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Low strength graywater characterization and treatment by direct membrane filtration

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Abstract

The potential of direct membrane filtration on either ultrafiltration (30, 200 and 400 kDa MWCO) or nanofiltration (200 Da MWCO) membranes was studied as a method of treatment of low load graywater for on site reuse. The graywater collected from a sports center's public showers and characterized for its chemical composition and physical properties fitted well to a low strength type (29.8 mg/l TSS and 170.3 mg/l as COD). Particle distribution analysis showed that colloidal size particles are the dominant fraction in terms of number distribution, while the much fewer, larger particles make up most of the particle volume (mean particle diameter ≈ 0.1 μm). When treated by ultrafiltration, COD and turbidity concentration of permeates complied with established restrictions (45–70% and 92–97% reduction range, respectively), however BOD values were still above the requirements in all cases. Permeate produced by nanofiltration was of high quality with high rejection of soluble organic matter (>90%) and ionic species (50%). It can be concluded that direct dense-membrane filtration is a favorable candidate for efficient treatment of graywater for unrestricted reuse. MWCO optimization still needs to be done in order to achieve better economics at an acceptable quality of permeate produced.

Keywords: Graywater; Graywater treatment; Membrane filtration; Ultrafiltration; Nanofiltration; Particle size distribution

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1. Introduction

In recent years, graywater recycling has been receiving increasing attention from the general public, as well as from the scientific community. Such recycling schemes involve the separation of domestic wastewater into so-called graywater (all domestic wastewater sources other than toilet flushing) and blackwater (toilet flush wastewater), and the subsequent treatment of the graywater on-site for non-potable reuse purposes such as irrigation, toilet flushing, etc. Graywater is a major fraction of domestic wastewater, amounting to

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more than half the total wastewater volume produced [1–3]. In the design of in-situ, small scale graywater treatment systems there are currently several limitations. First and foremost, there is insufficient knowledge of graywater characteristics, much needed for determining appropriate technological solutions. Other limitations include the many technical and public issues associated with the installation of treatment systems in urban areas, e.g., space requirements, operational stability and dependability, maintenance, water quality, etc.

Traditionally, graywater is defined as all domestic wastewater sources other than toilet flushing, e.g. showers, wash basins, washing machines, etc., and is generally viewed as significantly less polluted than blackwater. Former studies reported organic loads of 48 and 72 g COD per person per day for graywater and blackwater, respectively [4] and characteristic graywater contaminant concentrations of 175 mg/l BOD_5 and 78 mg/l TSS, as opposed to 250 and 220 mg/l as BOD_5 and TSS, respectively, for blackwater [5]. However, recent studies [1,2], showing that there are significant differences in wastewater characteristics among the various graywater sources, have proven this definition to be inaccurate. A newer definition separates the waste stream further according to pollution loads — high load and low load graywater [6,7]. High load graywater consists of kitchen, washing machine and dishwasher wastewater. Low load graywater from the bath, shower and washbasin amount to 50–60% of the total graywater [1,2], but are considerably less polluted — low-load graywater contaminant concentrations lie in the range 210–501 mg/l as COD and 54–200 mg/l as TSS, while high load graywater contributes between 1079–1815 mg/l as COD and 165–235 mg/l as TSS [1]. A major characteristic of low load graywater is its availability in public as well as household appliances, the best example being public showers.

Treatment of graywater is cause for yet another debate, with several candidate technologies investigated. Of these, membrane technology has received much attention, mainly in the membrane bioreactor (MBR) configuration [8,9], and to a lesser extent — direct membrane filtration [10]. High effluent qualities were achieved by these two technologies, with TSS concentrations below detection limits (TSS ≈ 0) and organic matter concentrations below 10 mg/l (in terms of BOD_5 for the MBR and of COD for direct membrane filtration). While high quality effluents, suitable for recycling, can be achieved by both MBR and direct membrane filtration treatments, the possible advantage of the latter configuration over the MBR, seems to lie in its simplicity, being a physical, rather than biological-physical, process, especially when considering in-situ recycling.

Treated water quality, intended for on-site recycling purposes, must comply with the reuse criteria for major chemical and physical contaminants, as specified by the local authorities. Such requirements vary in severity, yet generally require similar pollution reductions, according to form of reuse intended for the treated water.

The objective of this work was to characterize the chemical composition and physical properties of low load graywater, as well as to examine the potential of direct membrane filtration on either ultrafiltration (UF) or nanofiltration (NF) membranes, as the method of its treatment for reuse. Permeate quality obtained was compared with different reuse criteria for major chemical and physical contaminants as specified by different countries and institutes worldwide [11] and the recommendations of the joint committee for unrestricted water reuse in Israel [12].

2. Experimental

2.1. Graywater collection

Samples of graywater were collected in a 200 L holding tank installed at the Technion sports center's public showers. An electrical valve installed along with the holding tank and controlled by a pre-set timer was opened at the hours of high activity (9 am—12 pm) to collect the water. The purpose for this was to enable the collection of a relatively large volume of graywater, while ensuring the sample freshness. These parameters pertain to the large variability expected from different operation cycles (e.g., two different people showering), and to the degradability of the graywater, as reported in the literature [4,12].

2.2. Direct membrane filtration tests

Direct membrane filtration studies of graywater samples were performed on either UF or NF modules.

UF experiments were conducted using a dead-end bench-scale laboratory module in which a flat sheet membrane is placed on a fret support at the bottom of the module, which is sealed using an O-ring, as shown in Fig. 1. The module is constructed of stainless steal, with an outer diameter of 100 mm and an inner diameter of 60 mm. Pressure was supplied to the module by using a compressed nitrogen gas cylinder, equipped with a pressure gauge and controlled by a manual valve. The module was filled with a graywater sample and the permeate was collected at the bottom of the module. Pressures used in the UF experiments were in the range of 1–2 bar, and were kept constant throughout each experiment.

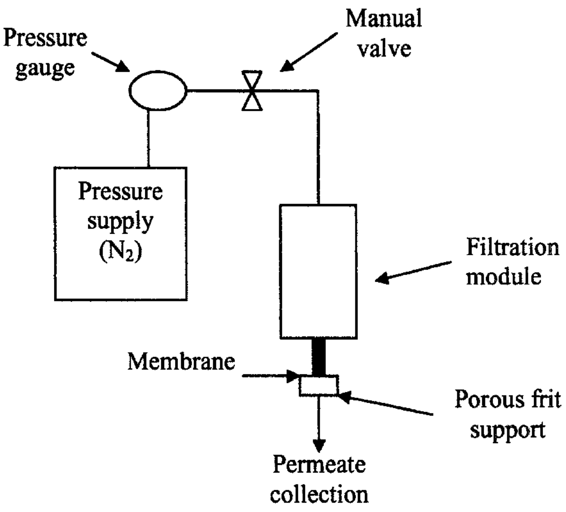

Fig. 1. General outlay of bench-scale membrane filtration system used for dead-end UF experiments.

NF experiments were conducted using a bench-scale crossflow filtration unit equipped with a MIC-RO 240 module (PCI) as described in Fig. 2. A volume of 30 l was used in each experiment in recirculation mode, with a constant flowrate of 150 l/h Trans-membrane pressures applied ranged between 6 and 10 bar.

Three flat-sheet UF membranes were tested as follows: polyacrilonitrile (PAN) with a MWCO of 400 and 200 kDa (Rochem UF–Systeme AG),

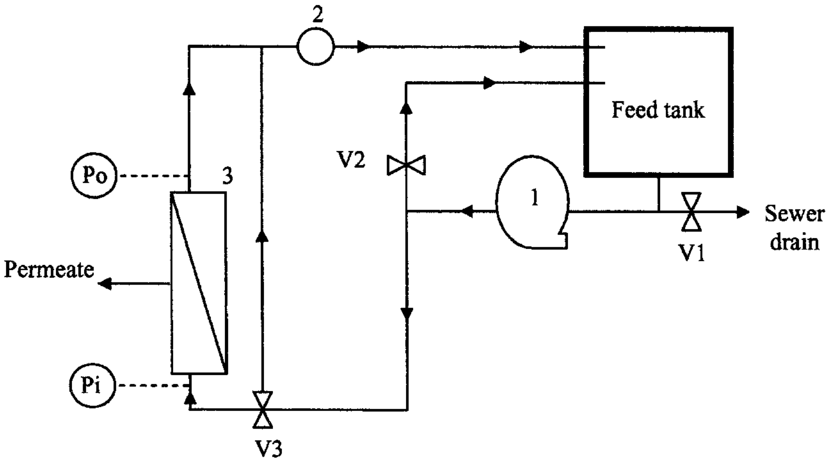

Fig. 2. General outlay of bench-scale crossflow membrane filtration system used for NF experiments. 1, pump; 2, rotameter; 3, NF module; Pi, inlet pressure gauge; Po, outlet pressure gauge; V1,V2,V3, manual valves.

polyethersulfone (PES) with a MWCO of 30 kDa (PM30, Millipore). A tubular NF membrane (30 cm length, 1.25 cm inner diameter) of polyamide (PA) with a MWCO of ~200 Da and 0.014 m^2 filtration area was used (AFC30, PCI). In all cases, each experiment was started with a new membrane.

2.3. Chemical and physical analysis

COD, TSS and BOD_5 measurements were conducted according to Standard Methods [13]. Total and soluble chemical oxygen demand (CODt and CODs, respectively) was measured, soluble COD obtained from samples filtered through a 1 μm rated glass fiber filter. Five day biochemical oxygen demand was measured using unfiltered samples and thus represents the total BOD_5. Conductivity and pH were measured using Cyberscan electrodes (EUTECH Instruments, waterproof series). Turbidity was measured with a 2100P turbidimeter (HACH). Ion content analysis was performed on a 761 compact IC model ion conductivity chromatograph-ICC (Metrohm) using Metrosep Cation 1–2 and Anion dual 1 columns upon filtration of the samples by 0.45 μm rated microfilter. Particle distribution by size and volume were calculated using a Coulter LS 230 – LS particle size analyzer. Inductively Coupled Plasma (ICP) analysis was conducted using Optima 3000DV (Perkin Elmer). TOC was measured with a TOC 5000A, Total Organic Carbon Analyzer (Shimadzu).

3. Results and discussion

3.1. Graywater characteristics

The main objective of this study was to characterize chemical and physical properties of low load graywater. Analysis for bulk contaminant constituents is presented in Table 1. As can be seen from the data presented, there is considerable variability in the concentrations of organic matter fraction expressed as either COD or BOD_5, as well as in suspended material measured by either TSS or turbidity. This variability seems to be an inherent characteristic of graywater, in line with previous observations [12]. As described above, the collection method was specifically designed towards the collection of an average daily sample in an effort to mitigate the assumed primary cause of this variability, which is the nature of such individual operations performed by different people (or the same person performing the same operation several times — in terms of water volume, quantity of soap/shampoo used, etc.). Even so, the graywater source consisted of merely three showers, therefore limiting the "averaging effect". Peak organic loads measured amounted to 250, 200 and 115 mg/l as total COD, soluble COD and BOD_5 (in different samples), respectively, and 59.5 mg/l of TSS, indicating that the collected graywater are indeed low-strength wastewater, with an average BOD_5/COD_T ratio of 0.46. The substantial difference between total and soluble COD, when considering the relatively low concentrations of TSS, may be attributed to the adsorption of surfactants onto the surfaces of suspended particles present in the graywater, thus contributing to the relatively high COD associated with suspended material.

The concentration of the major ionic species present in low load graywater, were analyzed by ICC, comparatively to tap water (Table 2). As evidenced by the data presented, major ionic species fall within the range of normal background concentrations of tap water, nevertheless an increase of K^+, NH_4^+ and PO_4^{3-}, and to a lesser extent Na^+, is noticeable. This may be attributed to surfactants and foaming agents, which are commonly found as sodium, potassium and ammonium salts. The low N-species and phosphate concentrations indicate a low nutrient load of the graywater.

Concentrations of heavy metals and boron were measured by ICP (Table 3), showing some variation between the two samples analyzed, mainly in Fe and Zn concentrations.

From a comparison of data obtained in this study, with data compiled from various publica-

Table 1
Chemical analysis of low load graywater samples

Sample no.	COD_T (mg/l)	COD_S (mg/l)	TSS (mg/l)	BOD_5 (mg/l)	Turbidity (NTU)	pH	EC (μS/cm)	TDS (mg/l)
1	—	95	18.5	40	—	7.6	1317	660
2	—	200	8.8	49	—	7.4	1233	617
3	176	160	20.8	86	—	7.7	1208	605
4	141	54	41.2	100	—	7.2	1170	586
5	172	108	29.6	85	—	7.3	1154	577
6	150	148	20.3	—	—	7.5	1100	559
7	—	—	34	—	—	7.7	1361	683
8	99	59	20	36	14.6	7.5	1019	565
9	102	72	14.8	46	19	7.4	1126	560
10	180	—	34	95	24.1	7.3	1149	574
11	151	116	21.2	72	18	—	—	—
12	147	108	19.2	98	22.4	—	—	—
13	265	195	31.7	115	15.8	7.4	1300	—
14	75	40	23.2	—	15.6	7.5	1400	—
15	186	95	42.3	78	—	—	—	—
16	178	84	47	110	28.7	—	—	—
17	250	—	59.5	—	45	—	—	—
18	226	—	27.6	—	15	—	1600	—
19	152	95	26.8	—	23.2	—	1300	—
20	216	143	28.4	—	23	—	1200	—
21	148	66	23.8	—	22.4	—	1300	—
22	224	125	—	—	35.3	—	—	—
Mean	170	106	29.8	78	23	7.5	1241	599
STD	49	42	11.3	26	8.5	0.2	143	43

Table 2
Major ionic species present in low load graywater in comparison to tap water

Ion (mg.l)	Range of values for tap water	Range of values for graywater	Mean values for graywater
Ca^{2+}	64.7–80.3	71.0–93.6	79.6
Mg^{2+}	46.2–53.6	43.2–50.0	47.6
K^+	5.2–10.2	9.8–12.4	10.4
NH^{4+}	ND	1.5–3.0	2.7
Na^+	92–128	93–142.7	106.0
NO^{3-}	0.3–2.0	0.05–1.7	0.67
PO_4^{3-}	ND	0.02–0.19	0.09
SO_4^{2-}	60.0–77.4	49.0–61.3	58.0

ND — non detectable

tions, it can be seen that there are considerable differences between low load graywater characteristics, as measured in different countries (Table 4). This high variability of graywater quality may reflect both method of samples collection and personal habits exhibited by users in different countries, where cultural as well as climatic effects come into consideration. The data

Table 3
Various chemical species concentrations obtained by ICP analysis of graywater samples

Specie (mg/l)	Sample no. 1	Sample no. 2
Ni	<0.02	<0.02
Li	0.03	0.02
Fe	0.19	0.06
Zn	0.18	0.03
B	0.14	0.11
Al	0.03	0.03
Sr	0.52	0.50
Ba	0.13	0.08
Pb, Bi, Ti, V, Mo, Hg, As, Se, Be, Cr, Cd, Mn, Co, Cu, Ag	<0.02	<0.02

collected in this study displayed the closest similarity to the data reported by Stephenson et al. [14]; however, these similarities should be analyzed with precaution since their data was derived from samples representing complete single operation cycles (complete volume of a single shower operation sampled).

According to various water quality criteria for reuse, there appear to be two major bulk contaminants present in graywater: organic compounds and suspended solids. Conductivity values as well as concentrations of single soluble ionic species — are marginally in compliance, while concentrations of nutrients, heavy metals and boron are well within limits.

3.2. Particle size distribution

In order to characterize the suspended material present in graywater, samples were analyzed for particle number and particle volume distributions using a particle size analyzer (Fig. 3). Samples analyzed contained ~20 mg/l total suspended solids, a value quite representative of low load graywater sampled in this study. Particle number distribution curves (Fig. 3a) clearly show that most particles were in the range 0.04–1 μm, 0.04 μm being the lower detection limit of the analyzer employed. Volume distribution enabled detection of coarser material, i.e., particle sizes up to 1 mm (Fig. 3b). Particles larger than 5 μm amount to more than 95% of the total particle volume, while making up less than 1% of total particle number. The only noticeable variability between samples could be seen in this range.

The distribution of particle size, by number, is shown in Fig. 3c, showing that 90% of the particles in the graywater are smaller than 0.18 μm with a mean particle size of 0.1 μm, suggesting a colloidal nature of graywater. These findings are in some discrepancy with those reported previously in the

Table 4
Graywater chemical constituents, as reported by various studies

	COD_T (mg/l)	BOD_5 (mg/l)	NH_4^+ (mg/l)	Turbidity (NTU)	TSS (mg/l)
Present study	170	78	2.7	23.0	30
Stephenson et al. [14][a]	420 (181)	146 (90)	—	84.8 (17.9)	—
Ahn et al. [10]	70	—	0.1	19.0	43
Funamitzu et al. [3]	—	92	2.5 (total-N)	—	—
Almeida et al. [1]	501	—	1.2	—	200
Butler et al. [2][b]	—	250	1.5	—	—

[a] values in parentheses represent a single sample (14 off)
[b] sample sources include bath water

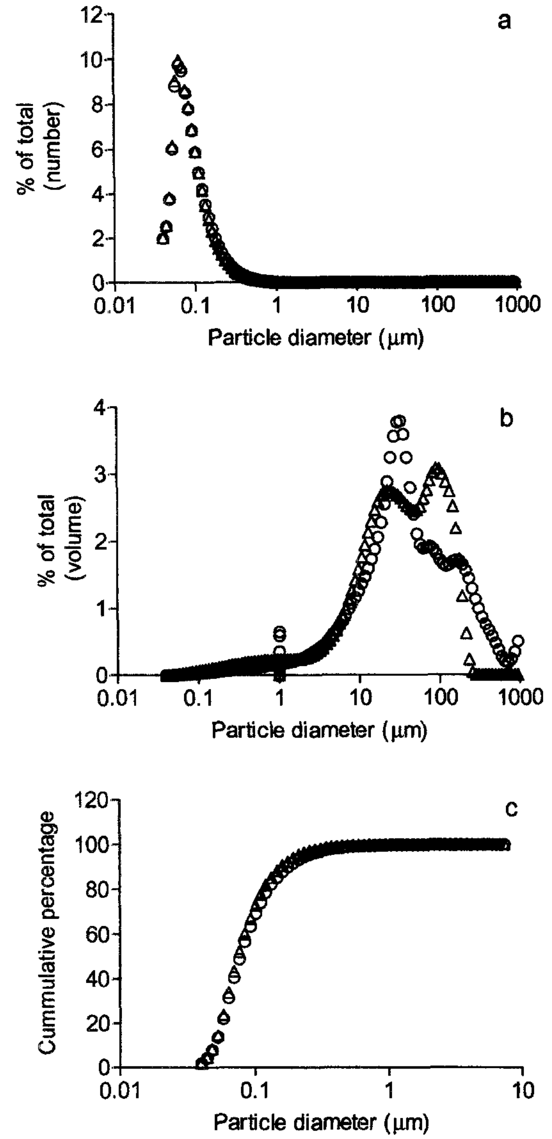

Fig. 3. Particle size distribution analysis of two individual graywater samples: (a) Distribution of particle sizes by number; (b) Distribution of particle sizes by volume. Detection limit of analyzer is 0.04 μm; (c) Particle size distribution — cumulative percentage. TSS concentration of samples ≈20 mg/l. (○): sample 1; (△): sample 2.

literature [10] showing a mean particle diameter of 2.18 μm, with the dominant range being 0.5–6.39 μm. This indicates again that graywater may exhibit considerable variability not only in chemical composition but also in physical components such as particle size distribution. Particle size distribution is of considerable importance in membrane filtration, as it influences cake resistance [15].

3.3. Graywater BOD curves

Data collected during BOD_5 measurements was analyzed using the least squares regression technique, in order to obtain values of the reaction constant, as well as ultimate BOD (BOD_U) figures. Regression was performed according to the standard equation for BOD, assuming first order kinetics [16]. Rate constant values estimated for the graywater varied widely, ranging between 0.23–0.95 d^{-1}, with an average value of 0.6 d^{-1}. These results are in agreement with the values reported in the literature, which estimated the rate constant to be 0.45 d^{-1} [4] and as high as 0.62 d^{-1} [12]. For comparison, common values for domestic wastewater BOD rate constants are in the range 0.12–0.46 d^{-1}, with a typical value of 0.23 d^{-1} [16]. BOD results may depend greatly on the sources and mode of collection and sampling of the graywater sample, e.g., concentration and type of organic matter present, bacteria concentration, possible perturbation by introduction of microbial inhibitors or presence of toxic substances such as chlorine containing cleaning agents, etc. Comparison of BOD_U values calculated by least square regression, with pseudo-BOD_U values observed after long incubation times of the samples (up to 20 days) showed an error of up to ±15% (data not shown). Even though the data obtained is somewhat inaccurate, it indicates that a treatment system designed to treat graywater must therefore take into account the high rate of oxygen consumption exhibited in order to prevent septic condition which may occur rather quickly.

3.4. Permeate quality

Following the characterization of the graywater collected, the ability to remove graywater contaminants by either ultrafiltration or nanofiltration was characterized (Tables 5 and 6, respectively). Membranes used in this study cover a wide range of molecular weight cutoffs, UF membranes ranging between 30–400 kDa and an NF membrane setting the bottom limit at 200 Da (if sieving rejection mechanisms are considered). Although different system configurations were applied for UF and NF, upon membrane availability, this was of little practical relevance since the study was aimed to test only the quality of the permeate produced as a result of the different MWCO used.

For UF, an increase of permeate quality with the decrease of the MWCO was observed, as could be somehow expected, with the most noticeable parameters affected being the organic matter concentration (expressed as COD) and suspended material (expressed as turbidity), whereas almost no change in soluble ions (expressed as electrical conductivity) took place. Turbidity reduction was very high, ranging between 92–97%, according to the decrease of MWCO. Similarly, organic matter removal ranged from 45–70%. The narrower range of values observed for the 30 kDa membrane permeate, compared with those obtained for 200 and 400 kDa permeates, are mainly due to the difference in the samples applied, suggesting that the major variations in particles size occurred in the range of those rejected by the 30 kDa membranes, but not by the other, more open membranes. The noticeable difference in conductivity between the 30 kDa permeate and the 200–400 kDa permeates should also be attributed the same samples variability and should not be mistaken as a rejection of ionic species by the UF membranes. A previous study [10] reported a COD_T removal of approx. 90% using different MWCO rated MF/UF membranes (0.1 μm–15 kDa), employing graywater containing initial COD_T and TSS values of approx. 70 mg/l and 40 mg/l, respectively (compared to 170 and 30 mg/l, respectively, in our samples). The significant reductions reported previously [10] may be explained by a larger average particle size in their sample. Moreover, the rejection data presented by them showed little variation between the different

Table 5

Permeate quality obtained with UF membranes

Parameter	COD_T, mg/l			Turbidity, NTU			EC, μS/cm		
	400 kDa	200 kDa	30 kDa	400 kDa	200 kDa	30 kDa	400 kDa	200 kDa	30 kDa
No. of samples	5	5	3	4	5	5	4	3	5
Mean	80	74.3	50.6	1.4	1	0.8	1212	1296.3	1080
STD	21.5	28.6	6.6	0.4	0.5	0.2	88.7	263.4	178.8
Range	54–102	40–103.4	45–58	1–1.9	0.6–1.6	0.5–1.0	1154–1343	1130–1600	1130–1600
Removal efficiency, %	45.2	49.1	69.3	92.3	94.2	96.6	none	none	none

Table 6

Feed and permeate quality during NF crossflow experiment (0.2 kDa)

	COD_T, mg/l	TOC, mg/l	TSS, mg/l	Turbidity, NTU	EC, μS/cm
Feed	226	37.7	27.6	29.5	1500
Permeate	15	6.2	none	0.6	700
Removal efficiency, %	93.3	83.5	100	98.1	53.3

MWCO applied, demonstrating that organic matter was possibly present mainly as suspended material, being larger than the nominal MWCO of the membranes used. The particle size distributions measured in this study clearly indicated the presence of smaller particles dominating the range. It may therefore be concluded that the graywater treated in this study, contain a significantly higher concentration of soluble organic matter with a relatively low molecular weight, explaining the lower rejection obtained by the various UF membranes. Overall, permeate quality obtained by the UF membranes, including the lower MWCO, is probably not in compliance with criteria for BOD_5, according to wastewater treatment regulations stipulated worldwide [11] (for example, regulations in Israel [12] stipulate <10 mg/l BOD_5, quality obtained $BOD_5 \approx 23$ mg/l, assuming a BOD/COD ratio of 0.46), but other criteria are met and even surpassed (e.g. TSS ≈0 obtained, <10 mg/l required in Israel; COD ≈50 mg/l obtained, <100 mg/l required in Israel).

For direct treatment by NF excellent permeate quality was obtained, with very high organic matter removal (over 93% of COD_T removed, to a final concentration of approx. 15 mg/l corresponding to a calculated $BOD_5 = 7$ mg/l and 84% removal of TOC to final value of approx. 6 mg/l), 50% reduction of soluble ionic species, and nearly complete removal of turbidity and suspended solids (Table 6). The membrane applied is capable of quite effectively retaining divalent ions (rejection of up to 75% $CaCl_2$, according to the manufacturer). This rejection was observed by ICC analysis as well with rejections of Mg^{2+}, Ca^{2+}, Na^+ and SO_4^{2-} calculated as 37%, 37%, 30% and 83%, respectively. The substantially higher removal efficiency achieved by nanofiltration, compared to that obtained by the various UF membranes, demonstrates further the presence of soluble, low molecular weight organic matter. This fraction may be attributed, at least in part, to surface active substances, and poses the greatest difficulty for removal by direct membrane filtration. The potential of NF for removal of commercial surfactants from aqueous solutions in industrial processes aimed at their reclamation, with rejection of 95–99.9%, has been recently reported [17]. As it appears from these findings, the NF permeate produced seems to be well suited for all purpose-unrestricted reuse.

The performance of NF separation was almost constant along the 150 min of operation carried out at each condition. The average steady-state fluxes achieved at 6 and 10 bar were approx. 15 and 35 $l.m^{-2}.h^{-1}$, respectively. The plot of membrane resistance corrected for viscosity change with temperature at 10 bar, displayed a hyperbolic behavior, reaching an asymptotic value of roughly 1.6×10^{14} m^{-1} after 60 min of operation (data not shown). This behavior is in agreement with the theory of crossflow filtration where steady state conditions between the flow and the filtration cake are achieved.

4. Conclusions

The graywater characterized in this study are of low strength type, with major pollutants being suspended solids and organic matter. Treated by UF, permeate quality obtained increased with decreasing MWCO of the membrane used. Despite compliance with COD and TSS concentration restrictions, it is assumed that BOD requirements are not met, for the permeate obtained by the UF membranes, even for the denser one (MWCO 30 kDa). Permeate produced by nanofiltration was of high quality and is suitable for unrestricted reuse with high rejection of soluble organic matter and partial rejection of ionic species.

Organic matter rejection data, expressed as COD, obtained by the various pore size/MWCO rated membranes, enabled a rough estimation of MW distribution of the organic matter present in graywater, as presented in Table 7. Based on this, it can be seen that approx. 60% of the organic matter is below 1 mm particle diameter. Particle

Table 7
Estimated molecular weight/particle size distribution of organic matter in low load graywater

% of total COD[a]	Molecular weight/particle size
37.5	>1μm
14.2	400 kDa (0.002μm)–1 μm
6	200 kDa–400 kDa
13.5	30 kDa–200 kDa
20	200 Da–30 kDa
8.8	<200 Da

[a] Percentage was calculated from rejection of COD by filtration at different MWCO ratings.

distribution analysis showed that colloidal size particles are the dominant fraction in terms of number distribution, while the larger particles make up most of the particle volume. Mean particle diameter was measured as 0.1 μm, particle sizes ranging from 0.04 μm (detection limit) to 1 mm.

It can be concluded that membrane technology using dense membranes is a favorable candidate for a simple and efficient treatment of graywater for all purpose-unrestricted reuse. Nonetheless, further research is required for establishing the correct mode of operation and selection of the optimal MWCO, by studying the continuous membrane filtration of graywater under various conditions. Although it is concluded that NF is an appropriate method of treatment, slightly higher MWCO rated membranes can potentially demonstrate better economics, at an acceptable quality of permeate produced and should be considered for this purpose, through further research.

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