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Removal of virus to protozoan sized particles in point-of-use ceramic water filters

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ABSTRACT

The particle removal performance of point-of-use ceramic water filters (CWFs) was characterized in the size range of 0.02–100 μm using carboxylate-coated polystyrene fluorescent microspheres, natural particles and clay. Particles were spiked into dechlorinated tap water, and three successive water batches treated in each of six different CWFs. Particle removal generally increased with increasing size. The removal of virus-sized 0.02 and 0.1 μm spheres were highly variable between the six filters, ranging from 63 to 99.6%. For the 0.5 μm spheres removal was less variable and in the range of 95.1–99.6%, while for the 1, 2, 4.5, and 10 μm spheres removal was >99.6%. Recoating four of the CWFs with colloidal silver solution improved removal of the 0.02 μm spheres, but had no significant effects on the other particle sizes. Log removals of 1.8–3.2 were found for natural turbidity and spiked kaolin clay particles; however, particles as large as 95 μm were detected in filtered water.

1. Introduction

Ceramic water filters (CWFs) are used for point-of-use water treatment around the world. CWFs are a low-cost technology that can be locally produced using naturally available clay soil and fine organic materials such as sawdust or rice hulls. The material is fired in a kiln burning away the organic material and leaving behind small pores. The pore sizes and surface charge of the ceramic determine the ability of the filter to remove particles and pathogens from the water. CWFs are typically coated with silver to provide an additional disinfection mechanism. However, because this silver leaches out of the filters over time, the long-term pathogen removal may be based primarily on the filtration characteristics.

The reported measured pore sizes in CWFs from the Potters for Peace (PFP) factory in Nicaragua, used in our study, range from 0.6 to 3 μm in areas of the filters without cracks (which have lengths up to 500 μm) (Lantagne, 2001); and 0.02–200 μm with 14 μm the predominant size (Van Halem, 2006). The pore sizes in ceramic disks produced in the lab from flour, grog, and clay were primarily 0.02–15 μm, with a few 100–490 μm (Oyanedel-Craver and Smith, 2008). These results indicate that filtration-based removal of viruses (<0.1 μm), should be poor, particularly since the bulk of the water is likely to flow through the cracks or larger pores.

Previous studies have measured the removal of protozoans, bacteria, and viruses by CWFs. In studies on CWFs without silver the microorganism removal can be attributed to filtration. Brown (2007) found 1.8–2.4 log removal and 1.3–1.9 log removal of *Escherichia coli* and MS2 bacteriophage, respectively, in laboratory studies. The removal of MS2 was not significantly different in filters with silver applied,

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perhaps because the long term nature of the study (600 L treated) minimized the impacts of silver. Four PFP filters without silver in Van Halem's (2006) study removed >99.99% of total coliforms in 85% of all samples taken over 12 weeks; in 6% of the samples the total coliform removal was 97–97.99%. In challenge tests with PFP filters without silver, log removals of 2–5.99, 2–5.99, and 1.06–2.31 were achieved for Clostridium spores, *E. coli*, and MS2, respectively. Silver coated CWFs achieved only 0.51–1.44 log removal of MS2. Oyanedel-Craver and Smith (2008) reported 97.86–99.97% removal of an *E. coli* spike in their filter disks, with higher removal efficiency in the filters with smaller median pore diameters.

While the expected trends of higher removal of pathogens with larger sizes have generally been observed, differences due to production and use history have not been evaluated. In this work we tested six filters from the first PFP factory in Managua, Nicaragua, that were subjected to more than four years of laboratory and/or field use prior to the current experiments. Fluorescent microspheres of varying sizes were used as surrogates for pathogens. Polystyrene latex microspheres ranging in size from 0.1 to 6 μm and with various surface modifications have previously been used in filtration studies as surrogates for bacteriophages, viruses, bacteria, and protozoans (Bales et al., 1997; Dai and Hozalski, 2003; Emelko and Huck, 2004; Hendricks et al., 2005; Hill et al., 2005; Långmark et al., 2005). Many of these studies found similar removal efficiencies of the bioagent and the microspheres. For example, Hill et al. (2005) achieved 86% recovery of 4.5 μm fluoresbrite polystyrene microspheres compared to 80% recovery of *Cryptosporidium parvum*. However, Hendricks et al. (2005) found 2.3–2.4 log removal of 6.0 μm latex microspheres compared to 3.2–3.4 log removal of *Cryptosporidium* and *Giardia*. Therefore, microspheres may be appropriate surrogates, but care should be taken to select ones with similar size, density, and surface charge to the pathogens of interest; these characteristics will impact filtration-based removal and are summarized in Table 1.

The objectives of this research were to characterize the physical removal of microspheres of different sizes, Kaolin clay and natural turbidity, and characterize the variability between filters. The role of the silver coating on particle removal was evaluated by retesting the same filters after recoating with silver.

2. Methods

2.1. Filters

The characteristics of the six filters produced at the PFP factory in Managua, Nicaragua, are summarized in Table 2. Two filters were purchased new with the silver coating and

Table 1 – Comparison of literature reported pathogen and surrogate characteristics.

Particle	Diameter, μm	Density, g/mL	Isoelectric point or point of zero charge	Zeta potential, mV
Poliovirus PV1	0.023[d]	~1.34[r]	6.6–7.15[d]	−20[q]
MS-2 bacteriophage	0.026[c] 0.030[f]	1.38–1.46[r]	3.9[c]	−0.8to13[f]
Escherichia coli	~1 × 3[p]	1.08–1.10[a]	3–4.2	−34 to−48[p]
Cryptosporidium parvum oocysts	4–7; mean 4.7[h]	1.025–1.07[h]	3.3[e] 2.18–3.90[g]	−38[e] −1.5 to−12.5[h]
Giardia cysts	9.3 × 12.2[b]	1.036[b]	2.2[e]	−17[e]
Kaolin clay	<2 to 15[j] 4.3 mean,[k]	2.60[j,k]	2.2[j] 4.3 [k]	−40[l]
Carboxylated polystyrene microsphere [n]	0.02, 0.10, 0.50, 1.0, 2.0	1.05	2.2–2.5[m]	−23 to −60 at pH 4–7[i]
Carboxylated polystyrene microspheres[o]	4.5, 10	1.05[h]	<2[c]	−7.4 to−50.2[h]

a Woldringh et al., 1981.
b Medema et al., 1998.
c Pontius et al., 2009.
d Schijven et al., 2003.
e Hsu and Huang 2002.
f Knappett et al., 2008.
g Brush et al., 1998.
h Dai and Hozalski 2003.
i Cail and Hochella 2005.
j Besra et al., 2000.
k Sawyer et al., 2001.
l Li et al., 2008.
m Menshikova et al., 2005.
n Molecular Probes.
o Polyscience.
p Soni et al., 2008.
q Kondo et al., 2004.
r Rowlands et al., 1971.

Table 2 – Characteristics of the tested filters.

Abbrev	Year made	Silver coating	Use history prior to current experiments	Avg Full flow rate, L/hr	Avg Effluent Silver, ppb	
					Pre recoat	Post recoat
1Lab	2002	Initially and recoated in 2003	Laboratory flow tests with dechlorinated tap water, turbidity removal, and natural particle size removal tests	1.4 ± 0.3	0.9	13
2Lab	2003	Initially	Turbidity removal tests in the lab	0.9 ± 0.2	2.4	12
1Field	1999	Initially	Field use by family until oven dried and shipped to CU in 2003	1.8 ± 0.3	0.20	11
2Field	1999	Initially	Field use by family until oven dried and shipped to CU in 2003; lab test treating 2 batches *Escherichia coli* spiked water	1.4 ± 0.2	0.05	2.1
1NS	2003	No	Turbidity removal tests in lab	1.5 ± 0.1	0.13	NA
2NS	2002	No	Flow tests in lab with dechlorinated tap water	2.0 ± 0.3	0.05	NA

NA = not applicable, since these filters were not recoated with silver.

used in a variety of laboratory experiments prior to the current study (1Lab and 2Lab). Two filters with silver coating were used for about four years by families in Nicaragua before being shipped to the University of Colorado in 2003 (1Field and 2Field). Finally, two filters without a colloidal silver coating were used in experiments prior to the current study (1NS and 2NS). The filters span the full range of the acceptable initial flow rates of 1–2 L/hr. Because the filters had been used in the lab and/or field for a number of years prior to the microsphere experiments, the majority of the silver had already leached out. After a first set of particle removal and later the microsphere removal experiments, the Lab and Field filters were recoated with colloidal silver, *E. coli* removal tests were conducted (Bielefeldt et al., 2009) and the microsphere and particle removal retested. Prior to all experiments the filters were pre-saturated with dechlorinated tap water to simulate continuous use conditions.

2.2. *Microsphere removal*

Fluorescent labeled carboxylate-coated polystyrene microspheres from Molecular Probes (Fluospheres F8784, 8800, 8888) and Polysciences (Fluoresbrite 16592–5, 18142–2) were used, with characteristics listed in Table 1. Individual sizes of the microspheres were dosed into dechlorinated tap water resulting in the initial concentrations shown in Table 3. The average characteristics of the tap water were: total organic carbon 1–4 mg/L, pH 6.5, and turbidity 0.12–0.28 NTU.

For each microsphere size the microsphere-spiked tap water was added to the CWFs (Fig. 1) and run through each filter. Samples were taken from inside the CWF reservoir and from the effluent, typically at 1.5, 2.5, 3.5 and 4.5 h. This was repeated two additional times for each microsphere size. For the experiments prior to silver recoating, each batch was made separately and run on three sequential days. After silver recoating, a single barrel of spiked water was used for all three batches to reduce variability, with one batch run the first day and two batches on the second day. Because the microsphere concentrations in the water inside the filter did not vary over time and were not significantly different than the spiked water (data not shown), the spiked water concentrations were used to compute the particle removal efficiencies. There were typically not significant differences in the effluent microsphere concentrations observed from a single filter at the different sampling time points or between the three batches of water (data not shown). Therefore, this data has been averaged together to determine comparisons between filters and microsphere sizes.

For each sample the pH, turbidity (HACH 2100 N Turbidimeter), fluorescence (F 3010 Fluorescence Spectrophotometer), water temperature, and water depth was measured. Four fluorescence measurements were made on each sample. The

Table 3 – Inlet microsphere concentrations and detection limits.

Average Sphere Diameter, μm	Fluorescence Excitation/ emission wavelength	Average spiked conc, #/mL		Minimum Detection Limit Range, #/mL
		Initial	Post silver recoat	
0.02	535/575	1.3 E10	9.1 E10	0.9–2 E8
0.10	540/560	1.2 E8	0.6 E8	0.1–3 E5
0.50	505/515	1.4 E6	2.8 E6	7–9 E3
1.0	505/515	8.6 E4	NT	180–449
2.0	505/515	8.1 E3	9.1 E3	3–11
4.5	441/486	1.3 E4	NT	2–7
10	441/486	NT	4 E3	2–5

NT = experimental condition not tested.

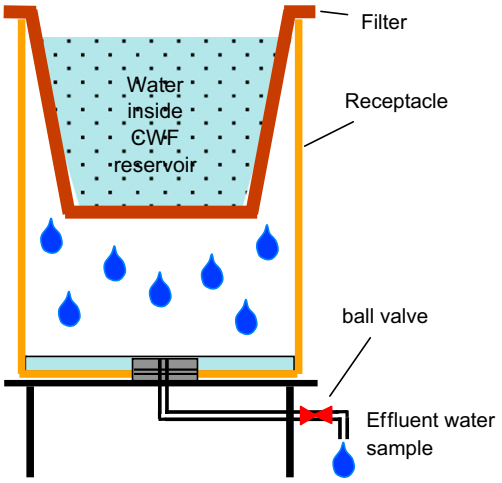

Fig. 1 – Experimental set-up.

minimum detection limit is shown in Table 3 and was different for each size of microsphere and each batch of water, due to variability in the fluorescence of the tap water. When particles in the effluent were not detected, the values were set to the detection limit to enable calculation of the lower limit of removal efficiency. Silver concentrations were measured according to US EPA Method 1638 using a Varian Inductively Coupled Plasma Mass Spectrometer (ICP-MS). The minimum detection limit was 0.004 ppb silver. The silver concentration in the tap water was variable, ranging from 0.01 to 0.79 ppb.

2.3. *Particle size characterization of natural turbidity and clay*

Two sets of experiments were conducted to evaluate the removal of larger, natural particles. Samples of water from Boulder Creek, CO, were evaporated in an oven to concentrate the suspended solids. This solution was spiked into dechlorinated tap water. When the 1Lab and 1NS filters were new after treating only ten batches of 10 NTU water, particle size characterization was conducted on the first and 8th batches of 40 NTU water treated. Samples of the spiked raw water (pre-treatment) and effluent water were measured for particle size using a MET ONE Liquidborne Particle Sensor (Model LB1020) with an Automatic Batch Sampler (Model 2500).

A second set of particle size experiments were conducted after all of the fluorescent microsphere tests described above. Lab-grade kaolin clay (254 mg) was added into dechlorinated tap water (50 L) to an initial turbidity of 3 NTU and loaded into all six filters. Samples from inside the filters and the effluent were taken at 1.5, 2.5, and 3.5 h, and measured for turbidity and particle size.

3. Results and discussion

3.1. *Microsphere results*

3.1.1. *Removal of 0.02 μm and 0.1 μm microspheres*

Spiked water containing 1–9 E10 particles/mL of the 0.02 μm spheres was treated by the six filters, with the resulting log removal efficiency summarized in Fig. 2. The filters were tested in three different sets: low silver due to prior long term testing; after recoating the lab and field filters with silver and treating approximately 30 batches of water; and after treating another ~20 batches of water. Silver was never applied to the NS filters. Removal by the NS filters was consistent for the three test cycles and served as a baseline to determine if silver application to the other filters had a significant impact. The reapplication of silver increased the 0.02 μm microsphere removal in three of the four filters (2-tailed heteroscedastic *t*-tests comparing log removal data from each sampling time: $p < 0.01$ for 1Lab and 2Field filters; $p = 0.07$ for 2Lab filter). However, the removal efficiency of these three filters declined with time (p values < 0.03). The exception to these trends is the 1Field filter, which behaved more similarly to the 2NS filter.

The large error bars on the data are due to variability across the three sequential test batches. The results in the 3rd set of tests are the least variable, perhaps because of the stable silver concentrations during these tests. In the first set of experiments with low silver, there was a trend to an increased removal over the 3 batches in three of the filters ($p \leq 0.03$; data not shown). In the experiments conducted after the filters were recoated with silver there was a decrease in removal efficiency over the three batches in four of the filters ($p \leq 0.06$), which could correlate to the decreasing silver. For example, batch 2 tested during this period had effluent silver concentrations of 22 ppb from the Lab filters, compared to only 6–10 ppb effluent silver by batch 19. Similar decreases in effluent silver concentrations were observed in the Field filters.

Previous tests to characterize removal of MS2 bacteriophage found 1.06–2.31 log removal by CWFs without silver (Brown, 2007; Van Halem, 2006); this is similar to the treatment efficiency of the 0.02 μm microspheres in this study. Silver-coated filters achieved only 0.51–1.44 log removal in VanHalem's study. This is the opposite of our results which found generally improved removal shortly after silver recoating. Brown found no significant differences in filters with silver, perhaps due to the longterm nature of the study which washed out a significant amount of the silver. This is similar to our later post silver recoating experiments where treatment effectiveness declined in the lab filters.

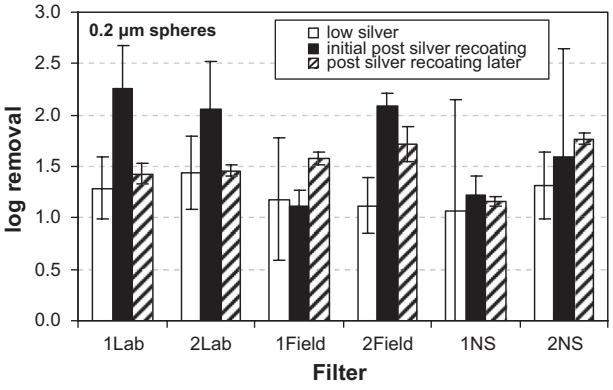

Fig. 2 – Log removal of the 0.02 μm microspheres by the six CWFs. Error bars represent the range in removal across the 3 sequential test batches of water treated.

After treating three batches of spiked water, a clean water batch was run through the filters. The 0.02 μm microspheres were found at concentrations of 6.5E8 to 2.0E9 microspheres/mL in the treated water, indicating that they detached from the filter. These effluent concentrations were 16–61% of the effluent microsphere concentrations measured from the previous batch of microsphere-spiked water. The in-filter samples were clean for 5 of the 6 filters, indicating little desorption from the inside surface of the filters. Therefore, it is likely that the microspheres were weakly associated with the pore walls and detached into the clean water. Similar results of contamination of clean water treated after spiked tests have been observed with *E. coli* (Bielefeldt et al., 2009).

Overall, the measured removal of the 0.1 μm spheres was no different than the 0.02 μm spheres in the low silver tests, which is counterintuitive. However, there was a higher degree of variability in the 0.1 μm spheres results. Removal of the 0.1 μm spheres was not enhanced after the reapplication of silver (data not shown). Perhaps the 0.1 μm spheres are too big for electrostatic removal mechanisms that were significant for the 0.02 μm spheres.

3.1.2. *Removal of larger microspheres and of* E. coli

A summary of the average removal efficiency of all of the microsphere sizes is shown in Fig. 3. For the larger spheres, there were minimal differences between the treatment effectiveness of the different CWFs and regardless of silver recoating. However, detection limitations of low microsphere concentrations may have prevented these experiments from determining these differences. As expected, on average the removal efficiency increased with larger particle sizes, with: log removal = 0.7228 log (microsphere size in μm) + 2.4439 ($R^2 = 0.92$). The *E. coli* removal by these six CWFs was previously characterized (Bielefeldt et al., 2009). These data from the low silver tests are compared to the microsphere results in Fig. 3. Unlike the particles, the *E. coli* removal generally declined significantly as three sequential batches of highly contaminated water were treated; across the six filters the *E. coli* disinfection in the first batch ranged from 2.9 – 4.1 log, compared to 0.3–4.4 log in batch 3 (data not shown). The average of the three batches is shown in Fig. 3. The average *E. coli* removal by the CWFs ranged from 2.0 to 4.2 log compared to >2.5–3.0 log removal of the similarly sized 1–2 μm microspheres. Therefore, it appears that the microspheres provide a reasonable estimate of the filtration-based removal of *E. coli* bacteria.

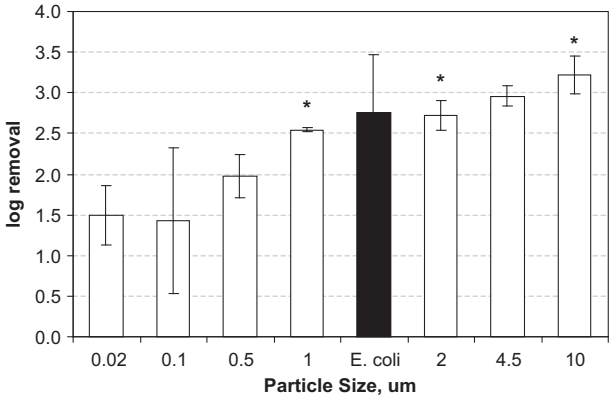

Fig. 3 – Average log removal of the microspheres and *Escherichia coli* by the CWFs. The error bars represent the standard deviation of the removal by all six filters across multiple batches and sampling time points. The * over some bars indicates that more than half of the effluent data was below the detection limit, so the actual removal efficiency is likely higher.

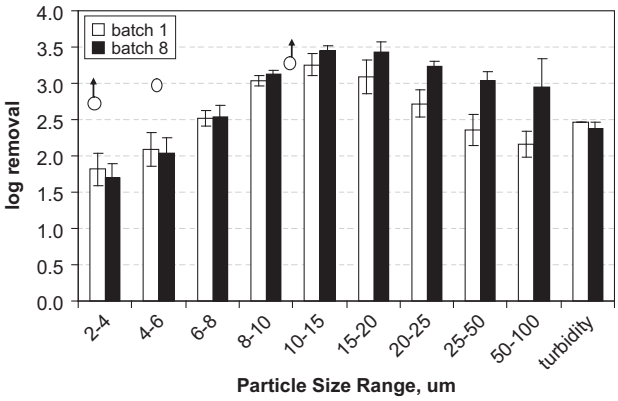

Fig. 4 – Log removal of different natural particle size ranges in the first and eighth batch of 40 NTU water treated. Error bars represent the removal range by filters 1Lab and 1NS. The oval symbols represent the log removal of the 2, 4.5, and 10 μm diameter microspheres, with upward arrows indicating that due to detection limits the actual removal efficiency is higher than the value shown.

3.2. *Removal of natural and clay particles greater than 2 μm*

In the first set of particle removal experiments using water spiked with 40 NTU of natural turbidity, the 1Lab and 1NS CWFs were new and had only previously treated ten batches of 10 NTU water. Averaged results of the two filters are summarized in Fig. 4. Equal removal by these two filters was found, except for the smallest particles, 2–7 μm, in which the 1Lab filter performed slightly better than the 1NS filter (data

Table 4 – Average concentration of particles of different sizes in the raw water prior to treatment by the CWFs.

Size Range, μm	Natural Particles		Kaolin Clay	
	# Particles/mL	Percent of total	# Particles/mL	Percent of total
2–4	13,219	12.01	1770	45.45
4–6	8778	7.97	821	21.08
6–8	25,926	23.55	914	23.47
8–10	24,696	22.44	222	5.70
10–15	28,167	25.59	124	3.18
15–20	6935	6.30	28	0.72
20–25	1560	1.42	9	0.23
25–50	737	0.67	6	0.15
50–100	54	0.05	0.3	0.01
TOTAL: 2–100	110,072		3894	

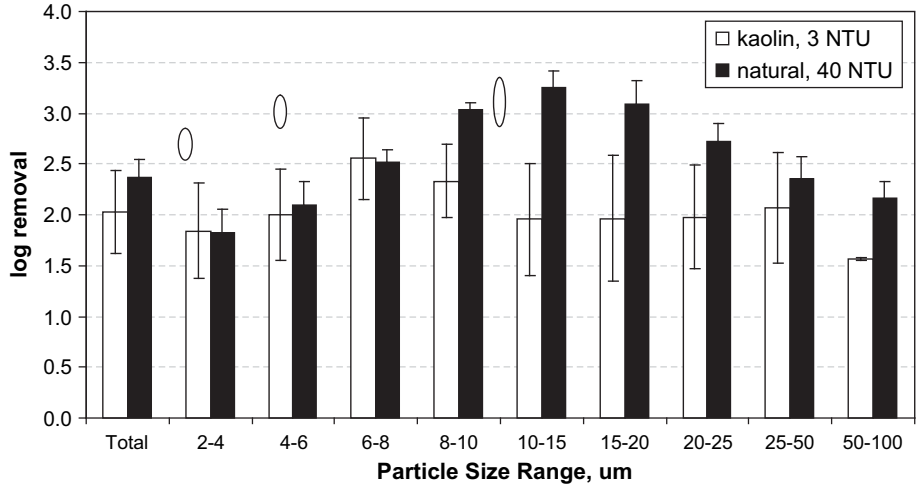

Fig. 5 – Comparison of the removal of lab-grade kaolin clay and natural surface water particles by the 1NS and 1Lab filters in the first batch of treated water. The oval symbols indicate the average ± the standard deviation of the log removal of the 2, 4.5, and 10 μm microspheres.

not shown). For these small particles, higher removal efficiency was achieved in batch 1 compared to batch 8. This is counterintuitive because the build-up of a cake layer of particles removed from the water over time should improve the particle removal efficiency. The average removal efficiency increased with increasing particle sizes up to 15 μm and then began to decrease. This drop in removal efficiency of the largest particle sizes may be caused by the lower concentration of particles > 15 μm (<9% of total particles), as shown in Table 4. Because particles as large as 95 μm were present in the effluent water, at least some of the pore spaces in the filters were larger than this diameter. The overall particle removals were very similar to the measured turbidity removal. The median and mode of particle sizes in the raw water were 8–9 μm and 10–15 μm, respectively, compared to 3–4 μm and 2–3 μm in the treated water. As expected, the larger particles were preferentially removed by the CWFs. Also shown on Fig. 4 is the removal of the 2, 4.5 and 10 μm microspheres, which indicates that the removal of small microspheres was about a log higher than the natural particles, while similar removals were found for 10 μm particles.

In the second set of experiments, kaolin clay was used as the particle source and added at a lower concentration (see Table 4). The total number of clay particles in the raw water was only ~3900/ml (3 NTU) compared to 110,000/ml (40 NTU) natural particles. The clay particles (median diameter 4–6 μm) were smaller than the natural turbidity (median diameter 8–10 μm), with more than 99% less than 20 μm. Despite these differences, the removal of the kaolin particles less than 8 μm by the 1Lab and 1NS filters was similar to that for the natural turbidity in the creek water, as shown in Fig. 5. For particles greater than 8 μm, there was significantly less removal of the clay. This may be caused by the rapid build-up of a filter cake with the high turbidity water which increased the removal of the natural particles. There was significant variability in the clay removal abilities of the six CWFs as indicated by the error bars. The 1NS filter achieved the best treatment efficiency for all of the particle size ranges. The removal efficiency varied from 1.2 to 3.3 log for different filters and particle sizes. The low removal observed for the >50 μm particles may be biased by the small amount of these particles in the untreated water.

The removal of 10 μm microspheres was similar to the removal of the 9–15 μm natural particles ($p = 0.71$) and higher than the clay particles ($p < 0.01$). The removal efficiency of the 2 and 4.5 μm microspheres was significantly higher than the removal efficiency of the natural and clay particles (p values < 0.01). Based on the physical characteristics of pathogens, microspheres, and kaolin that were summarized in Table 1, the clay has a significantly higher density which should lead to increased filtration-based removal via sedimentation; the isoelectric point indicated that all types of particles should have been negatively charged at the pH of these experiments and zeta potentials are fairly similar. Hendricks et al. (2005) also found that filters removed about 1 log more *Giardia* and *Cryptosporidium* (~3.3 log) compared to natural surface water particles of similar size (~2.3 log), with removal of 6 μm latex microspheres the highest at ~4 log. Therefore, the attachment efficiency of the microspheres appears much higher than clay or natural particles.

4. Conclusions

The ability of six CWFs to remove submicron (0.02, 0.1, and 0.5 μm) diameter microsphere varied significantly. Higher silver generally increased the removal of the virus-sized 0.02 μm microspheres. The expected trend of better removal of larger microspheres was observed, although in many cases the effluent water contained undetectable levels of microspheres which limited the ability to accurately quantify removal efficiencies. The removal of 1 and 2 μm microspheres by the CWFs was similar to average *E. coli* removal, indicating that the microspheres appear to be a reasonable surrogate. The CWFs removed a higher percentage of the 2 and 4.5 μm microspheres than the removal of similarly sized natural turbidity and kaolin particles. The CWFs removed 10 μm

microspheres at a similar efficiency as natural turbidity of similar size, and these were both significantly higher than the removal of kaolin particles. The varied surface properties of the particles may account for these differences. The results indicate that it may be possible to use simple surrogates to model filtration-based pathogen removal in CWFs.

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