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Calcite-amended horizontal roughing filtration for clay turbidity removal

Stephen J. Rooklidge and Lloyd H. Ketchum Jr

ABSTRACT

Crushed basaltic river rock, calcite limestone, and calcite-amended basalt were three types of media tested in pilot-scale horizontal roughing filters pretreating slow sand filters fed from the Santiam River over 60-day periods. Montmorillonite and kaolinite clay challenge tests were conducted on ripened and unripened roughing filters to test performance at turbidities greater than 150 NTU, and clay removal trends were qualitatively examined using clay peak area ratios derived from X-ray diffraction. Enhanced montmorillonite removal was evident in the calcite roughing filter, but schmutzdecke growth was inhibited in the calcite-fed slow sand filter. The basalt, calcite and calcite-amended roughing filters in combination with slow sand filtration achieved 88.0, 95.3 and 99.6% clay turbidity removal, respectively, during the challenge tests. The slow sand filter pretreated with a calcite-amended roughing filter was the only configuration to produce effluent that complied with the 1 NTU regulatory requirement using influent of <1 to 150 NTU.

Key words | amendments, limestone, roughing filters, slow sand filters, turbidity

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INTRODUCTION

Slow sand filtration is an increasingly viable drinking water treatment option for many small and rural communities. The long history of the technology has proved its proficiency for removing or reducing contaminants found in water sources used for public consumption. As the worldwide demand for adequate water treatment is expanded by rural population growth and increasingly stringent regulations, the lack of standard slow sand filter (SSF) configurations to properly treat raw water with high turbidity fluctuations becomes more apparent. This inadequacy limits the use of the technology to raw water sources that have naturally low turbidity, or requires the addition of pretreatment systems to eliminate particulates prior to SSF application. Pretreatment for removal of suspended material allows slow sand filters to produce drinking water from surface sources where use of this economical treatment system is otherwise prevented, such as mountain streams and rivers that contain excessive clay contamination during rain events.

Gravel rock roughing filters have been used for decades as a pretreatment technique for surface waters subject to high fluctuations of turbidity. Horizontal roughing filters are long troughs (5–7 m) with a series of flow-through compartments containing decreasing sizes of gravel media ranging from 20 mm average diameter in the first compartment to 4 mm average diameter in the last, and common flows from 0.3 to 1.5 $m^3\ h^{-1}\ m^{-2}$ of horizontal filter area. Cleaning of the media is commonly performed by increasing the head above the media surface and quickly opening bottom drains to discharge at initial rates above 45 m h^{-1}, and comprehensive reviews of engineering design considerations are provided in Wegelin (1996) and Collins *et al*. (1994). The primary removal mechanism for non-colloidal particles is physical sedimentation within the interstitial voids of the rock media. Limestone contactors have been used to aid corrosion control in water distribution systems by altering the water chemistry to less corrosive conditions, and the combination of these techniques to enhance removal of colloidal clay within the roughing filter is a method that has shown promise when applied to sources with moderate pH and alkalinity (Lehmann 1996). Crushed limestone as roughing filter media was used in early studies by Wegelin in Tanzania (1983) with influent water of approximately 60 NTU and 100 mg l^{-1} suspended solids. These bench-scale experiments were followed by a 15-m long horizontal roughing filter experiment that produced effluent of less than 20 NTU from a natural water source, and the remaining turbidity was attributed to residual colour in the source water.

Previous limestone roughing filter clay removal laboratory studies by this research group, using montmorillonite (M-clay) as a representative smectite and kaolinite (K-clay) as a non-expanding control, indicated a statistically significant increase of M-clay removal at calcium additions less than 3 mg l^{-1}. Zeta potential and X-ray diffraction studies suggested the M-clay removal mechanism was particle destabilization and sedimentation, without the occurrence of interlayer cation exchange (Rooklidge *et al.* 2002). The use of limestone in a roughing filter increases raw water ionic strength and may aid coagulation by slightly reducing the clay's repulsive forces through double-layer compression (Rae and Parker 1998). However, the previous studies did not preclude destabilization from surface charge neutralization due to cation exchange, and the actual clay removal mechanism within the tortuous route through the filter media voids may very well be a combination of phenomena.

High-calcium limestone use in a roughing filter is a self-regulating system that is strongly dependent on solution pH and mass action dissolution rates corresponding to the open-to-atmosphere stoichiometric reaction idealized below (Chou *et al*. 1989; Stumm and Morgan 1996).

$$CaCO_3 + H^+ \rightarrow Ca^{2+} + HCO_3^- \qquad (1)$$

Calcium hardness is added to the water as the mineral dissolves, and the formation of bicarbonate ions with a corresponding decrease of free hydrogen ions presents a rise in pH and alkalinity. The potential benefits of using limestone-amended roughing filters prior to slow sand filtration on raw water of moderate pH and alkalinity include destabilization of negatively charged contaminant particles from added calcium ions, enhanced buffering capacity of the effluent from increased alkalinity, and increased filter pH compensating for normal reduction in a filter system due to biological activity.

This study coupled pilot-scale roughing filter pretreatment with slow sand filters fed raw water from the Santiam River in western Oregon during the winter and early spring of 2001. The research site was built at the City of Salem, Oregon Geren Island water treatment facility, which experienced raw water smectite clay turbidity greater than 140 NTU during a flood in 1996 (Bates *et al.* 1998). The calcite roughing filter and slow sand filter (CRF/SSF) clay removal efficiencies were compared with a basalt roughing filter combination (BRF/SSF) and calcite-amended (CBRF/SSF) configuration. Each filter system was subjected to 60-day studies of low to moderate turbidity to examine the consistency of filter removal efficiencies. High turbidity clay challenge experiments were performed on the pilot filter systems, using suspensions of laboratory-grade kaolinite and montmorillonite at a K-clay/M-clay ratio of 0.3 to represent the approximate ratio of clay constituents in the Santiam River at the time of the 1996 flood event (Glassman 1997). Montmorillonite and kaolinite removal trends were examined in the basalt and calcite roughing filters, during challenge experiments on day 3 and day 40 of each study, using sample peak area ratios derived from X-ray powder diffraction patterns.

MATERIALS AND METHODS

Two parallel horizontal roughing filters were manufactured from exterior-grade plywood supported by a beam and pier-block foundation, and sealed with epoxy at wood connections and fastener points to form a watertight box. Each 2.14-m long, 0.9-m wide roughing filter had longitudinal filter cells, separated by wire mesh baffles fixed between vertical wood lath screwed to the filter interior wall, at a horizontal length ratio of 3:2:1 holding media gradations with size ranges of 30–20, 20–12 and 12–9 mm, respectively. The baffle mounting lath and rough plywood

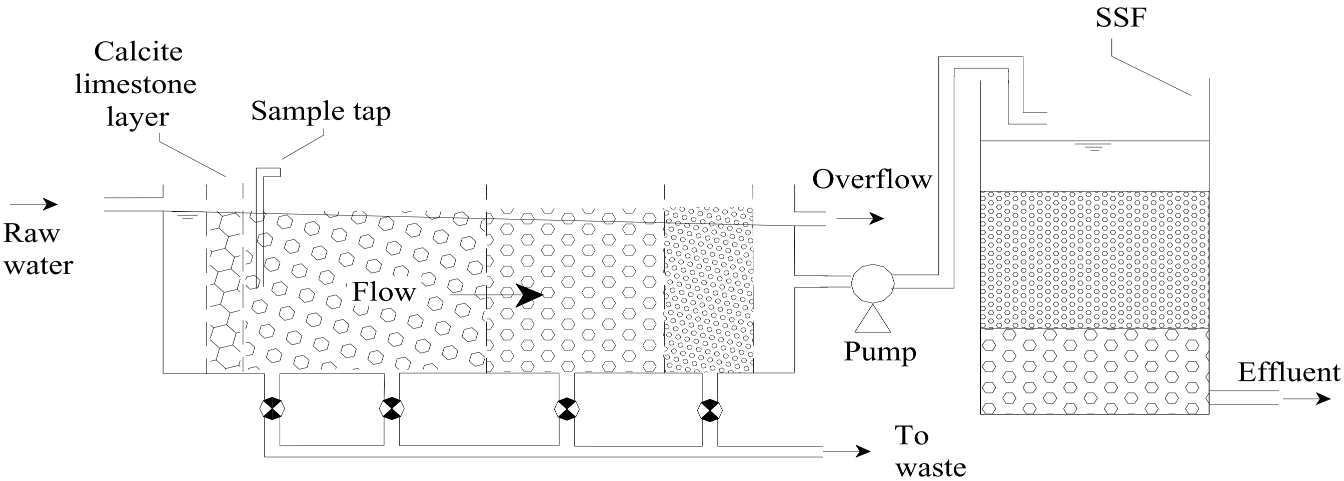

Figure 1 | Pilot roughing and slow sand filter configuration showing CBRF calcite layer and sample tap. Roughing filter freeboard not shown.

finish diminished potential side wall effects, and baffle flow restrictions and sediment deposition were not considered significant. The BRF and CRF configurations were run for the first 60-day study period, followed by media removal and washing with high pressure river water. The BRF was then modified for the second 60-day study with the addition of a 14-cm calcite limestone layer placed vertically in the first filter cell (CBRF), with basalt river rock making up the remaining filter media at a length ratio of 0.46:2.54:2:1 (Figure 1). The calcite layer had an identical media size gradation to the first basalt cell. A small siphon sample tap was installed just downstream of the calcite/basalt interface, and was allowed to run continuously during the clay challenge experiments. The water level was kept below the 50-cm surface depth of the rock media by an overflow, and roughing filter headloss was not monitored. Roughing filter influent was provided at a superficial velocity of 0.5 m h^{-1} by a pump placed in the treatment facility intake structure, which fed directly into a weir above a 15-cm long baffled equilibration chamber. Flow through the filters was influent-controlled using a ball valve, and calibrated with a graduated cylinder and stopwatch. The filter media produced a calculated average pore volume detention time of 108 minutes, which was verified by turbidity breakthrough during the clay challenge experiments.

A 100-l continuously mixed plastic reservoir held the clay contaminant suspension used during the challenge experiments. The clay slurry was made from raw river water, mixed in the reservoir for 24 hours prior to the challenge tests, adjusted to a neutral pH using 0.1 N HCl, and delivered into the raw water influent by an injection port upstream of the roughing filter weir. Turbidity was monitored at the weir by grab samples, and the peristaltic feed pump was adjusted to maintain the required turbidity influent concentration of 150 ± 20 NTU.

Pilot slow sand filters were built from 3.05-m tall black polyethylene tanks with a 1.2-m internal diameter, and provided with 2-cm PVC influent and overflow piping. The 0.6-m deep crushed river rock support media complied with AWWA B100 standards. The sand media depth in the filters was 0.92 m, with a uniformity coefficient of 2.26 and effective size of 0.30 mm. The sand media was washed clean during production, and both filter runs were preceded by pipe flushing, scraping of SSF schmutzdecke, and chlorination to ensure an unripened condition at the start of both study periods. Each assembled filter configuration was allowed to run to waste for a period of 3 days before initial samples were drawn. This provided enough wasted discharge for the slow sand filters to produce effluent less than 1 NTU, without allowing biological maturation of the filters. Slow sand filter influent was adjusted to a superficial velocity of 0.2 m h^{-1}, and the filters were not covered during the pilot study.

Headloss was monitored in the slow sand filters as an indicator of biofilm growth in the schmutzdecke and filter grains of the media bed. Piezometers were placed in the slow sand filters with a zero reference at 10 cm below the sand/gravel support media interface (P1). Two more piezometers were placed in the filter at 20 cm below the sand/water interface (P2) and at the sand/water interface (P3). Headloss of the most active biological layer (schmutzdecke) was calculated as P3–P2. All pH and temperature measurements were made using a Hach SensION2 pH/ISE meter with a temperature-compensating Hach-One platinum series electrode using KCl reference gel and a 0.01 resolution at 0.05% accuracy of reading. Compensation for salt concentrations was not necessary due to the low ionic strength of all water samples. Tests were performed immediately after sample collection from the RF equilibration chambers, CBRF sample tap and SSF effluent pipes. Calcium concentrations were also measured using a Hach SensION2 meter with a calcium half-cell electrode and a single junction reference electrode filled with Ag^{+} saturated KCl. All samples were adjusted for ionic strength using 1 ml of 4 M KCl per 50-ml sample. Total alkalinity was determined by the low alkalinity potentiometric titration of *Standard Methods* (1998), with an initial end point of pH 4.5 because of the presence of alumino-silicates. Average values from duplicate turbidity measurements were calculated using a daily formazin-calibrated portable turbidimeter (2100P, Hach Co., Loveland, Colorado).

Clay from the CRF and BRF influent and effluent samples was analysed using intensities of X-ray powder diffraction (XRD) peaks generated by a diffractometer equipped with a variable slit (Rigaku Miniflex, Japan), with continuous measurement using Cu Kα radiation. The method of sample preparation used for the roughing filter study was a modified version of the glass slide method (Moore and Reynolds 1997). The technique used 500-ml water samples evaporated in a steam bath at 60°C to prepare the solids for XRD analysis. The evaporated samples were resuspended on a glass slide using drops of distilled water, placed in an oven at 50°C for 10 minutes to aid clay adhesion and then fixed in the analyser mount.

Table 1 | Summary of Santiam River raw water quality parameters and standard deviations (StD) during pilot studies

Parameter	Mean	StD
Temperature (C)	8.1	1.5
pH	7.3	0.2
Alkalinity (mg l^{-1} as $CaCO_3$)	30.1	3.0
Hardness (mg l^{-1} as $CaCO_3$)	15.5	1.7
Conductivity (μS cm^{-1})	36.6	3.2
Calcium (mg l^{-1})	4.1	1.3

Standards and samples were scanned with a 1-second step time at 0.02° 2θ increments between 22° and 30°. The near-neighbour kaolinite peak at *c*. 25° and montmorillonite peak at *c*. 27° obviated potential diffraction intensity loss in the smaller effluent samples. The roughing filter sample XRD pattern K-clay to M-clay peak area ratios, derived from integrating software (Jade 3.1®), were compared to a calibration curve developed from evaporated raw water specimens with known additions of laboratory-grade clay concentrations (Sigma-Aldrich, 09866/03584) to determine actual kaolinite to montmorillonite ratios.

RESULTS

The Pacific Northwest experienced a very warm and dry winter during the experimental period. Raw water temperature during the first 60 days averaged 7.5°C ± 2.6, and the second study period averaged 8.5°C ± 2.8. This was not considered a significant fluctuation to affect calcium dissolution or filter biological maturation. Raw water chemistry of the Santiam River was very consistent throughout the pilot study because of a lack of high water release rates from the upstream reservoir at Detroit, Oregon. Average raw water quality parameters are listed in Table 1.

Results of the 60-day monitoring of pH, alkalinity and calcium values for each filter configuration are presented

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Table 2 | Summary of turbidity removal and percentage standard deviation (StD) of individual roughing filters and roughing/slow sand filter combinations during 60-day studies and clay challenge experiments

Filter	% Turb. removal 60-day studies	StD	% Turb. removal Clay challenge	StD
BRF	24.5	32.3	68.6	3.6
CRF	18.8	19.7	71.3	3.8
CBRF	28.4	17.8	75.4	3.8
BRF/SSF	73.9	8.6	88.0	4.9
CRF/SSF	59.3	15.7	95.3	1.4
CBRF/SSF	72.6	9.8	99.6	0.1

in Figure 2. The slight reduction of pH in the slow sand filters was consistent with the formation of biofilm during the study periods. The increased pH and alkalinity in the SSF effluents fed from the CRF and CBRF indicated a benefit to corrosion control in downstream distribution system piping. An increase in the CRF-fed SSF calcium concentrations was consistent with previous laboratory results by this research group, which identified continued dissolution of calcite in the effluent of pure limestone roughing filters. This behaviour may have influenced Wegelin's earlier study (1983) that assumed limestone roughing filter effluent turbidity was caused by residual colour. The average influent calcium concentrations were lower during the second study period, and a statistically insignificant loss of free calcium ions was experienced in the CBRF/SSF system. It is not known whether small losses of calcium ions from solution initiated charge neutralization on montmorillonite particles that led to the observed enhanced turbidity removal of the CBRF filter system.

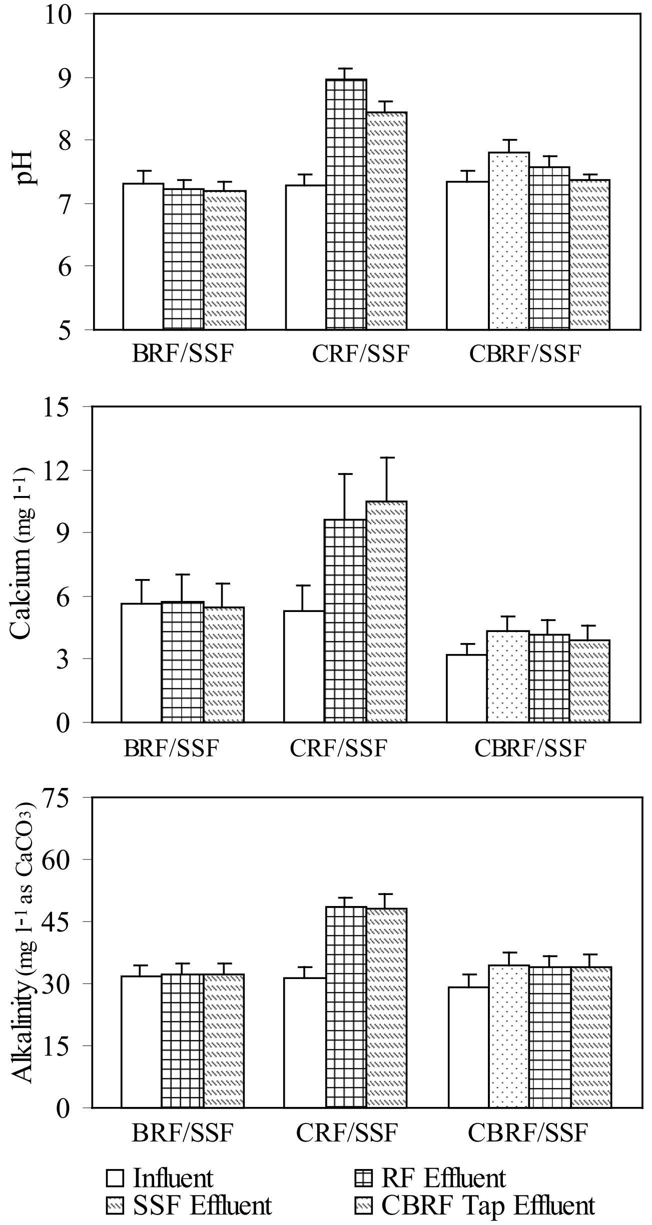

Figure 2 | pH, calcium and alkalinity results from influent, sample tap and filter effluent grab samples collected during the 60-day filtration studies.

Results of BRF-fed SSF, CRF/SSF and CBRF/SSF headloss accumulation throughout the 60-day pilot studies are shown in Figure 3. A very low rate of headloss formation was observed in the SSF fed influent from the CRF compared with the other filters. No quantitative biological assessments were performed, but schmutzdecke samples were taken at the end of each filter run and observed under an optical microscope. The filter mat of the CRF/SSF was very thin and uneven, but no obvious difference in microorganism populations was qualitatively identified between the slow sand filter schmutzdecke samples. It was assumed that the higher pH of the water caused by the dissolving limestone media of the CRF did

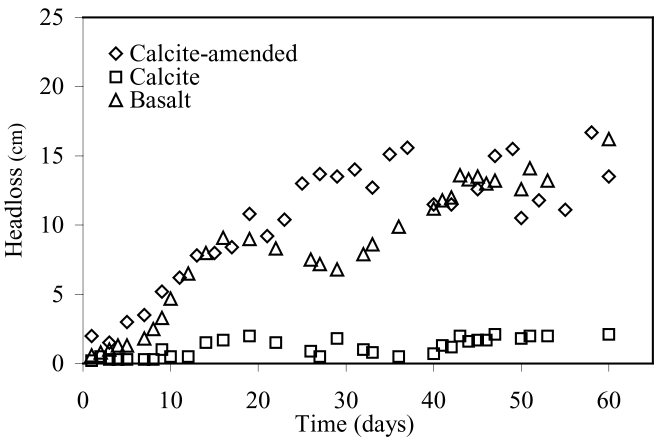

Figure 3 | Schmutzdecke headloss accumulation of the slow sand filters fed by the calcite, basalt and calcite-amended roughing filters.

not present a favourable environment for the microbial and higher life forms native to the river.

The Santiam River exhibited consistent turbidity below 7 NTU during each of the 60-day pilot study periods, and was generally less than 2 NTU. The BRF had an average turbidity removal efficiency of 24.5% under low influent turbidity conditions (Figure 4). The SSF connected to the BRF removed 64.1% of the BRF effluent turbidity, with an average effluent of 0.42 NTU. All pilot slow sand filters produced effluent less than 1 NTU after 12 continuous days of filtration, but no significant trend of increasing turbidity removal efficiency was observed in the BRF during the 60-day study. This behaviour was consistent with the observation that higher roughing filter removal efficiencies are associated with higher solids loading (Collins *et al.* 1994). The CRF produced an average turbidity removal efficiency of only 18.8%, and the calcite filter SSF removal efficiency was 49.9%, with an average effluent of 0.68 NTU. Roughing filter effluent turbidity readings may have been increased by the presence of calcite particles that subsequently dissolved in the SSF supernatant, as indicated by the higher calcium ion concentrations in the SSF effluent. During the second study period, turbidity through the calcite layer of the CBRF was occasionally above the influent turbidity, and the average turbidity removal efficiency of the calcite layer was 10.1%. The remaining basalt media removal efficiency was 19.6% of the turbidity passing the calcite/basalt interface. The SSF connected in series to the CBRF removed 71.9% of the remaining turbidity, and produced an average effluent of 0.30 NTU.

The 150 NTU clay challenge turbidity removal in the CRF ripened for 40 days averaged 71.3%, and its SSF removal averaged 81.5% after the 320-minute filter system detention time. The BRF average turbidity removal was 68.6%, and its SSF removal efficiency was 88.0% (Figure 5). Effluent from both filter configurations exceeded the 1.0 NTU limit during the challenge tests. The 14-cm calcite layer of the CBRF exhibited an average turbidity removal efficiency of 52.8% during the second study period clay challenge experiment. Average removal by the remaining basalt media layer was 48.5% of the turbidity passing the calcite/basalt interface, and the overall ripened calcite-amended roughing filter average removal was 75.4% of the raw water influent turbidity. The SSF turbidity removal was 95.5% of the CBRF effluent concentration, and produced water that did not exceed the 1.0 NTU limit during the challenge test. A summary of the average turbidity removal experienced in the roughing filters and slow sand filter/roughing filter combinations is exhibited in Table 2. The only filter configuration that produced effluent acceptable for the provision of potable drinking water not exceeding regulatory turbidity limits was the slow sand filter pretreated by a calcite-amended basalt roughing filter.

The use of qualitative XRD-derived sample K-clay/M-clay ratios in the pilot study was designed to indicate kaolinite and montmorillonite clay removal trends in ripened and unripened calcite and basaltic roughing filters. However, to be able to establish qualitative trends of clay removal, a linear standard curve between added clay concentration ratios (K/M) and XRD peak area ratios (k_a/m_a) had to be developed. Previous research by Glassman (1997) indicated the presence of kaolinite and smectite clay within the Santiam River during the flood of 1996, but there was a need to determine potential interference by clay contaminants present during the pilot study. Prior to the clay challenge experiments 3 l of raw water was evaporated and the solids were examined using XRD. The pattern generated from these samples is illustrated in Figure 6, and peaks near the crucial 2-theta

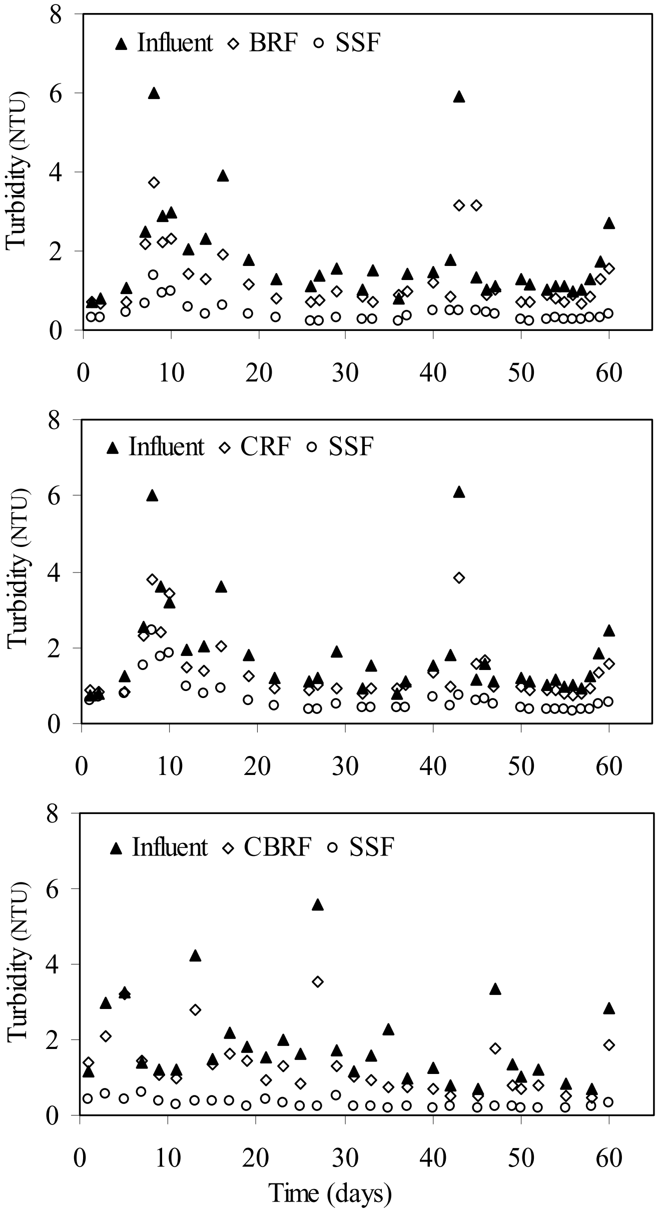

Figure 4 | Influent and effluent turbidities of the roughing and connected slow sand filters during the 60-day studies.

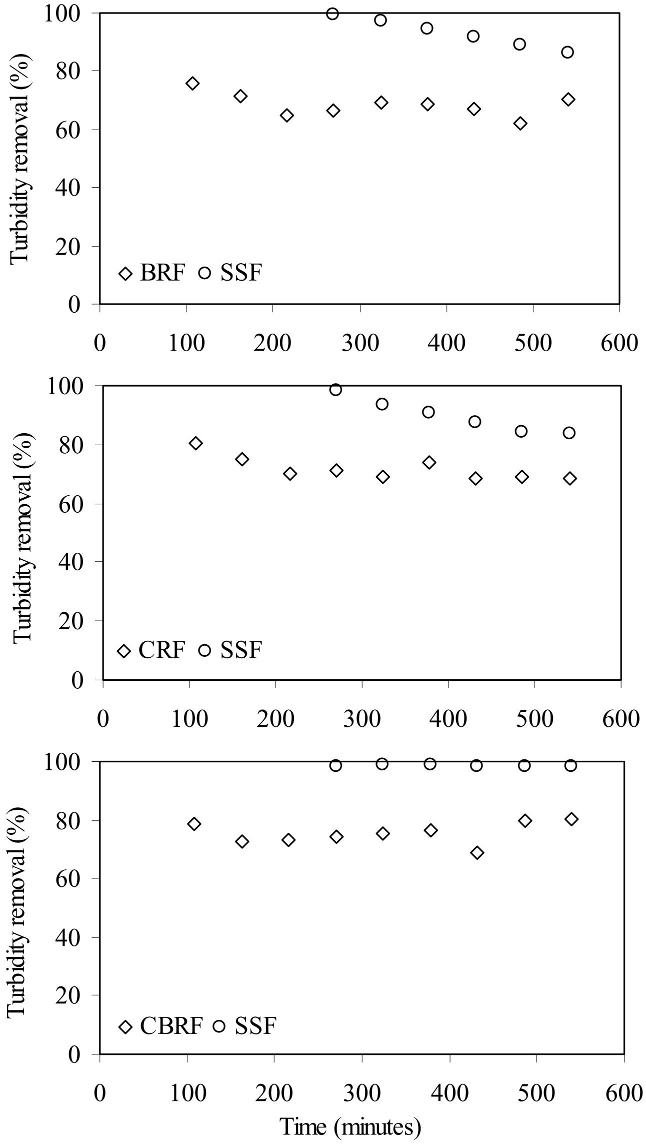

Figure 5 | Clay turbidity removal of roughing filters and connected slow sand filters during 150 NTU challenge experiments with K-clay to M-clay ratio of 0.3.

values of 25° and 27° suggest the presence of clay minerals in the raw water that may have also been present during the flood of 1996. Known concentrations of kaolinite and montmorillonite were mixed in 1-l beakers of raw water; the corresponding evaporated sample peak area ratios are presented in Figure 7. The standard curve generated exhibited linearity within acceptable limits, and it was assumed that XRD patterns of evaporated influent and effluent samples from the clay challenge experiments would give accurate representations of added clay ratios.

Trends of clay removal, as illustrated by the change of kaolinite to montmorillonite ratios, are shown for the unripened CRF and BRF (Figure 8). Montmorillonite is

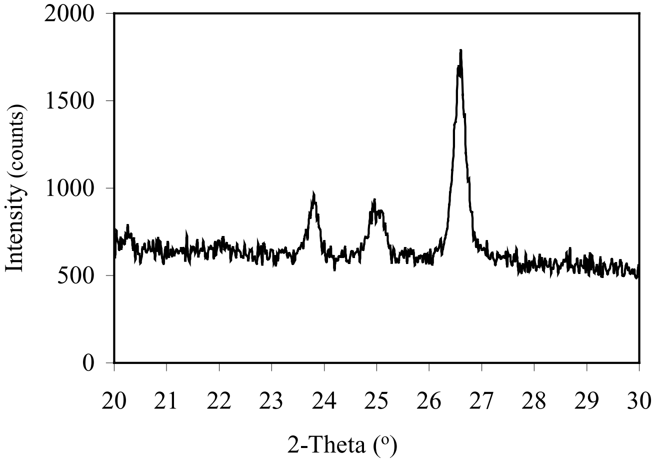

Figure 6 | X-ray diffraction pattern of Santiam River sample evaporated from 3 l of raw water.

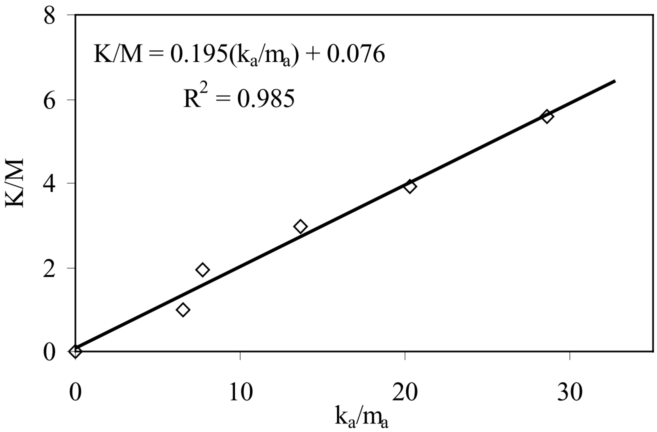

Figure 7 | Standard curve of XRD-derived K-clay/M-clay peak area ratios (ka/ma) plotted versus known added clay concentrations (K/M).

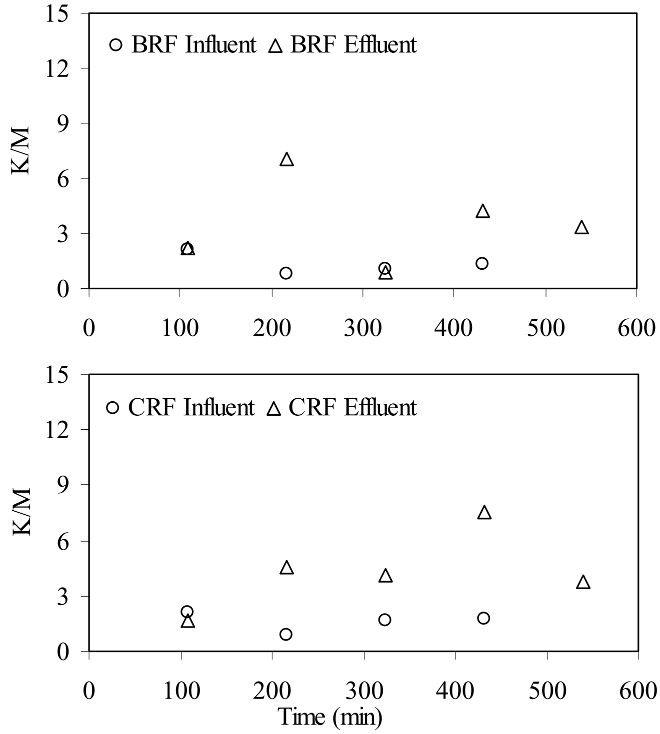

Figure 8 | Change of influent and effluent sample K/M ratios in unripened calcite and basalt roughing filters.

preferentially removed in both roughing filters, but a larger removal rate occurred in the unripened calcite filter. This behaviour was expected because smectite clays are more susceptible to surface charge alteration when exposed to increasing cations in solution (van Olphen 1977). The ripened BRF exhibited an increase of M-clay removal compared with an unripened condition, and removal trends were similar to those calculated from samples of the ripened CRF (Figure 9). The increase of BRF M-clay removal may have been due to clay particle interaction with microbial growth that was inhibited in the higher pH environment of the CRF. Biofilm formation on media grains presents a surface far more conducive to particle adhesion than clean media (Bellamy *et al.* 1985; Lehmann 1996), and this could be a more important removal process in ripened roughing filters than clay flocculation initiated by cation-induced particle double-layer compression or surface charge neutralization.

Unlike roughing filters with inert media, operational strategies for full-scale limestone RF treatment systems should consider the effects of clay particle sedimentation on limestone dissolution rates. Calcite dissolution in a CBRF may be slowed by clay deposition on the media, which decreases the available calcite surface area and blocks effective transport of bulk solution to the media surface layer. Continued applied research is warranted for design of adequate maintenance cleaning schedules for calcite-amended roughing filters, as more frequent flushing may be necessary to restore calcite dissolution rates before the roughing filter achieves a critical headloss.

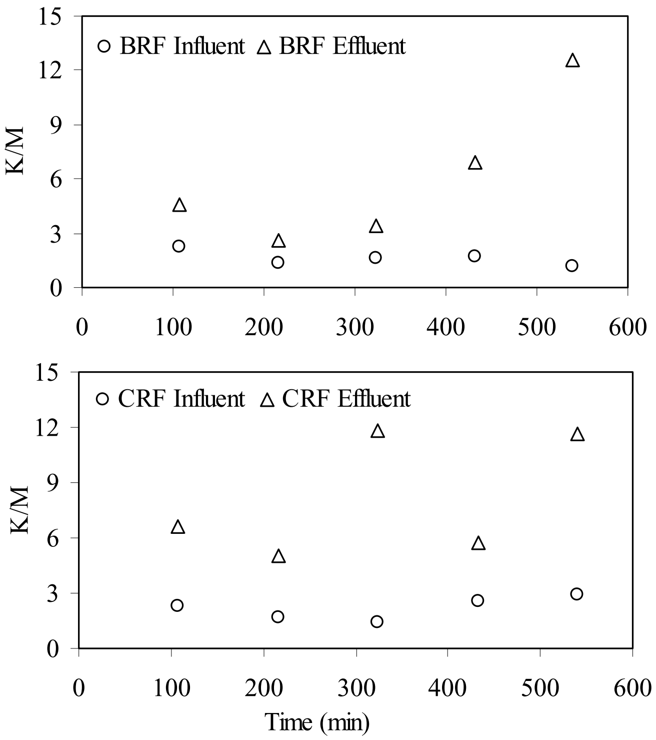

Figure 9 | Change of influent and effluent sample K/M ratios in ripened calcite and basalt roughing filters.

CONCLUSIONS

- Violations of the 1 NTU effluent turbidity limit were observed in slow sand filters pretreated by purely basalt or limestone roughing filters, where raw water clay turbidity >150 NTU. These pretreatment filter configurations were not considered appropriate for the Santiam River raw water source used in this pilot study.
- Headloss does not accumulate sufficiently in slow sand filters pretreated with roughing filters using limestone as the only filter media, and this was assumed to be a result of increasing water pH to a point no longer favourable to native aquatic biota. The design length of the calcite layer in amended roughing filters should be directly related to anticipated raw water pH and the potential for schmutzdecke microbial growth inhibition. Correlation of limestone layer design length and optimum filter performance merits further investigation.
- The use of limestone to augment a basalt roughing filter increased the clay turbidity removal efficiency more than 7% over an unaltered filter. Turbidity removal of the connected slow sand filter also increased more than 7%, and produced treated effluent from source water with turbidity ranging from <1 to 150 NTU, which complied with current regulatory requirements.
- Calcite-amended roughing filters pretreating slow sand filters increase effluent pH and alkalinity, which may enhance corrosion control of downstream distribution pipe systems.
- The effects of limestone dissolution and subsequent calcium release on clay removal in calcite-amended roughing filters may be most important during the filter ripening period before biological maturation.

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