o=34.5-36$  mg/L as  $\text{Fe}_2\text{O}_3$ ,  $\text{pH}=3$ ,  $v=1.54$  m/h)

For higher KCl concentrations, as the filtration proceeds, the removal efficiency follows the same pattern as that of the initial removal (i.e., 40 mM > 60 mM > 80mM). But after approximately 20 min of filtration, breakthrough is achieved with the trend in particle removal getting reversed with the removal efficiency of 80 mM > 60 mM > 40 mM. In the case of 40 mM, more particles are retained due to the favourable particle-filter attachment during the ripening stage (during which the filtration efficiency increases with time). However, this increased retention prevents further adhesion of particles, thus achieving quick breakthrough. Whereas, in the case of 80 mM, due to lower initial removal of particles, saturation of filter grains is delayed. As such the ripening period is prolonged over a longer period compared to that obtained during filtration experiments conducted at 40 and 60 mM KCl concentrations.

In these experiments, the headloss development was negligible and remained almost the same even when the ionic strength of the suspension was increased. The increase in the removal efficiency and thus the headloss development at higher ionic strength is counterbalanced by the increase in aggregate size. This is consistent with results obtained by O'Melia & Ali (1978) and Chang (1989), where the headloss development was observed to be less for large particles.

### Effect of organic substances on filtration

The effect of natural organic substances on the stability and aggregation of hematite particles is well documented (Tipping and Higgins (1982), Amal *et al.* (1992) and Tiller and O'Melia (1993)). Figure 3 shows the effect of fulvic acid on zeta potential of hematite particles and glass beads at constant pH of 3 and KCl concentration of  $10^{-3}$  M. As the fulvic acid concentration was increased from 0 to 1 mg/L, a steep decrease in zeta potential of hematite was observed with a reversal in the surface charge of the particles, after which it remained constant. If the adsorption of fulvic acid onto the particles had been governed just by electrostatic interactions, the zeta potential would have reduced in its value and remained at zero. Instead, the surface charge is reversed indicating that some specific chemical interactions are also responsible for the adsorption of fulvic acid (Amal *et al.* 1992). As the net charge on the particles starts reducing, at low fulvic acid concentrations and given ionic strength, aggregation of particles is favoured. At higher concentrations of fulvic acid, e.g., 2 mg/L and above, aggregation was negligible and the aggregate size was maintained at around 90 nm.

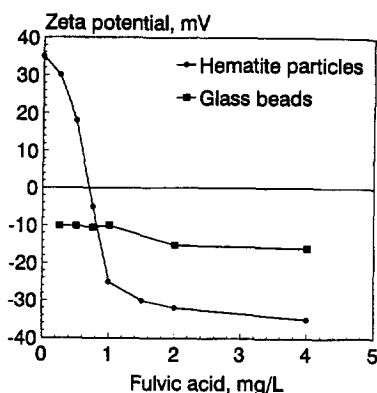


Fig. 3. Zeta potential of hematite particles and glass beads as a function of fulvic acid added to the solution (pH=3, KCl concentration= $10^{-3}$  M)

Five different concentrations of fulvic acid, 0, 0.25, 0.5, 0.75 and 2 mg/L were used to study the effect of organics on particle removal by filtration. Another experiment with 4 mg/L was carried out to check only the clean bed filter efficiency. The results obtained are discussed below in terms of removal efficiency and development of headloss in the initial and transient stages of filtration.

*Clean bed filter efficiency.* Table 3 presents the  $\eta_{\text{exp}}$  values for the filtration experiments of hematite suspensions conducted at different concentrations of fulvic acid. An attempt has been made to explain the changes in initial removal of particles in terms of aggregate size of particles and the charge of the particles and filter grains.

In the absence of fulvic acid, the suspension is stable due to electrostatic repulsion between positively charged hematite particles. When fulvic acid is added, the zeta potential is lowered and destabilisation of particles occurs. At low fulvic acid concentrations (e.g., 0.25 mg/L), most of the fulvic acid molecules added are adsorbed to the particle surface, thus bringing about destabilisation due to reduction in net surface charge. Thus during filtration experiments,  $\eta_{\text{exp}}$  for 0.25 mg/L fulvic acid was found to be higher than that in the absence of fulvic acid.

TABLE 3. AVERAGE AGGREGATE SIZE AND FILTER EXPERIMENTAL INITIAL REMOVAL EFFICIENCY VALUES AT DIFFERENT CONCENTRATIONS OF FULVIC ACID

Run	Fulvic acid conc., mg/L	$d_p$ , nm	$C_f/C_o$	$\eta_{\text{exp}} \times 10^{-3}$
1	0	85	0.7283	2.055
2	0.25	186	0.395	6.021
3	0.50	885	0.0206	25.16
4	0.75	1065	0.095	15.26
5	2.00	96	0.06	18.24
6	4.00	90	0.2064	10.23

At concentrations of 0.5 and 0.75 mg/L, fulvic acid brings down the net charge of the particles resulting in marked increases in aggregate sizes. As can be observed in Table 3, for 0.5 mg/L of fulvic acid, high  $\eta_{\text{exp}}$  is obtained. At this concentration, the reduction in repulsive forces between particles and filter grains become more predominant thus enhancing particle removal by adhesion of still positively charged particles onto the filter grains. However, at 0.75 mg/L of fulvic acid,  $\eta_{\text{exp}}$  is less which may be due to the fact that the size of the aggregates increases to above one micron which is the critical size of filtration at which particle transport (by any mechanism) is at a minimum.

When fulvic acid concentration was increased to 2 mg/L and above, aggregation was not favoured due to the increased stabilisation of the particles. Amal et al., (1992) and Tiller & O'Melia (1993) have reported that at such high concentrations of fulvic acid, all the particles are completely covered with fulvic acid molecules. Due to this coverage, the particles acquire negative charge and the diffuse layer extends into the solution with a resultant increase in electrostatic repulsion between the negatively charged surfaces. Tipping and Higgins (1982) also indicate that, due to the relatively high molecular weight of humic substances, steric stabilisation may be effective, which is the prevention, by an adsorbed layer, of the approach of particles to a separation small enough for dispersion forces to cause aggregation. In the present study, it was observed that in the case of fulvic acid concentrations of 2 and 4 mg/L, the particles remained almost non-aggregated ( $\approx 90$  nm). Interesting however,  $\eta_{\text{exp}}$  was equally as high as in the case of 0.75 mg/L. Presumably, though the net negative charge is high, the increase in transport of particles due to diffusion leads to high clean bed filter efficiency because the aggregate size is much lower than the critical size of 1  $\mu\text{m}$ .

In summary, it can be said that although the trend of  $\eta_{\text{exp}}$  with varying fulvic acid concentration does not follow a definite pattern, it appears that both steric effect (particle size) and particle charge play equally important roles. From the qualitative explanation given, one should expect the lowest clean bed filter efficiency when 2 mg/L or more of fulvic acid is present. However, the results from repetitive

experiments indicate that higher clean bed filter efficiencies are observed at high fulvic acid doses (2 & 4 mg/L as shown in Table 3). No conclusive explanation can be given at this stage. It is worth noting, however, that the transient stage filtration efficiency at these concentrations was lower as expected. This is discussed in the following section.

*Particle removal during transient stages of filtration.* The filter performance, in terms of particle removal and headloss development are presented in Fig. 4 and 5 respectively. Both concentration and headloss profiles were found to be different for different concentrations of fulvic acid added.

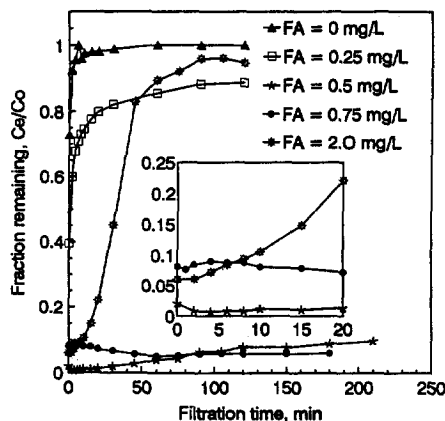


Fig. 4. Influence of different fulvic acid (FA) concentrations on the filtration of hematite particles ( $d_c=0.175$  mm,  $L=3$  cm,  $C_o=37-38$  mg/L as  $Fe_2O_3$ ,  $pH=3$ ,  $KCl$  concentration= $10^{-3}$  M,  $v=1.54$  m/h)

When a fulvic acid concentration of 0.25 mg/L was used, immediate breakthrough occurred due to the electrostatic repulsion between the still positively charged particles though slightly greater removal was observed over the duration of the run than found in the absence of organic presumably as a result of the somewhat lower surface charge of hematite particles. In this case, the headloss developed was slightly higher than in the absence of organics, possibly as a result of the higher particle retention when fulvic acid was present.

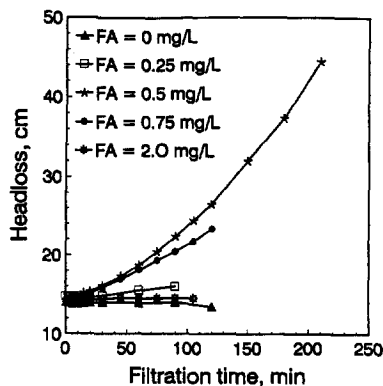


Fig. 5. Influence of different fulvic acid concentration on headloss development ( $d_c=0.175$  mm,  $L=3$  cm,  $C_o=37-38$  mg/L as  $Fe_2O_3$ ,  $pH=3$ ,  $KCl$  concentration= $10^{-3}$  M,  $v=1.54$  m/h)



At a fulvic acid concentration of 0.5 mg/L, the extent of destabilisation favours particle-particle interaction, thus improving the filter performance. In the case of 0.75 mg/L, particle removal during the transient stage still remains high. As can be observed in Fig. 5, the headloss increases very rapidly for both the cases which is due to high retention of particles.

At 2 mg/L of fulvic acid concentration, the hematite particles in suspension are stabilised by specific adsorption of negatively charged fulvic acid molecules. Inferior filter performance results because of the repulsion between suspended and attached fulvic acid coated hematite particles. In this case as particle retention is at a minimum, no significant increase in headloss was observed.

## CONCLUSION

In this paper, the results of filtration experiments of hematite suspension at varying ionic strengths and fulvic acid concentrations are presented and their effects are discussed. When KCl concentrations of 0-80 mM were used, the zeta potential of the particles remained relatively unchanged, though the aggregate size increased from 85 to 990 nM. It was found that at lower KCl concentrations, the initial removal is governed principally by the chemical aspects of the suspension (surface charge and particle attachment) and at higher concentrations, the particle size (steric effect) plays a more important role. The filter performance during the transient stages of filtration, however was found to be controlled by the favourable chemical conditions prevailing in the filtration system rather than the size of aggregates.

Suwannee river fulvic acid was used to study the effect of organics on filtration. From the experimental results obtained it was concluded that fulvic acid, at low concentrations (0.25-0.75 mg/L), reduces the stability of the primary particles by acting as an anionic polymer. This leads to enhanced particle aggregation and higher filtration efficiency. At concentrations higher than 2 mg/L, fulvic acid increases the stability of particles thus reducing filtration efficiency.

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