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Fouling in hollow fiber membrane microfilters used for household water treatment

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ABSTRACT

The Sawyer PointOne hollow fiber membrane microfilter is promoted for household water treatment in developing countries. Critical limitations of membrane filtration are reversible and irreversible membrane fouling, managed by backwashing and chemical cleaning, respectively. The PointOne advertised lifespan is 10 years; users are instructed to backwash as maintenance. Owing to reduced turbidity and bacterial removal efficiencies, six PointOnes were removed from Honduran homes after 23 months of use. In the laboratory, we tested sterile water filtrate for turbidity and bacterial presence before and after backwashing and chemical cleaning. Sterile water filtrate from uncleaned filters had turbidity of 144–200 NTU and bacteria counts of 13–200 CFU. Cleaned filter effluent was positive for total coliforms. On one new and one used, cleaned filter, we imaged membranes with scanning electron microscopy and characterized surface elemental compositions with spectroscopy. Images and spectroscopy of the used, cleaned membrane revealed a dense, cake fouling layer consisting of inorganic metal oxides, organic material, and biofouling. Burst fibers were visually observed. This PointOne was thus irreversibly fouled and non-functional after <2 years of use. Further research is recommended to determine: impacts of source water quality on PointOne performance, a cleaning regimen to manage fouling, and an appropriate filter lifespan.

Key words | drinking water, filter fouling, hollow fiber membrane, household water treatment, microfiltration, point-of-use

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ABBREVIATIONS

WHO	World Health Organization
HWTS	Household water treatment and safe storage
PWW	Pure Water for the World
CFU	Colony forming unit
E. coli	*Escherichia coli*
SEM	Scanning electron microscope
TSA	Trypticase soy agar
EMB	Eosin methylene blue
MAC	MacConkey agar
EDS	Energy dispersive spectroscopy
NTU	Nephelometric turbidity units
NOM	Natural organic matter

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INTRODUCTION

Worldwide, an estimated 780 million people drink water from unimproved sources (UNICEF/WHO 2012) and an estimated 1.2 billion more drink contaminated water from improved sources (Onda *et al.* 2012). Providing reliable, centrally treated piped water to every household is the ultimate goal, but the World Health Organization (WHO) also supports incremental water supply improvements – such as household water treatment and safe storage (HWTS) options – to accelerate the health gains associated with safer drinking water for those with unsafe supplies (WHO 2011b).

In 2014, to address recent increased interest in developing new HWTS options, WHO launched an international scheme to evaluate HWTS product laboratory performance in removing bacteria, viruses, and protozoan cysts that cause diarrheal disease (WHO 2014a). This scheme established tiered, health-based targets, classifying HWTS products as 'highly protective' (4-log bacteria and protozoa reduction and 5-log virus reduction), 'protective' (2-log bacteria and protozoa reduction and 3-log virus reduction), or 'limited protection' (achieving protective target for two pathogen classes and epidemiological evidence demonstrating disease reduction) based on a quantitative microbial risk assessment model (WHO 2011a).

The Sawyer PointOne Filter (PointOne) is a microfilter consisting of hollow membrane fibers bundled in a U-shape inside a plastic casing (Sawyer Products Inc., Safety Harbor, FL, USA). The PointOne is promoted for recreational use, disaster relief, and HWTS in developing countries (Sawyer Products 2014b; Sawyer Products n.d.). For household use, users attach the PointOne in-line with a delivery hose to a 20-liter bucket. Water flows via gravity into the filter casing inlet, through the 0.1 μm porous hollow fiber membrane walls into the membrane cores and exits the casing into a second storage container (Figure 1). Users are instructed to backwash the filter when flow slows, using the provided syringe and clean water. The filter lifespan is advertised as '10 + years', 'decades', '1 million gallons', and even potentially 'never need [ing] to be replaced', with a maximum daily throughput of 1,117 liters at sea level. PointOne filters have been distributed in over 70 countries worldwide (Sawyer Products 2014a; Sawyer Products 2014b; Sawyer Products n.d.).

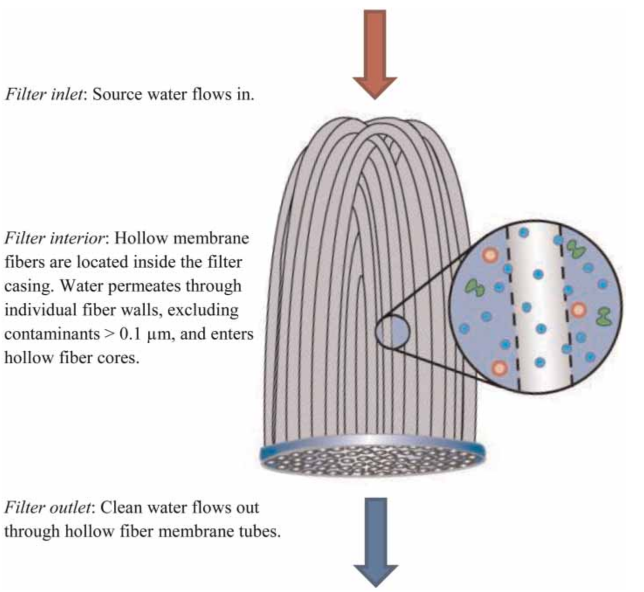

Figure 1 | Diagram of Sawyer PointOne filter interior membrane and filtering mechanism (modified diagram courtesy of Sawyer Products).

Documented benefits of the PointOne include that, in the laboratory setting, the PointOne is efficacious at removing bacterial (>6-log reduction) and protozoal (>5-log reduction) organisms that cause diarrheal disease (Hydreion 2005). In addition, in users' homes, the PointOne is simple to operate and maintain. High acceptance and usage – up to 100% over 3 months – has been documented during short-term follow-up (Give Clean Water 2009; MAP International 2011; Brune *et al.* 2013; Goeb 2013d).

However, documented potential shortcomings of the PointOne include filter blockage and breakage, lost or broken syringes for backwashing, requirement of clean water for backwashing, and low consistent use over longer periods of time (MAP International 2011; Goeb 2013b; Kohlitz *et al.* 2013). In one 3-year study, 52% of users reported consistent filter use, and 32% of filters were in disuse due to lost or broken parts (Kohlitz *et al.* 2013). In addition, field effectiveness data have found bacterial contamination in 18–54% of tested filter effluent water and 51–70% of stored, filtered water in studies ranging from 3 months to 3 years of use (Brune *et al.* 2013; Goeb 2013b, 2013c; Kohlitz *et al.* 2013).

Membrane filtration is an emerging technology used in biomedical, food service, wastewater, and drinking water treatment; it is appropriate for HWTS because it provides a physical barrier, removes turbidity, and can improve water taste (Peter-Varbanets *et al.* 2009). The largest obstacle to filter performance in all applications is membrane blockage, or fouling. Fouling is caused by organic, inorganic, and bacterial constituents and leads to loss of membrane permeability, observable by declining flow rate through the filter. Fouling behavior is complex and depends on solution chemistry, membrane characteristics, filter operating conditions, and physical and chemical properties of the foulants. Membrane fouling can be 'reversible', where particulate material retained in a 'cake' layer on membrane surfaces is removable by physical processes such as backwashing and air scouring, or 'irreversible' where solutes plug and adsorb to pores within the membrane, requiring other processes – such as chemical cleaning – to recover performance (Zularisam *et al.* 2006).

Pure Water for the World (PWW) is a non-governmental organization that provides safe drinking water, sanitation, and hygiene education to communities in developing countries. PWW installed over 200 PointOnes in six rural Honduran communities as a pilot between 2010 and 2013. Beneficiaries were trained on filter use and maintenance upon installation, and again during household follow-up visits 2 months after installation. Usage rates ranged from 50–95% 9–13 months after distribution to 66–68% 23 months after distribution (Goeb 2013a). Reported reasons for filter disuse included: broken casings, clogged filters, broken or missing syringes, damaged hoses, casings which had been opened by users, and filters abandoned by users (Goeb 2013b, 2013c, 2013d).

In one community, 52% of the 29 filters tested after 23 months of use produced effluent with >10 colony forming units (CFU) *Escherichia coli* (*E. coli*) per 100 ml, which is considered intermediate to high health risk (WHO 1997). Six of these filters had demonstrated >99.6% mean *E. coli* and 98–99% mean turbidity removal efficiencies when tested shortly after distribution in October 2011, but 21 months later demonstrated only 54% mean *E. coli* and 59% mean turbidity removal efficiencies, with no visible damage to the filters (Goeb 2013b).

In this study, we present the results of controlled laboratory testing completed to investigate reduced performance of these six filters, including bacteria and turbidity testing of filtered sterile water effluent following manufacturer-recommended cleaning, to identify biofouling; and scanning electron microscope (SEM) imaging and elemental analysis of membrane fouling layers, to characterize the fouling layer and its constituents.

METHODS

Six PointOne filters were removed from homes in Trojes, Honduras in September 2013, stored in a sealed plastic bag, and transported to the University of Maine in Orono, Maine, USA, and investigated in November 2013. A new PointOne was purchased from a Sawyer retail distributor to serve as a control.

Filtrate bacteria and turbidity testing

Thirty milliliters of sterile water was pipetted through each filter. Filtrate turbidity was measured with a Lab Quest turbidimeter (Vernier Software and Technology, Beaverton, OR, USA), and each filtrate was swabbed and streaked onto a trypticase soy agar (TSA) plate. Plates were incubated at 37 °C for 48 hours, and bacteria colonies were counted. Colonies were considered too numerous to count above 200 CFU.

Sawyer Products was contacted for information on membrane cleaning procedures, beyond backwashing specified in product literature, recommended to restore filter flow. Per recommendations, all six used filters were soaked in hot water for 30 minutes and backwashed four times with 60 ml deionized water, then soaked for 30 minutes in 5% white distilled vinegar and backwashed with deionized water four additional times (John Smith, personal communication, Sawyer Products). Additional sterile water was filtered through each unit. The visually least-turbid filter effluent was chosen for microbiological assessment using the membrane filtration method; 100 ml was filtered through a 0.45 μm membrane filter, the filter was incubated at 37 °C for 48 hours on a TSA plate, and colonies were counted.

To identify unexpected live cultures, effluent from the new filter and two used filters with low turbidity was swabbed and streaked onto an Eosin methylene blue (EMB) plate to determine total coliform presence, and a MacConkey agar (MAC) plate to determine fecal coliform presence. Plates were incubated at 37 °C for 48 hours, and clonal growth described. A MUG-agar plate was inoculated from the EMB and MAC plates of both used filters to determine *E. coli* presence. The MUG plates were incubated at 37 °C for 48 hours and colonies counted.

Membrane imagery and surface elemental analysis

The used filter with highest effluent bacteria count and a new PointOne filter were each cut open at the inlet side, visually examined, and photographed. One membrane fiber from each filter was removed and imaged with a Zeiss NVision 40 SEM (Carl Zeiss, Jena, Germany) at increasing magnification levels. Samples were imaged uncoated near the second crossover voltage to minimize charging effects.

The used and new filter imaged as described above were stored at the University of Maine, and then sent to Tufts University in April 2014, and imaged with a Phenom G2 Pure Tabletop SEM (FEI, The Netherlands). Membranes were frozen in liquid nitrogen and fractured using a microtome blade for cross-sectional imaging and cut with a razor along the hollow core to image interior membrane surfaces. Samples were sputter-coated with gold (~1 nm) to prevent charging and beam damage.

Energy dispersive spectroscopy (EDS, Bruker, Germany), integrated into a Hitachi TM-3000 tabletop SEM (Hitachi High-Technologies Corporation, Tokyo, Japan) was used to characterize elemental composition of inner and outer membrane surfaces (top 1–10 μm) from each filter. Uncoated samples were used in low vacuum charge-up reduction mode, and spectra were collected for 60–90 seconds to obtain good signal-to-noise ratio.

RESULTS AND DISCUSSION

Filtrate bacteria and turbidity testing

When first flushed with sterile water, the new filter effluent measured 0.1 nephelometric turbidity units (NTU), and the effluent from five of six used filters ranged from 114 to >200 NTU (Table 1). One used filter (Filter #7) accepted 10 ml, but produced no effluent. There was no bacterial growth on the TSA plate for the new filter effluent; used filter effluent ranged from 13 to 18 CFU, with one plate too numerous to count (Table 1). Soaking and backwashing restored flow in the blocked filter, but effluent from all used, cleaned filters was visually turbid. Effluent from Filter #4 was visually least-turbid (10 NTU), suggesting that it may have been the cleanest of the filters; however, analysis by membrane filtration showed confluent colony growth on a TSA plate, indicating bacterial presence in filtered sterile water effluent.

Table 1 | Turbidity and bacterial growth in sterile water filtered through Sawyer PointOne filters. Filter 1 is a new filter, and Filters 2–7 were removed from households 23 months after distribution

Filters number	Turbidity of filtrate (NTU)	Bacterial growth of filtrate swabbed on TSA plate (CFU)
1	0.1	0
2	>200	18
3	>200	15
4	114	14
5	168	13
6	>200	TNTC
7	–	–

TSA: trypticase soy agar; Filter #7: water did not pass through filter, although 10 ml was introduced; TNTC: too numerous to count; turbidimeter detection limit 200 NTU.

There was no bacterial growth on the EMB or MAC plates from the new filter's effluent, indicating the absence of total coliforms and fecal coliforms. Plates from both used filter effluents showed dark pink lactose(+) growth on the EMB plates, and light pink presumptive of lactose (+) growth on the MAC plates, indicating potential total coliforms in effluent from both used, cleaned filters. MUG-agar plates of these two filter effluents exhibited no fluorescence, indicating the absence of *E. coli* in effluent from the cleaned filters.

Membrane imagery and surface elemental analysis

Visual inspection of the cleaned, used filter interior (Filter #7) showed discolored membrane fibers and high sediment build-up as compared to the new filter (Figure 2). Membrane fibers from the used filter were brittle, in contrast to new filter fibers, which were flexible and difficult to break. In addition, several fibers appeared to have broken, potentially allowing water to enter the hollow fiber tubes directly, without filtration through porous fiber walls.

In SEM imagery of a membrane fiber removed from the new filter, individual open pores can be observed; but those pores are blocked by a cake layer in the used, cleaned filter membrane (Figure 3). The inner surface of the hollow membrane fiber (Figure 4(a)) is highly porous, with large circular voids and a larger effective pore size. Deposits were observed on these inner pores of the used membrane (Figure 4(b) and 4(c)). Observation of cross-sectional membrane fiber images confirms the presence of a thick cake layer on the exterior, and a thin, but dense, cake layer and particles on the inner fiber surface (Figure 4(d)–4(f)).

EDS of the new filter's membrane surface identified carbon, oxygen, and sulfur, as expected for polysulfone or

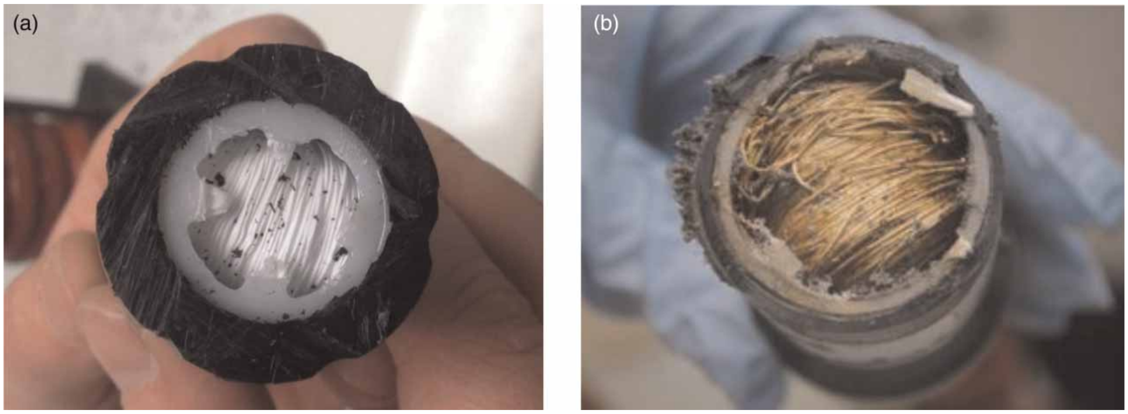

Figure 2 | (a) Interior of inlet end of a new Sawyer PointOne filter, showing the looped ends of the hollow membrane fibers. (b) Interior of inlet end of a Sawyer PointOne filter removed from the field after 23 months of household use and cleaned in the laboratory. Filter interior shows discoloration and sediment build-up indicative of membrane fouling.

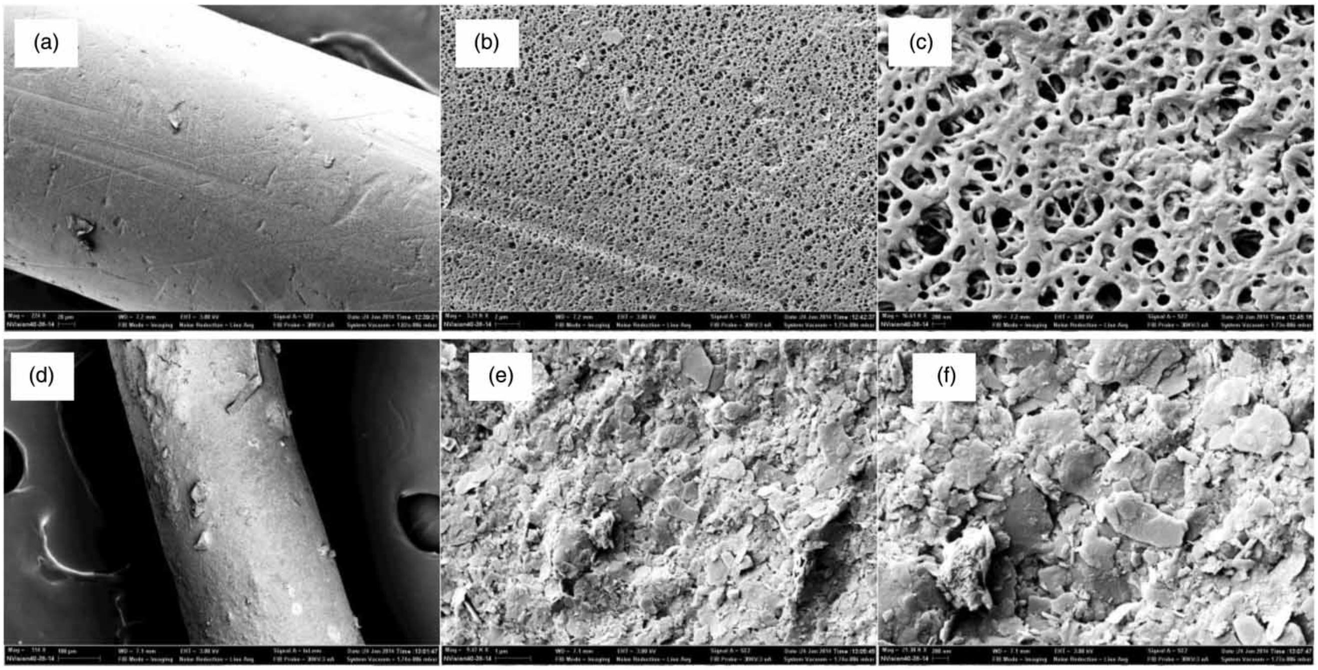

Figure 3 | SEM images of a new Sawyer PointOne filter hollow fiber membrane (a–c), and a membrane from a PointOne removed from the field after 23 months of household use and cleaned in the laboratory (d–f), showing a fouling layer. Images at increasing magnification [(a) 224 ×, (b) 3,210 ×, (c) 16,610 ×, (d) 114 ×, (e) 9,470 ×, (f) 21,300 ×].

polyethersulfone membranes typically used for water treatment (Table 2). Nitrogen was also observed on the inner surface, possibly from a preservative or adhesive used in manufacture. The used filter's membrane surface showed little carbon and sulfur, key elements found in the base membrane, but contained large amounts of oxygen, silicon, aluminum, iron, and lead; and lesser amounts of calcium, potassium, and magnesium on the outer surface (Table 2). The inner membrane surface contained a significant amount of lead, and other elements (Table 2), indicating fouling penetrating into the hollow fiber membrane interior.

Discussion

In this laboratory investigation of six Sawyer PointOne filters removed from the field after 23 months of use and

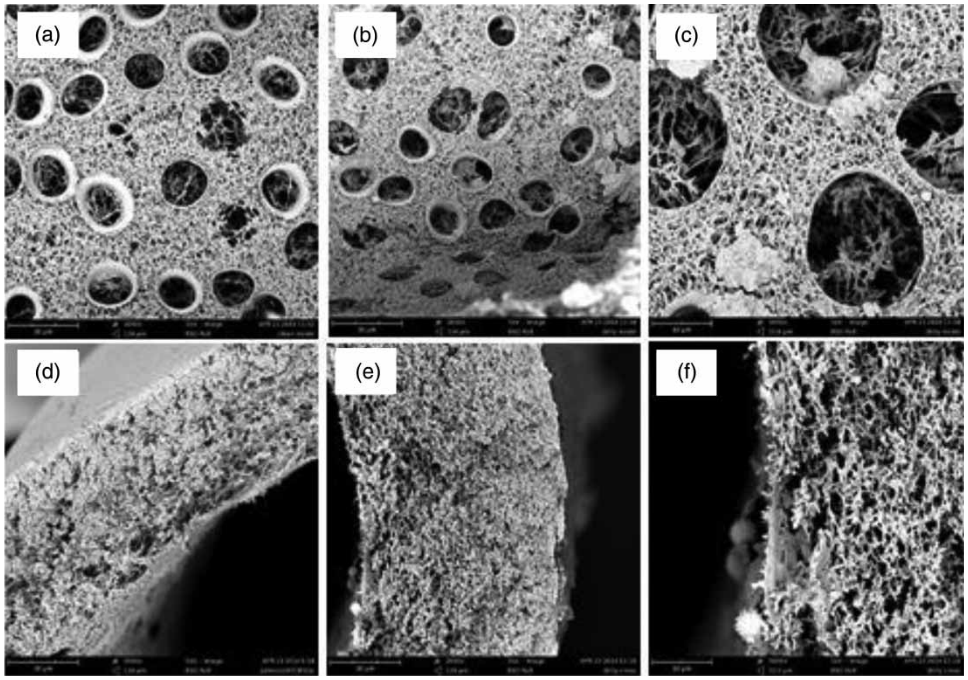

Figure 4 | SEM images of Sawyer PointOne filter hollow fiber membranes. (a) Inner surface of a new membrane (2,000 ×), (b–c) inner surface of membranes removed from the field after 23 months of household use, showing fouling within the inner pores of the membrane [(b) 2,000 ×, (c) 5,000 ×], (d) cross-sectional image of the new membrane (2,000 ×), (e–f) cross-section of used membranes as above, showing fouling layer on the outside and particles on the inner surface [(e) 2,000 ×, (f) 5,000 ×].

Table 2 | Elemental surface composition of a new PointOne filter membrane, and a membrane from a filter removed from a household after 23 months of use and cleaned in the laboratory

	Normalized weight %			
Elements	**New membrane, outer surface**	**New membrane, inner surface**	**Used membrane, outer surface**	**Used membrane, inner surface**
Carbon	75.4	70.0	19.6	58.5
Oxygen	13.3	19.8	34.9	15.1
Sulfur	6.4	8.8	2.2	14.4
Nitrogen	0.0	1.5	0.0	0.0
Silicon	0	0	8.2	1.4
Aluminum	0	0	6.6	1.2
Iron	0	0	4.4	0.7
Lead	0	0	1.8	8.0
Potassium	0	0	0.8	0.2
Calcium	0	0	0.5	0.4
Magnesium	0	0	0.4	0.1

cleaned with physical and chemical processes per manufacturer recommendations, we observed: (1) filtered sterile water exiting with turbidity and bacteria loading; (2) pore blockage by a fouling layer of inorganic metal oxides and organic matter on exterior and interior membrane fiber surfaces; and (3) brittle and burst membrane fibers. These results indicate irreversible membrane fouling, including biofouling, and potential short-circuiting of unfiltered water within this membrane. It is not known if these conditions represent an isolated incident or are indicative of an endemic problem with the PointOne in Honduras or other developing country settings; however, the results raise three concerns: (1) the Sawyer PointOne filter's applicability for treating source waters of varying quality, (2) appropriate filter membrane cleaning procedures, and (3) the filter's useful life span.

Membrane fouling depends on interrelated water quality parameters including, but not limited to: turbidity; particulates; organic content; biofilm-forming bacteria; hardness; and metal ions such as iron, manganese, and lead (Alpatova *et al.* 2004; Peng *et al.* 2004). Irreversible fouling occurs when organic biomacromolecules, such as proteins, humic acids, and polysaccharides, adsorb to the membrane (Kimura *et al.* 2004). Some of these compounds, such as natural organic matter (NOM), are naturally present in surface water; others are generated by organisms in the water source. Biomacromolecules can (1) bind together inorganic particulates, exacerbating cake fouling and preventing removal by physical methods (Schafer *et al.* 1998), and (2) initiate biofouling by helping microorganisms in the influent water adhere to the membrane surface. These microorganisms can then grow and form impermeable biofilms (Peng *et al.* 2004). Irreversible fouling can become increasingly difficult to manage if not remedied early in the biofilm formation process.

The used filter membrane analyzed herein contained a complex mixture of metal oxides on the outer membrane surface, with especially high quantities of silicon, aluminum, and iron (Table 2). Positive bacteria presence in sterile water effluent from used filters suggests membranes were biofouled. This indicates the irreversible fouling layer is likely a composite of inorganic particles held together by organic foulants and/or microorganisms, and that various source water constituents contributed to filter membrane fouling.

Consistent with common microfiltration fouling prevention techniques, Sawyer Products recommends two methods to minimize PointOne fouling: pretreating source waters and backwashing when flow is reduced (Sawyer Products 2014a). Chemical cleaning instructions were only available upon request. Some implementing organizations also recommend pretreatment of turbid source water by filtration, sedimentation, or coagulation with locally sourced alum before PointOne use, and backwashing the filter with each use, regardless of whether flow is blocked (Brune *et al.* 2013; MAP International 2011).

Results presented herein demonstrate that operation and maintenance of the PointOne is essential, as seen with other HWTS options, but cleaning according to manufacturer's instructions is not always sufficient. The six poorly performing PointOnes were removed from households where users received filter operation and maintenance instruction, demonstrated correct knowledge of backwashing procedure, and self-reported backwashing with adequate frequency (Goeb 2013b). With the exclusive use of backwashing, all commercially available filter membranes will eventually foul irreversibly, resulting in progressively reduced flow (Guo *et al.* 2012). Irreversible fouling is controllable by chemical cleaning with acidic, alkaline, or biocide solutions (Gao *et al.* 2011). A membrane cleaning regimen should be chosen in accordance with known water parameters; for example, backwashing to partially remove cake layers on membrane surfaces, alkaline solution to remove microorganisms and organic material, and acidic cleaning to remove inorganic scale (Mo & Huanga 2003).

The PointOne filter's membrane structure, which features small pores on the exterior (Figure 3), and large circular voids with porous walls on the interior (Figure 4(a) and 4(d)), is a common structure that sustains high permeability while offering mechanical support. Its outside-in membrane configuration, where microorganisms and particulates are retained on the surface and purified water permeates into the hollow interior, provides a large surface area. However, during backwashing, this geometry concentrates mechanical stress where U-shaped membrane fibers attach to the module. PointOne backwashing instructions encourage users to 'be forceful' to dislodge the cake layer. While this approach is sound when the fouling mode is reversible and the cake layer is loose, in the presence of extensive irreversible fouling, forceful backwashing may push fibers to their burst pressure. Broken membrane fibers could lead to improved flow mistaken for the successful removal of the cake layer, when in fact it allows short-circuiting of influent water and loss of turbidity and bacterial removal. After this point, the filter will no longer filter microorganisms, and may instead act as a reservoir for biofilm-forming bacteria. If PointOne membrane fibers burst, functionality cannot be restored without filter replacement and there is no external indication to the user that PointOne use should be discontinued.

The Sawyer PointOne has not yet been assessed under the WHO HWTS product evaluation scheme, but based on available evidence, the PointOne could meet WHO requirements for the 'limited protection' classification. The results presented herein highlight the need to test products with representative water sources (specifically with high turbidity, hardness, and NOM), complete the recommended 'clogging point' sample (WHO 2014b), and evaluate product longevity and end-of-life indicator mechanisms before classifying a HWTS product.

Limitations of this work include that few filters were analyzed, and the lack of source water testing beyond turbidity and bacteria. Source water in the homes from which the poorly performing PointOnes were removed had mean turbidity of 62 NTU (range 7–87 NTU), as measured in stored, untreated water in the home at the time of filter testing (Goeb 2013b), and may not be representative of source waters in other settings where PointOnes are recommended for HWTS. In addition, although users were trained in filter operation and maintenance, self-reported user behavior cannot be verified. As such, we cannot isolate source water characteristics that contributed to filter membrane fouling, determine the extent of irreversible fouling in PointOne distributions, or know the extensibility of these results to situations with different water sources or program implementation.

The Sawyer PointOne microfilter has been shown to be effective at removing bacteria and protozoan cysts in the laboratory setting and improving the microbiological quality of household drinking water over the short-term, when applied where technologically appropriate and accompanied by user education. However, this case study illustrates the need for further research of PointOne performance before scaling-up distribution, including (1) establishment of bacterial removal rates and filter effectiveness in household settings; (2) characterization of the impact of variable water quality, including turbid, high-NOM, and hard influent water on filter microbiological and flow rate performance in the laboratory; (3) further investigation of membrane fouling, biofilm formation, and burst fibers within deployed filters; (4) determination of recommended backwashing and chemical cleaning regimen for filter fouling management; (5) long-term filter performance studies in laboratory and household settings, including component breakage and membrane fouling rates, to establish filter lifespan; and (6) development of an end-of-life indicator to prevent users from drinking effluent water that may be more contaminated than influent water.

CONCLUSIONS

The Sawyer PointOne filter is capable of bacteria and protozoan cyst removal in the laboratory setting, and is a HWTS option widely promoted for long-term use in developing countries. In this investigation of poorly functioning PointOnes used for 23 months for household water treatment, we identified an internal membrane that: exhibited a dense, highly cohesive irreversible fouling layer of inorganic particles, organic biomacromolecules, and biofouling on the exterior membrane fiber surface; was fouled on the inner fiber surface; and appeared to have burst fibers. Further research of PointOne membrane filter performance is recommended, including: characterizing filter effectiveness and the impact of source water quality on filter performance, investigating the extent of membrane fouling and bacterial growth within deployed filters, establishing a cleaning regimen to manage fouling, and developing an appropriate filter lifespan and end-of-life indicator.

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