

# Optimization of filtration to relaxation mode using woven fiber microfiltration system for water and wastewater treatment

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#### ABSTRACT

In this study, the fouling behavior of flat sheet woven fiber microfiltration (WFMF) system for water and wastewater treatment was investigated. In the first phase, WFMF system was operated for treating wastewater under filtration to relaxation mode (FRM) of  $30_{min} -10_{min}' 45_{min} -15_{min}$  and  $60_{min} -20_{min}$ corresponding to 36, 24 and 18 cycles/d, operated at an average flux of 8 L/m<sup>2</sup>/h. Results revealed that  $45_{min} -15_{min}$  was optimum FRM while variation in removal rate of COD, PO<sub>4</sub><sup>3–</sup>–P and NH<sub>4</sub><sup>+</sup>–N was 58%–71%, 21%–34% and 16%–30%, respectively. In the second phase, WFMF system was evaluated for water treatment having turbidity of 23–50 NTU, total suspended solids (TSS) 200–400 mg/L and fecal coliform (FC) 120 ± 20 CFU/100 mL under optimized FRM. The removal rates of turbidity, TSS and FC were 64%–96%, 58%–85% and 2–3 log, respectively. Physical and chemical cleaning were applied separately on the membrane and it was found that pore blockage causing irreversible fouling can only be removed by chemical cleaning.

Keywords: Chemical cleaning; Filtration cycles; Flat sheet membrane; Membrane-based septic tank; Water quality

#### 1. Introduction

Industrialization and urbanization imbalance the availability and demand of water, leading to water scarcity which is becoming a big threat to the existence of human kind [1]. Moreover, existing fresh water resources are being contaminated by release of industrial and municipal wastewater without proper treatment. The impact on groundwater quality of wastewater is well documented and a major concern globally [2,3]. Worldwide, 2.6 billion people do not have access to proper sanitation [4]. Lack of sanitation infrastructures in rural and semi-urban areas have increased the risk of waterborne and excreta-related diseases such as yellow fever, dengue, malaria and trypanosomiasis [5].

There is significant difference in effluent treatment efficiency of pollutants in developed and developing countries with respect to effluent discharge standards [6]. In developed countries, occasionally noncompliance occurs with respect to discharge standards and currently efforts are underway to control it sustainably. On the contrary, in developing countries, a significant gap between effluent concentrations and discharge standards exist and concentrated efforts are required to achieve compliance.

Decentralized or on-site treatment systems such as septic tank [7], trickling filter, constructed wetland [8] and smallscale membrane bioreactor [9] are feasible for developing

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countries [10] which offer adequate treatment to wastewater [11]. Conventional septic tank can remove 30%–45% of chemical oxygen demand (COD) and 50%–70% of total suspended solids (TSS) through sedimentation and anaerobic decomposition while, anaerobic filter can achieve better removal (60%–80% of COD and 80%–90% of TSS) at 48 h of hydraulic retention time (HRT) [12].

Over the past two decades, significant advancement in membrane technologies has brought great advantages in water and wastewater treatment [13], that is, better effluent quality, small areal footprint and lower waste production [14]. The conventional septic tank was modified by inserting low-cost woven fiber microfiltration (WFMF) system for secondary treatment in a septic tank. Applying conventional filtration to relaxation mode (FRM) cycle (10-15 min/cycle), colloidal particles start depositing on the membrane surface and inside the membrane fibers resulting in rapid membrane fouling [15]. Membrane fouling is an inevitable phenomenon which forced to incorporate membrane cleaning as compulsory procedure to restore membrane permeability [16]. There are several membrane cleaning protocols depending upon the type of fouling, that is, physical cleaning by flushing the cake layer from membrane surface and chemical cleaning to remove irreversible fouling layer for restoration of membrane flux [17]. Khan et al. [18] carried out a study on membrane-based septic tank (MBST) having FRM of 8<sub>min</sub>-2<sub>min</sub> by varying flux. However, due to large working head (distance between submerged membrane module and suction pump) and relatively short relaxation time, the membrane permeability could not be restored and rapid membrane fouling was observed. Furthermore, terminal trans-membrane pressure (TMP) of 70-80 kPa affected the filterability as the flat sheet (FS) membrane was exposed to rapid cake compression causing irreversible fouling layer and 80% flux reduction [18]. In this regard, a laboratory-scale MBST setup was established using WFMF system to assess the effect of new FRMs on fouling behavior and treatment performance as well as effect of cleaning protocols on membrane hydraulic resistances (cake and pore blocking resistance).

#### 2. Materials and methods

# 2.1. Setup design and operation

The experimental setup of laboratory-scale MBST as shown in Fig. 1 was operated having 2.6 L of working volume and dimensions were 33 cm of height, 20 cm of length and 4 cm of width.

FS WFMF system was immersed in the membrane tank having pore size of 1–3  $\mu$ m with effective filtration area of 0.05 m<sup>2</sup> and dimensions: 21 cm of height, 15 cm of length and 1 cm of width. Operational flux was maintained at 8 L/m<sup>2</sup>/h (LMH) resulting in HRT of 4 h. Specifications of membrane module are reported in Table 1. Membrane module was placed 5 cm above from membrane tank bottom, 5 cm below the water level and free board was 2 cm. Suction pressure was created by connecting one membrane port with peristaltic pump (Boading Longer, BT300–2J, China) through tubing, while second port was used occasionally to remove trapped air from the module. Digital manometer (840099 Data-logging, Sper Scientific, USA) was used for continuous



Fig. 1. Schematic diagram of membrane-based septic tank (MBST).

Table 1

Specifications of WFMF membrane module for water and wastewater treatment

Items	Specifications
Membrane type	Flat sheet, outside in, dead-end mode
Material	Woven polyester
Configuration	(2 sheet + 2 spacer)/module
Outlet ports	2
Pore size	1–3 μm
Operational flux	8 LMH
Terminal pressure	50 kPa
Effective filtration area	0.05 m <sup>2</sup>

recording of TMP and timer was used for controlling the filtration cycle during operation.

FS WFMF system was also evaluated as an on-site water treatment technology. Surface raw water was collected from Rawal Water Treatment Plant in Islamabad, Pakistan, and 1 mm mesh sieve was used to remove larger particulate matter. The same membrane tank was used for water treatment where raw water was pumped into membrane tank using peristaltic pump. System was operated utilizing same specification of membrane, operational conditions and equipment as mentioned in Table 1.

#### 2.2. Wastewater characteristics

Medium strength synthetic domestic wastewater having C:N:P as 100:5:2 was used as influent for MBST. Recipe of synthetic wastewater to maintain 200 mg/L COD included glucose 205 mg/L, ammonium chloride 38.4 mg/L, potassium di-hydrogen phosphate 17.5 mg/L, calcium chloride 2 mg/L, magnesium sulphate 2 mg/L, ferric chloride 0.6 mg/L and manganese chloride 0.4 mg/L. pH of 7–7.5 was maintained using sodium bicarbonate. This recipe of synthetic wastewater was based on real domestic wastewater analysis from university campus having characteristics reported in Table 2.

#### 2.3. Analytical parameters

Influent sample from feed tank and effluent sample from permeate tank were analyzed for water quality parameters such as TSS, COD, fecal coliform, ammonium nitrogen, phosphate–phosphorous and turbidity using techniques and equipment reported in Table 3 as per Standard Methods for the Examination of Water and Wastewater [19]. The treated drinking water was disinfected by chlorination with sodium hypochlorite having concentration of 0.03% w/v [20].

#### 2.4. Membrane resistance analysis

MBST operation was stopped when TMP reached 50 kPa, followed by disconnecting the membrane module from peristaltic pump and manometer. Total hydraulic resistance ( $R_i$ ) was measured prior to membrane cleaning comprising of intrinsic membrane resistance ( $R_m$ ), cake resistance ( $R_c$ ) and pore block resistance ( $R_p$ ).  $R_m + R_p$  were measured after applying physical cleaning while  $R_c$  was measured by subtracting  $R_m + R_p$  from  $R_i$ .

<sup>*m*</sup> The total hydraulic resistance ( $R_t$ ) was calculated using equations as listed below [7].

$$J = \Delta P / (\mu R_{\star}) \tag{1}$$

 $R_t = R_m + R_c + R_p \tag{2}$ 

where *J* = mean flux (LMH);  $\Delta P$  = trans-membrane pressure (Pa);  $\mu$  = permeate viscosity (Pa s);  $R_i$  = total hydraulic resistance;  $R_m$  = membrane resistance;  $R_c$  = cake resistance;  $R_n$  = pore resistance.

#### 2.5. Membrane cleaning

Fouled membrane was cleaned by applying both physical and chemical cleaning protocols. In physical cleaning, membrane was sun dried for 6 h until the cake layer got dried followed by cleaning with brush and finally washing with detergent and tap water, while in chemical cleaning, membrane module was submerged in (0.03% w/v) NaOCl for 6 h followed by washing with tap water.

#### Table 2

Characteristics of primary settled wastewater

### 2.6. Filtration to relaxation mode

FRM is a combination of filtration interval where suction is applied on membrane surface for treatment of water/ wastewater and relaxation interval where suction is paused in order to restore the membrane permeability. In this study, laboratory-scale MBST setup was operated at three FRM of  $30_{min}-10_{min'}$ ,  $45_{min}-15_{min}$  and  $60_{min}-20_{min}$  corresponding to filtration cycles of 36, 24 and 18/d, respectively, having average flux of 8 LMH while maintaining different instantaneous fluxes as mentioned in Table 4.

#### 3. Results and discussion

3.1. Evaluation of WFMF system as membrane-based septic tank: phase 1

#### 3.1.1. Optimization of filtration-relaxation mode

The membrane operation was stopped when TMP reached 50 kPa and instantaneous flux declined to approximately 60% of the initial value. Cao Ngoc Dan et al. [21] operated spiral woven fiber membrane (SFWM) system from 2 to 6 LMH flux and found that higher membrane resistance was observed at 2 LMH because of accelerated deposition of organic substances on membrane surface and recommended that flux via SFWM should be maintained at least above 4 LMH. The objective of our study was to optimize an effective FRM by evaluating TMP depicting fouling trends at a reasonable flux of 8 LMH keeping in view previous studies.

#### 3.1.2. Membrane fouling trends

A significant difference in fouling trends was observed in terms of TMP at three FRM  $(30_{min}-10_{min'}, 45_{min}-15_{min})$  and  $60_{min}-20_{min})$  for three successive cycles as shown in Fig. 2. WFMF membrane followed three-stage fouling pattern [22]

## Table 4

Membrane operation under different filtration to relaxation modes (FRMs)

Parameters	Raw wastewater	Parameter	Filtration	Relaxation	Filtration
COD (mg/L)	160–190		interval (min)	interval (min)	cycles
pH	7.5-8.0	FRM	30	10	36
Ammonium nitrogen (mg/L)	11–12.5		45	15	24
Phosphate-phosphorous (mg/L)	10–14		60	20	18

Table 3

Water quality parameters, technique and equipment/material

Parameter analyzed	Technique	Equipment/material
Total suspended solids (TSS)	Filtration-Evaporation	1.2 μm (GF/C, Whatman); 105°C oven
Turbidity	NTU	HACH turbidimeter 2100N
Chemical oxygen demand (COD)	Closed reflux	COD Tube; 150°C oven
Ammonium nitrogen (NH <sub>4</sub> <sup>+</sup> –N)	Hach reagents	Spectrophotometer (DR 2010, HACH)
Phosphate–phosphorous (PO <sub>4</sub> <sup>3–</sup> –P)		
Fecal coliform (FC)	MF filtration	Filtration assembly, Media EMB Agar
Disinfection	Chlorination	(0.03% w/v) sodium hypochlorite (NaOCl)

under all FRM. In the first stage, TMP increased rapidly for first few days due to rapid deposition of colloidal particles inside membrane pores, then became slower and stable for next few days where cake layer started consolidating on the membrane surface and finally, the consolidation of fouling layer resulted in permeate flux decline up to 60%.

In FRM  $(30_{min}-10_{min})$ , membrane fouled within 3 d (16.7 kPa/d) and reason behind this rapid fouling was excessive filtration cycles per day (36 cycle/d) [23]. During this mode, WFMF membrane was not able to recover the desired permeability as the relaxation time was not enough to achieve the desired recovery of TMP. While, fouling rate in 2nd and 3rd runs was 25 and 28 kPa/d, respectively, due to irreversible fouling incorporated by pore blockage.



Fig. 2. TMP profiles under filtration to relaxation modes (FRM) with physical cleaning.

Fouling behavior of WFMF membrane is very much dependent on instantaneous flux [23]. The permeate flux of 6.6, 9.9 and 13.2 LMH was observed at start-up of first filtration cycle for each  $30_{\min}$  –  $10_{\min}$  ,  $45_{\min}$  –  $15_{\min}$  and  $60_{\min}$  –  $20_{\min}$  FRM, respectively. The instantaneous flux at FRM (60<sub>min</sub>-20<sub>min</sub>) was observed to be relatively higher which causing rapid consolidation of cake layer on the membrane surface. While, due to less filtration cycles (18 cycle/d), membrane managed to run for 5 d before fouling (10 kPa/d). The membrane fouled in 4 and 2 d with fouling rate of 12.5 and 25 kPa/d in 2nd and 3rd filtration run, respectively, due to progressive irreversible fouling. The results for  $45_{min}-15_{min}$  FRM revealed to be optimum with fouling frequency of 7 d (fouling rate: 7.1 kPa/d). For  $45_{min}$ - $15_{min}$  FRM, the instantaneous flux of 9.9 LMH was relatively higher than  $30_{min}-10_{min}$  FRM but due to greater number of filtration cycles (24 cycle/d) than that of  $60_{\min}$ -20<sub>min'</sub> it provided feasible operating condition for WFMF. The fouling rates increased in subsequent runs to 10 and 12.5 kPa/d, respectively, due to pore blockage and development of irreversible fouling layer.

Table 5 presents hydraulic membrane resistances of WFMF membrane under different FRMs where the total hydraulic resistance ( $R_i$ ) was observed to be high under all FRM conditions due to physical cleaning. The results depicted that with physical cleaning only, pore blocking resistance ( $R_p$ ) and cake resistance ( $R_c$ ) rapidly increased after each run and contributed 27%–28% and 66%–68%, respectively, to membrane fouling. With the introduction of chemical cleaning following physical cleaning, both  $R_p$  and  $R_c$  reduced and

Table 5

Hydraulic resistances of virgin and fouled membrane under each filtration to relaxation mode with physical and chemical cleaning

Resistance (10 <sup>12</sup> m <sup>-1</sup> )	Physical cleaning: flux 8 LMH			
		FRM (30–10)	FRM (45–15)	FRM (60–20)
R <sub>m</sub>		0.7	0.7	0.7
R <sub>n</sub>	Run 1	1.4	2.0	2.5
(Åvg. 3.23)	Run 2	2.8	3.5	3.9
	Run 3	3.8	4.2	5.0
$R_c$	Run 1	5.45	6.4	7.7
(Avg. 7.73)	Run 2	6.8	7.3	9.1
	Run 3	8.6	9.5	10.5
$R_t$ (average)		10.3	11.7	13.6
$R_m/R_t$		6%	6%	5%
$R_{\nu}/R_{t}$		27%	28%	28%
$R'_{t}/R_{t}$		67%	66%	68%
	Chemical cleaning: flux 8	LMH-FRM (45-15)		
$R_{v}$ (Avg. 0.97)	Run 1	0.8		
,	Run 2	1.0		
	Run 3	1.1		
$R_{c}$ (Avg. 2.27)	Run 1	1.2		
	Run 2	2.5		
	Run 3	3.1		
$R_t$ (average)		3.9		
$R_m/R_t(\%)$		18%		
$R_{p}/R_{t}(\%)$		25%		
$R_c/R_t(\%)$		57%		

contributed 25% and 57%, respectively, exhibiting the effectiveness of chemical cleaning protocol.

#### 3.1.3. Treatment performance of MBST

MBST treatment performance was determined in terms of COD, TSS,  $NH_4^+-N$  and  $PO_4^{3-}-P$  concentrations and removal rate. Removal rates of three FRM are reported in Table 6.

The COD removal varied 60%-70% at different FRM. Thanh and Dan [7] reported that at FRM of 8<sub>min</sub>-2<sub>min</sub> MBST was able to remove 60% of COD having influent concentration of  $124 \pm 28$  mg/L. This slight variation in COD was due to different instantaneous flux under each FRM condition. Nitrogen in wastewater (ammonia, ammonium, nitrite and nitrate) can be removed by nitrification and denitrification [24]. Due to relatively short HRT in membrane tank and in the absence of suspended biomass, nitrification and denitrification was not observed. However, it was witnessed that after continuous filtration, the surface pore size  $(1-3 \mu m)$  of WFMF membrane further decreased due to deposition of organic layer and consequently the ability to retain pollutants on membrane surface enhanced. This secondary layer of WFMF membrane may have managed to remove 16%–30% of ammonium nitrogen through physical adsorption.

The phosphate–phosphorus removal depend on bacterial growth in septic tank [25]. Due to short HRT, limited bacterial activity occurred in membrane tank, but WFMF effectively removed 26%, 28% and 32% under the three FRM conditions, respectively. Overall, COD,  $PO_4^{3-}$ –P and  $NH_4^+$ –N of MBST permeate were found to be within National Environmental Quality Standard (NEQS), Pakistan Environmental Protection Agency (Pak-EPA) [26].

# 3.2. Influence of membrane cleaning protocols: phase 2

Physical cleaning protocol was followed in phase 1 and it was found that there was 7% increase in irreversible fouling after three successive filtration runs which resulted in rapid membrane fouling. Phase 2 was conducted to reduce irreversible fouling by cleaning the membrane with 0.03% NaOCl using concentration of 2,000 mg/L for 6 h following physical cleaning. However, excessive use of chemical can affect the properties and performance of membrane [16]. System was operated at optimized FRM of  $45_{min}$ – $15_{min}$  and resistance analyses was conducted between successive cycles. It was observed that after chemical cleaning irreversible fouling was reduced from 7% to <1%, while fouling rate also reduced from 10.0 to 6.5 kPa/d which prolonged filtration duration as shown in Fig. 3.

Table 6 Removal rate of MBST at different filtration–relaxation mode

On average, the total hydraulic resistance ( $R_i$ ) was reduced from 11.7 ×10<sup>12</sup> to 3.9 × 10<sup>12</sup> m<sup>-1</sup>, that is, 67% reduction in  $R_i$  with chemical cleaning as reported in Table 3. Furthermore, cake resistance and pore block resistance were also reduced to approximately 70% with chemical cleaning retarding the fouling frequency and reducing the reversible and irreversible fouling.

# *3.3. Evaluation of WFMF system for surface water treatment: phase 3*

Three consecutive filtration cycles were performed at optimized FRM  $45_{min}$ - $15_{min}$  to evaluate fouling behavior of WFMF membrane as shown in Fig. 4. As the filtration started, an initial jump in TMP was observed but in later stages gradual change in TMP was observed. However, a slight fluctuation in TMP was observed due to variation in TSS and turbidity of raw surface water. The fouling rate in 1st, 2nd and 3rd filtration cycle was almost similar as 7.5, 7.1 and 9 kPa/d, respectively.



Fig. 3. TMP profile with physical and chemical cleaning.



Fig. 4. TMP profile of WFMF membrane using an on-site water treatment technology.

Parameters	Influent	% Removal			Effluent	NEQS (1997)
		(30–10)	(45–15)	(60–20)		
COD (mg/L)	$170 \pm 20$	61 ± 3	65 ± 2	69 ± 2	45-60	150
PO <sub>4</sub> <sup>3-</sup> –P (mg/L)	$13 \pm 2$	$26 \pm 5$	$28 \pm 4$	$32 \pm 2$	9–11	40
NH <sub>4</sub> <sup>+</sup> –N (mg/L)	$11 \pm 2$	$20 \pm 4$	$24 \pm 3$	$26 \pm 4$	8-10	40
pН	7.6-8.1	7.8-8.0	7.8-8.0	7.8-8.0	7.5–8.0	6–9

Parameters	Influent	Effluent	Removal	WHO (2011)	NSDWQ (2008)
Turbidity, NTU	37 ± 13	1–18	64%-96%	<5	<5
TSS, mg/L	$300 \pm 100$	30-170	58%-85%	-	-
E. coli, CFU/100 mL	$120 \pm 20$	1–5	2–3 log	Must not be	Must not be
				detected in 100 mL	detected in 100 mL
				sample	sample

Table 7 Water quality parameters and removal rate of on-site water treatment



Fig. 5. Optimum dosage of chlorine for water disinfection.

# 3.3.1. Treatment performance of on-site raw water treatment system

The raw water was fed with turbidity of 23–50 NTU and TSS of 200–400 mg/L. Influent and effluent turbidity results revealed that WFMF can efficiently treat the raw turbid water of <30 NTU, within World Health Organization (WHO) Guideline [27] and National Standards for Drinking Water Quality (NSDWQ), Pakistan, [28] while *Escherichia coli* removal was found to be 90%–99% and needed further disinfection to be within limits of NSDWQ as shown in Table 7.

#### 3.3.2. Chlorination

Chlorination is considered as an economical method for disinfection. Different dosages of sodium hypochlorite (NaOCl) were added in 1 L of sample as 0.5, 1, 1.5, 2, 2.5 and 3 mg/L. Residual chlorine was measured after 1 h in each sample and found that 2 mg/L as an optimum dosage for disinfection having residual chlorine of 0.5 mg/L as shown in Fig. 5.

#### 4. Conclusions

In this study, woven fiber microfiltration membrane was immersed in a bio-tank as MBST for wastewater treatment as well as for surface water treatment. In MBST, membrane fouling control was investigated by varying FRM and found  $45_{min}-15_{min}$  as the optimum FRM with fouling rate of 7 kPa/d, while average effluent concentrations of COD, PO<sub>4</sub><sup>3-</sup>–P and NH<sub>4</sub><sup>+</sup>–N were 55, 10 and 9 mg/L and found to be within National Environmental Quality Standards (NEQS). Irreversible fouling was controlled from 7% to <1% by chemical cleaning using NaOCI: 0.03% w/v. For surface water treatment, WFMF system was able to effectively treat low turbid (<30 NTU) raw water.

TSS and turbidity removal rate were 85% and 96% while for *E. coli*, removal rate was 2 log at filtration start-up and reached up to 3 log at the end of filtration run.

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