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Potential of ceramic microfiltration and ultrafiltration membranes for the treatment of gray water for an effective reuse

Priyankari Bhattacharya[a], Sandeep Sarkar[a], Sourja Ghosh[a,]*, Swachchha Majumdar[a], Aniruddha Mukhopadhyay[b], Sibdas Bandyopadhyay[a]

[a]*Ceramic Membrane Division, CSIR-Central Glass and Ceramic Research Institute, Kolkata 700032, India*
Tel. +91 33 2473 3469/76; Fax +91 33 2473 0957; email: sourja@cgcri.res.in
[b]*Department of Environmental Science, University of Calcutta, Kolkata 700019, India*

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ABSTRACT

Gray water may serve as a water resource for agricultural uses due to their high nutrient content. In this study performance evaluation of indigenously developed low-cost ceramic membrane was investigated for treatment and reuse of high organic loaded gray water. The efficiency of microfiltration (MF) and ultrafiltration (UF) process was studied individually and as a two-stage treatment involving MF followed by UF. Effect of time was observed on reduction of chemical oxygen demand (COD) in the permeate stream and permeate flux in different processes. Depending on the variation in feed loading, about 73–90% COD reduction was achieved in the single-stage UF at 30 min of filtration with operating pressure of 2 bar, which was about 84–94% for two-stage treatment. Permeate quality in terms of organic loading, oil and grease and coliform concentration were found suitable according to the discharge norms for agricultural reuse of water. The effect of untreated, MF- and-UF treated wastewater was observed on a popular palm species, *Chrysalidocarpus lutescens* H. Wendl. The study showed significant potential for use of the MF- and-UF treated water which facilitated an enhanced uptake of most of the essential nutrients in plants compared with that of fresh water.

Keywords: Ceramic membrane; Microfiltration; Ultrafiltration; Gray water; Reuse; *Chrysalidocarpus lutescens*

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1. Introduction

Recycling of wastewater is one of the main options when looking for new sources of water in water scarce regions and treatment of wastewater provides an effluent of sufficient quality that can be put for beneficial use instead of discharging it into the environment. Instead of growing research studies, a strong demand exists for the development of an effective, as well as, economic treatment of gray water particularly in the developing countries. Gray water comprises of water from showers, kitchen-sinks and laundry washing in homes, offices, etc. These wastewaters are potentially different from industrial wastewater as they might contain high organic load from food processing, utensil washing in the kitchen, soap and detergents, with the main contaminants

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*Corresponding author.

being proteins, carbohydrates, detergents, oil and grease and other dissolved and suspended compounds as well as harmful pathogens. Studies were carried out to reuse wastewater for non-potable applications in urban housing area. Here, two systems were applied for water reuse scheme, that is, using fixed-bed biofilm system for treating household wastewater and membrane bioreactor system for the treatment of wastewater from anaerobic pond [1]. UV disinfection method was employed in pilot-scale municipal wastewater treatment for subsequent agricultural reuse [2].

Membrane-based processes are being widely used for gray water treatment. Submerged hollow fiber membrane module had been extensively studied for the treatment of domestic wastewater with effective removal of organic matter and ammoniacal nitrogen [3]. Removal of organic matter and nutrients from municipal wastewater was achieved using submerged membrane bioreactor under anaerobic conditions [4]. Synthetic domestic wastewater was treated using a step-feed hybrid membrane bioreactor with effective removal of nitrogen and organic matter [5]. With increasing interest to produce pathogen-free water use of ultrafiltration (UF) membrane is gaining importance. The performance of an UF pilot plant was studied under two opposite operating conditions. Results from both the conditions were with par of the discharge limit with effective removal of pathogenic bacteria [6].

Although polymeric membranes are widely used; however, stability problem arises in prolonged uses. Majority of the polymeric materials suffer from morphological degradation in the harsh operating conditions [7]. Inorganic membranes, particularly, the ceramic membranes overcome these drawbacks and emerge as potential alternative for wastewater treatment. The Central Glass and Ceramic Research Institute have indigenously developed ceramic membranes from low-cost raw materials like clay and alumina [8]. The ceramic microfiltration (MF) treatment was used in combination with various physicochemical processes for different types of industrial [9,10] and domestic wastewater [11,12]. The combined processes were successful in terms of removal of organic load, suspended solids, turbidity, etc. and treated water quality were at par with the discharge norms of wastewater. In this study, performance evaluation of indigenously developed low-cost ceramic MF and UF membranes [13] in single-channel configuration (10 mm od/7 mm id) was investigated for the treatment of high organic loading wastewater. Efficiency of the MF and UF process was studied individually, and finally, a two-stage treatment involving MF followed by UF was proposed for reuse of the wastewater in gardening or agricultural uses. Domestic or gray water may serve as a good option for agricultural purposes due to their high nitrogen and phosphorus content. The literature survey reveals that although extensive studies have been conducted worldwide for the treatment of different types of wastewater; however, there is a need to study the reusability of the treated effluent including its effect on the environment. It has been observed that different constituents present in gray water may be potentially harmful to plants as well as soil and there is wide scope of scientific studies regarding changes in soil chemistry, as well as, specific short and long-term effects of using gray water on plants [14]. Therefore, apart from performance evaluation of ceramic MF and UF process, this study also brings another aspect, that is, responses of land plants toward wastewater during various courses of treatment. *Chrysalidocarpus lutescens* H. Wendl. [15], commonly known as Areca palm which is a popular palm species growing easily in indoor as well as outdoor, was selected for this study. Palms are those groups of plants that are widely grown in tropical, subtropical, mediterranean, and warm temperate climates. These groups of plants are monocots having single-shoot meristem and very much susceptible to slight variation in its nutritional pattern. Nutrient deficiency in terms of potassium and manganese shows visible effect in terms of physical appearance and health of palms on most soil types [16,17]. In this study, effects of untreated (feed), as well as, treated wastewater (permeate) from different membrane-based processes were observed on *C. lutescens* for short-term duration and the results were compared with respect to fresh water application.

2. Materials and methods

2.1. Wastewater characterization

Wastewater containing portion of solid and liquid food stuffs, oily residuals, and detergents was collected from washing of utensils in an office canteen at lunch time. The sample of wastewater was collected daily for a long period and subjected to characterization to identify a range of the various polluting matter present in the wastewater which were represented in terms of total suspended solids (TSS) [Tarsons, India], chemical oxygen demand (COD) [COD Digestor 2015 M, Spectralab, India], biochemical oxygen demand (BOD) [BOD Trak^{TM}II, Hach, USA], total dissolved solids (TDS), pH, and conductivity [Multiparameter Sension 156, Hach], turbidity [2100ANIS Turbidimeter, Hach] etc. Bacteriological analysis was performed on

Table 1
Characteristics of wastewater collected from canteen during different times of year

Parameters	Max	Min
pH	8.98	3.75
COD (mg/l)	10,500	780
BOD (mg/l)	2,020	340
O&G (g/l)	7.8	1.2
Turbidity (NTU)	450	38.7
TDS (mg/l)	7068.8	1,320
TSS (mg/l)	2,288	200
Conductivity (μS/cm)	2,436	993
MPN (per 100 ml)	9,10,000	2,20,000

treated and untreated wastewater and represented in terms of most probable number (MPN) per 100 ml [18] (Table 1). In characterization of water samples, the standard methods of APHA [19] were followed. Chemical analysis of wastewater and plant parts was carried out in Atomic Absorption Spectrophotometer (AAS) to measure the concentration of various inorganic components using AAnalyst 400 of Perkin Elmer, USA make.

2.2. Ceramic MF and UF membrane

The study was carried out with both porous ceramic tubular support and UF membrane prepared over the tubular support. Low-cost porous tubular supports were prepared by extrusion of a plastic body made of clay–alumina mixture through an indigenously prepared ceramic extrusion die. The green extruded support were then dried at room temperature over rotating rollers spun for 24 h before being heat treated at 1450°C for 1.5 h [13]. After sintering the porous support was coated with γ-alumina by conventional sol-gel and dip-coating technique [20]. For this study, UF-coated membrane (10.40 mm OD/6.55 mm ID/125.63 mm L) with average pore size of about 20 nm was chosen and tubular support (10.57 mm OD/6.65 mm ID/125.62 mm L) with 1 μm average pore size was selected for the MF study. The membrane surface was characterized by pore size, scanning electron micrograph images and clean water permeability. Pore size of the MF membrane was measured by mercury intrusion porosimeter (Quantachrome, PM60, USA). Pore size of the UF membrane was measured from SEM images (Leo S430I, Carl Zeiss, Germany). Filtration area of membrane was about 0.0026 m^2. For determination of permeate flux, 100 ml of permeate was collected at constant operating pressure and the permeation time was noted for three consecutive points the average of which was reported. Clean water flux of the MF membrane was about 648 $Lm^{-2}h^{-1}$ and that of UF membrane was about 404 $Lm^{-2}h^{-1}$ at an operating pressure of 2 bar. SEM images shown in Fig. 1(a) and (b) revealed the structural morphology of the tubular MF and UF membrane.

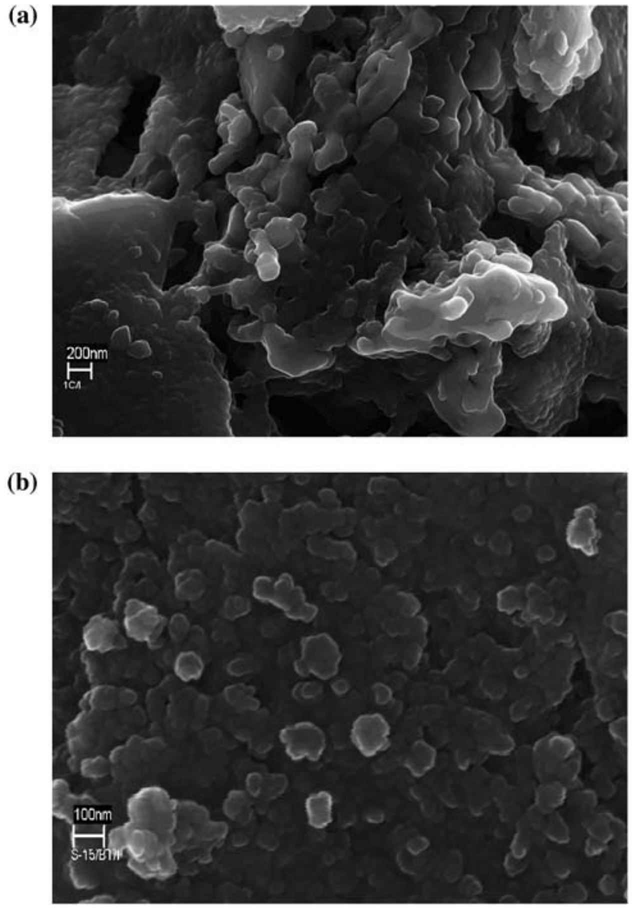
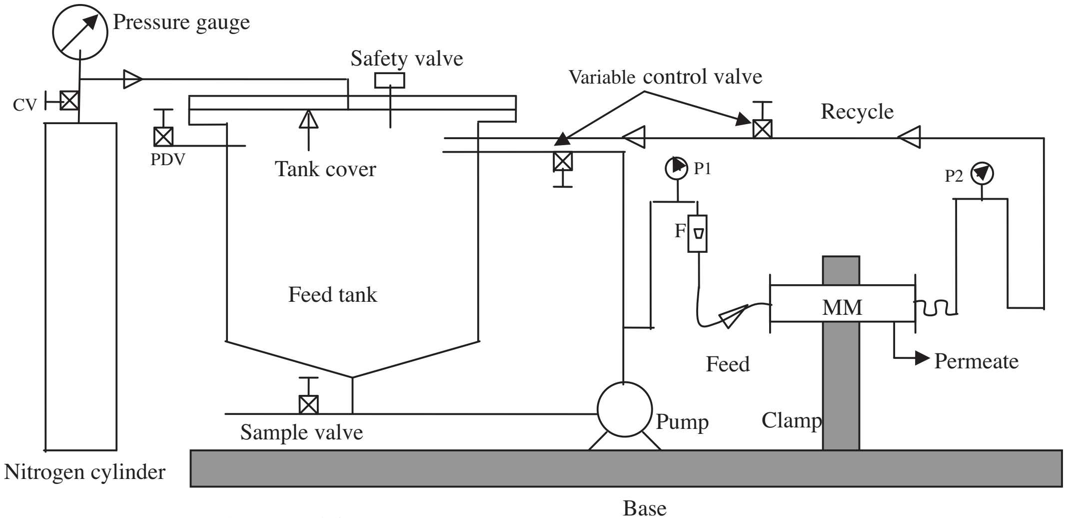


Fig. 1. SEM images of internal surface of unused membrane: (a) MF and (b) UF.

2.3. Experimental setup and process description

Crossflow filtration study was performed using an indigenously designed and fabricated membrane filtration unit (Fig. 2). The wastewater was introduced in the cylindrical feed tank (capacity 10 l, stainless steel body) by opening the tank cover. The tank was sealed by "O" ring after it was filled. The tubular membrane module was of Perspex make. The feed was circulated through the membrane using a positive displacement pump at a fixed flow rate by controlling the variable valves. The working pressure was obtained with a nitrogen gas source (nitrogen cylinder) as shown in the figure and was controlled by adjusting the cylinder and pressure



MM-Membrane module
CV-Cylinder valve
PDV-Pressure discharge valve
P1-Inlet pressure
P2-Outlet pressure
F- Flowmeter

Fig. 2. Schematic representation of membrane filtration unit for wastewater treatment.

discharge valves. All the experiments were carried out at room temperature of about 27–30˚C and transmembrane pressure (TMP) of 2 bar. Feed flow rate was fixed at 2 l/min. The membrane was conditioned by soaking it in pure deionized water for a minimum of 24 h before performing the experiments. Wastewater was treated by three processes, that is, MF, UF, and combination of MF and UF. In this study, COD (mg/l) was selected as representative parameter to denote concentration of organic matter in wastewater. The effect of constant pressure filtration study was carried out to observe the effect of COD removal with time. Permeate flux was expressed as LMH ($l/m^2/h$).

A periodical cleaning of the membranes were carried out, once stability was attained at a particular pressure in order to reduce the effect of variation of cake density on its flux due to different pressures. The experimental setup was cleaned using 0.1 N of nitric acid solution followed by 0.1 N of sodium hydroxide solution and finally with deionized water to remove the traces of cleaning chemicals.

2.4. Reusability study with wastewater

Effect of untreated and treated wastewater was observed on *C. lutescens*. Four sets of young plants of same age and height were selected among which one was for fresh water (control), one for untreated wastewater, one for MF-treated wastewater, and another for UF-treated wastewater. For each system, three sets of data were generated. The plants were watered for about three months, and the effect of treated and untreated wastewater was observed by measuring the concentration of various nutrients in different parts of the plants and soil.

2.5. Preparation of samples for elemental analysis of different parts of plant

Concentration of various elements present in root, stem, leaves, and soil of plants were measured using AAS. The samples were prepared according to the methods described by Kalra [21]. About 0.5–1.0 g each of root, stem, leaves, and soil were taken and digested using 5 ml of nitric acid in a beaker. The beaker was placed inside fume chamber with cover and digested at 125˚C for 1 h. Thereafter, the solution was allowed to cool and 1–2 ml of 30% hydrogen peroxide was added. The procedure was repeated until a clear solution was obtained. After complete digestion, the cover of the beaker was removed and the temperature was reduced to 80˚C. Heating was continued till near dryness. A white residue was obtained which was dissolved in the mixture of hydrochloric and nitric acid and subjected to analysis.

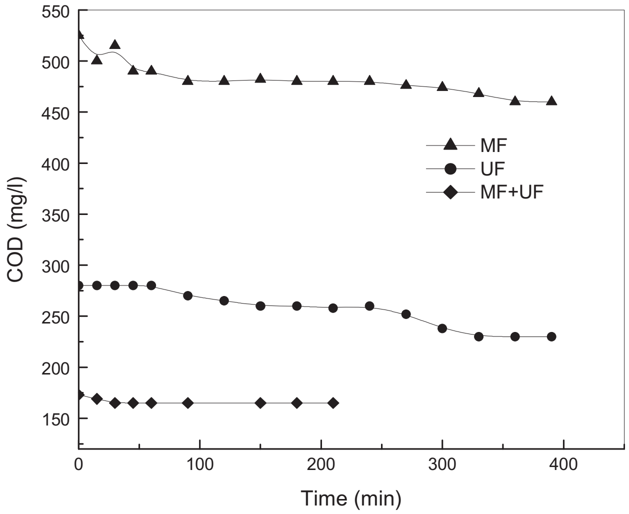


Fig. 3. Variation of COD with time during different types of membrane treatment (COD$_0$: 2050 mg/l; TMP: 2 bar; feed flowrate: LPH).

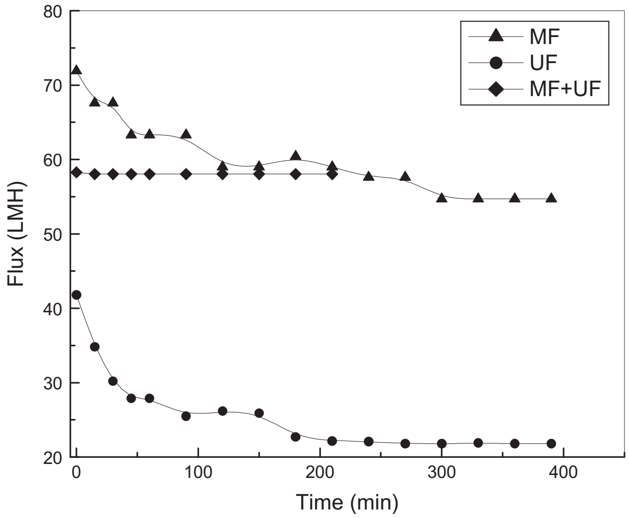


Fig. 4. Variation of permeate flux with time during different types of membrane treatment (COD$_0$: 2050 mg/l; TMP: 2 bar; feed flowrate: LPH).

3. Results and discussions

3.1. *Effect of time*

Effect of time was observed at a constant TMP of 2 bar with feed COD of 2050 mg/l (Fig. 3). In the single-stage MF and UF processes, permeate COD value gradually decreased with time. After 150 min of filtration, COD was found as 482 mg/l. Similarly, in the UF process, permeate COD value reduced from 280 mg/l to about 260 mg/l after 150 min which still continued to decrease with time. During filtration at constant pressure and flow rate, different solute particles in the feed got polarized near the membrane surface and eventually built up a dynamic layer over the surface of membrane. In addition to the concentration polarization effect, surface adsorption by the ceramic membrane and partial pore blocking of the membrane possibly contributed toward the gradual reduction in the COD values of permeate samples. Interestingly, in the two-stage process of MF followed by UF, permeate COD value was quite steady with time. The prior MF treatment resulted in substantial reduction in the organic load of the UF feed, and consequently, the effect of concentration polarization was almost negligible. Therefore, a constant COD value of 165 mg/l was obtained during 210 min of filtration.

Fig. 4 showed flux profile with time during constant pressure filtration. In both the MF and UF process, flux value gradually declined with time primarily due to the higher resistance offered by the accumulated layer on the membrane surface. It may be observed that in MF after 150 min of filtration, flux reduction was about 18% compared with the initial flux value. For UF, flux decline was more significant (38%) due to the smaller pore size of the membrane, and consequently, greater effect of concentration polarization combined with pore-blocking mechanism. Again in the UF preceded by MF, a steady-state flux value was found during the entire operating range of experiment. In this two-stage process, flux value was about 58 LMH which was about 120% higher than the flux value obtained after 150 min in the single-stage UF process.

3.2. *Characterization of wastewater before and after membrane filtration*

Tables 2 showed detailed characterization of the wastewater and permeate samples obtained in the different membrane processes along with the discharge norms for water reuse for agricultural purpose as per the recommendation of USEPA. The data showed that both MF and UF membranes effectively retained the turbid and the suspended materials present in the wastewater. However, substantial removal of other parameters likes organic loading (BOD and COD), oil and grease, and pathogenic microorganisms were obtained in the UF as well as, MF followed by UF process. For both these processes, permeate quality satisfied the discharge norms for agricultural reuse of water, either for food crops not commercially processed including surface or spray irrigation of any food crop or for food crops commercially processed and for non-food crops [22]. Based on these results, the application of treated wastewater from the single-

Table 2
Characterization of wastewater during various stages of treatment

Parameters	Untreated wastewater	Permeate (MF)	Permeate (UF)	Permeate (MF + UF)	Discharge norms for agricultural reuse[*]
pH	6.8–7.8	6.4–6.9	6.4–6.8	5.8–6.8	6–9[a,b]
COD (mg/l)	920–2,960	403–525	250–291	151–172	
BOD (mg/l)	340–768	24–31	⩽2	⩽4.5	⩽10[a], ⩽30[b]
O&G (g/l)	2.4–5.6	0.72–1.2	0.0001–0.0002	ND[**]	
Turbidity (NTU)	89–115	0.339–0.507	0.098–0.227	0.09–0.12	⩽ 2[a], –[b]
TDS (mg/l)	3030.5–6874.2	3,000–6,011	1,377–1,432	1,240–1,245	
TSS (mg/l)	84–345	16–29	ND	ND	–[a], ⩽30[b]
Conductivity (μS/cm)	845–1,578	800–958	452–500	380–480	
MPN (per 100 ml)	2,20,000–9,10,000	28,000–35,000	ND	ND	ND[a], <200[b] (Feacal coliform)

Notes: [*]as per Guidelines of water reuse – USEPA for Agricultural reuse-under category of ‘Urban Reuse’; [a]food crops not commercially processed, surface or spray irrigation of any food crop; [b]for food crops commercially processed and for non food crops (Blumenthal et al. 2000); [**]Not detected.

stage MF and UF process were observed on growth of Areca plant. Since no significant differences were observed between the separation achieved for the single-stage UF process and the combined MF and UF process, and hence, reuse study on plant was not conducted for the MF + UF-treated water considering practical aspect.

3.3. Characterization of ceramic UF membrane surface after wastewater treatment

Fig. 5 demonstrated microstructure of the internal surface of ceramic UF membrane observed using scanning electron micrograph after wastewater applications. The clean membrane surface (Fig. 1(b)) showed porous structure with surface roughness and irregularities. On the other hand from Fig. 5, it was observed that membrane surface after filtration showed deposition of particles in irregular fashion over the membrane surface, thereby resulting in blocking of some of the active pores.

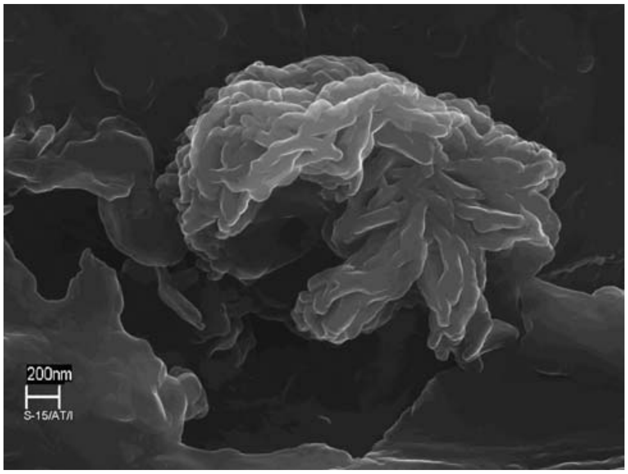


Fig. 5. SEM image of the internal surface of ceramic UF membrane after use.

3.4. Physiological effect

The plants grew well for both the MF-and the UF-treated wastewater in contrast with the plant administered with the untreated wastewater where visible effects like chlorosis of leaf and wilting was observed. It may be noted that palm plants are more prone to nutritional deficiency [23]. From Table 1, it was observed that the wastewater was rich in organic matter and oil which were indicated by the high COD and BOD loading and oil and grease content, although gray water contained considerable amount of phosphorus, potassium, manganese, magnesium, and iron. (Table 3), which are potentially beneficial for plant growth; however, the oil rich wastewater had substantial negative impact on palm plant. Similar observations were found in earlier studies [24,25] where the effect of oil spills on salt marshes and mangroves were found to be vulnerable facing acute and long-term damage. It was assumed that the presence of oils interfered with respiration of roots of these plants. Before the onset of experiments, the soil was analyzed for its chemical constituents and it was observed that the soil was very rich in iron (about 95 mg/kg), zinc (10 mg/kg), potassium (55 mg/kg), magnesium (85 mg/kg), etc.

3.5. Nutrients uptake

Apart from the physiological observations, remarkable effects were noted on mineral uptake by plants. Fig. 6 showed the concentration of various elements

Table 3
Concentration of different elements in the untreated and treated (MF and UF) wastewater used for the reuse study (concentration in mg/l, ±0.2).

Parameters	Untreated wastewater	MF treated wastewater (permeate)	UF treated wastewater (permeate)
Iron (Fe)	1.04	0.02	0.01
Calcium (Ca)	39.86	34.14	34.17
Magnesium (Mg)	13.82	14.84	15.08
Sodium (Na)	136.7	147.43	169.53
Potassium (K)	10.06	12.92	16.99
Manganese (Mn)	0.18	0.17	0.31
Phosphorus (P)	1.16	0.54	0.35
Copper (Cu)	1.28	0.01	0.19
Lead (Pb)	0.76	0.32	0.31
Zinc (Zn)	1.56	0.01	0.01

as found in analyzing the different parts of a plant, viz. roots, stems, leaves, and soil due to the application of different types of water. It could be observed from Fig. 6(a) that after administering untreated wastewater, calcium (Ca) content in root (1.4 mg/kg) decreased than that of control (6.18 mg/l). Similar trend was observed for stems (Fig. 6(b)) and leaves (Fig. 6(c)). On the contrary, calcium increased in soil to about 34% (Fig. 6(d)). This indicated that Ca uptake by plants decreased severely due to wastewater. This trend was observed for other nutrients like magnesium, sodium, and phosphorus. However, potassium (K) uptake was found higher in stems which might be attributed due to the presence of higher concentration of K in untreated wastewater, and consequently, concentration of K in soil (78.21 mg/kg) was more than that of control (50.5 mg/kg). It was observed that iron (Fe) concentration was low in roots. This was inhibitive for plant growth, since iron is essential for chlorophyll synthesis and deficiency of Fe leads to interveinal chlorosis [21]. In general, it was observed that application of untreated wastewater caused deficiency in some of the essential nutrients like calcium, magnesium, and sodium in different parts of the plant. Here, these elements were accumulated in soil mostly and the distribution of them through plant body was largely interrupted. Deficiency of potassium and manganese proves fatal for growth in palms [16].

On the contrary, application of the MF- and UF-treated water showed significant enhancement in the uptake of nutrients (Fig. 6) compared with that of the control. From Table 3, it may be observed that concentration of some of the nutrients like magnesium, sodium, potassium, etc. were more in MF- and-UF treated water compared with untreated water. This might be due to the fact that these minerals were masked in the untreated water due to the presence of high organic load, oil and grease. MF and UF process could not remove these minerals but were able to reduce the organic loading efficiently, resulting in reduction in the masking effect. Consequently, the treated water had higher concentration of these nutrients. However, after MF and UF treatment, iron, zinc, copper, and lead concentrations were reduced. In general, similar trends were observed in Fig. 6 for both of the treated water, viz. substantially higher uptake in the roots and leaves and lower accumulation in soil for most of the essential elements like Fe, Ca, Mg, K, Mn, and P. Here, it might be noted that Ca, Mg, K, and P were taken up by leaves more than that of roots and stem. Concentration of K was more in leaves than in stem and root. This could be due to the fact that potassium was a mobile nutrient, and due to its higher mobility, it reached leaf more quickly [26]. It might be noted that uptake of zinc by the plant followed similar pattern for all the three types of water, that is, untreated, MF treated and UF treated, whereas for fresh water, zinc was in more concentration in soil rather than that of root, stem, and leaves. The wastewater contained zinc which might contribute toward enhanced zinc uptake. Most common nutritional disorder in palms is caused due to potassium deficiency. Potassium deficiency causes leaflet tip necrosis. In many palms like *Cocos nucifera* (coconut palm), *Elaeis guineensis* (African oil palm), *Dypsis lutescens* (Areca palm), *Chamaerops humilis* (European fanpalm) etc., potassium deficiency was found to cause translucent yellow or orange spots on leaflet as well as necrotic spots [23]. From Fig. 6(c), it was observed that application of untreated wastewater caused decreased potassium concentration in leaves of palm plant, whereas concentration of potassium of those treated with MF and UF permeate was more. Calcium is the main component of cell wall [27], and it was observed that calcium content in leaves was more in case of MF- and UF-treated water (Fig. 6(c)) compared with that of untreated and fresh water. Well distribution of iron was found in soil and other parts of plant necessary for plant growth for both MF- and-UF treated water. Compared with MF-treated water, elemental uptake in roots was still higher to an extent of about 33, 35, 41, 21, and 23% for iron, calcium, magnesium, potassium, and phosphorus, respectively, for the UF-treated water. Concentration of these elements in

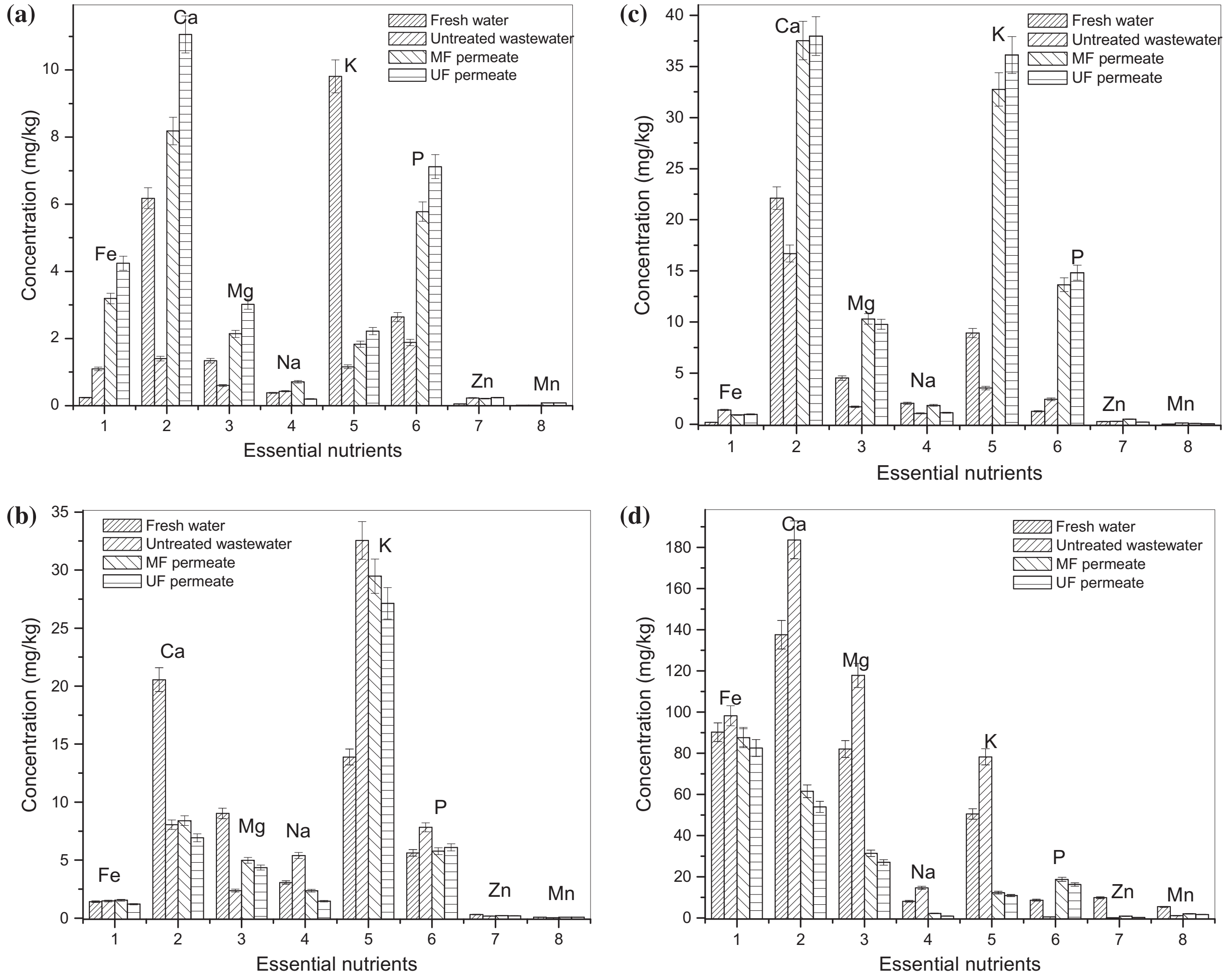


Fig. 6. Responsive behaviour of *C. lutescens* towards uptake of essential nutrients with application of various types of water: (a) in root; (b) in stem; (c) in leaves; and (d) in soil.

leaves was found almost similar for both type of water. Distribution of the elements like Fe, Ca, Mg, Na, K, and Mn were slightly higher in stems for the MF-treated water.

Behavior of the plant in the presence of wastewater varies for different plant species and plant physiology. Nutrient uptake by plants is a complex phenomenon which depends on potential difference of plant and surrounding soil. Increase in the negative charge of the water potential inside the plant compared with that of the surrounding soil facilitates the intake of more nutrients by plants from soil [21]. In this experimental study, the application of untreated wastewater proved to be detrimental to *C. lutescens.* This could be explained that higher amount of organic matter present in wastewater might interacted with the soil to produce large amount of negatively charged solids [27], which severely inhibited the intake of nutrients by the plant. The MF and UF treatment resulted in substantial reduction in organic loading, as well as, oil and grease content with an increased concentration of the minerals in the treated water. Hence, application of this treated water greatly facilitated the intake of the nutrients by the plants and a better distribution of nutrients was noticed compared with that of fresh water.

4. Conclusion

Ceramic MF and UF membranes prepared from cheap raw materials were used for the treatment of high organic loading and oil rich gray water. For wastewater with initial COD loading of 2,050 mg/l, COD reduction after 30 min at operating pressure of

2 bar was about 75% in the MF process which increased to about 86% in UF process. A two-stage treatment of MF followed by UF further increased the COD removal to 92%. Permeate flux in the single-stage UF was much lower (30 LMH) than the MF (68 LMH); however, the prior MF treatment substantially increased the flux in two-stage UF, that is, about 93%. For the MF followed by UF process, % COD removal was stable and a steady-state flux was obtained. Turbidity reduction was ⩾99% with substantial reduction of TSS in both MF and UF. Complete removal of oil and grease and coliform concentration were achieved in the UF process. Studies on application of different kinds of wastewater on *C. lutescens* revealed that effects of untreated wastewater on the plant were detrimental. The high organic loading and oil content in wastewater caused major interruption in the uptake of essential nutrients through the plant body, whereas for both MF- and-UF treated water, significant enhancement in the uptake of nutrients were observed in the plant compared with that of the fresh water. Nutrient uptake was higher for UF-treated water compared with the MF. The membrane treatments resulted in substantial reduction of organic loading, as well as, oil and grease content. The ceramic membrane–based treatment of high organic loading wastewater showed potential for reuse of the water in agricultural or gardening activity. The same may be reused for other applications, viz. floor and car washing, toilet flushing, etc. Based on these laboratory-scale results, the process may be upscaled for community applications which will be part of our future work.

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