



## Foulant and chemical cleaning analysis of ultrafiltration membrane used in landfill leachate treatment

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### ABSTRACT

Odayeri leachate treatment plant has a capacity of 2,400 m<sup>3</sup> leachate/d, and it has a full-scale membrane bioreactor (MBR) system with an external ultrafiltration (UF) membrane. A foulant analysis was conducted to the UF membrane after three years of operation by using inductively coupled plasma (ICP), total organic carbon (TOC), scanning electron microscope, energy-dispersive spectroscopy and Fourier transform infrared spectroscopy analyses. Studies showed that there is a 3- $\mu$ m thick fouling layer, which consists of complex inorganic and organic interactions on the membrane surface. Following the analysis, the membranes were chemically cleaned to find an effective cleaning method for improving the membrane performance. Acidic, alkaline and oxidative solutions were used separately or in sequence. In summary, for the removing of foulants from the membrane surface, the application of an effective chemical cleaning strategy is very important. Using acidic, alkaline or oxidative solutions separately was not an effective way to remove foulants from the membrane surface. Even in the combined use, the sequence of the chemicals was important. It was found that correct sequence should be using first the alkaline and then the acidic solution or using first the oxidative and then the acidic solution to obtain an effective cleaning for UF membranes used in MBR for treating landfill leachate.

*Keywords:* Leachate treatment; MBR; UF membrane fouling; Foulants; Chemical cleaning

### 1. Introduction

Recently, membrane technologies have become “conventional technology” for wastewater treatment. However, fouling still stands as a big obstacle for the development of this technology. By reducing the fouling, it is possible to increase the range, type and lifetime of the membrane applications.

Pre-treatment of feed flow, optimization of the operation conditions and enhancement of anti-fouling

characteristic of membranes may prevent fouling before occurring [1]. Fouling can be related to different mechanisms including pore blockage and cake layer formation [2]. Foulants first interact with membranes; then foulant–foulant interaction occurs [3]. Generally, different fouling mechanisms are combined one by one, or they occur at the same time [4,5]. Membrane cleaning should be regularly applied to restore membrane properties and its initial flux [1]. While restoring the initial flux, both foulant–foulant and membrane surface–foulants interactions should be annihilated [6].

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The removal of the substances from the membrane surface or pores is called as membrane cleaning [7]. It is possible to clean membranes in two ways. If chemicals are used, it is called as “chemical cleaning”, and if mechanical forces are used, then it is called as “physical cleaning” [8].

Acids/bases, oxidants, surfactants, chelating agents and enzymes [9] are commonly used chemicals for the cleaning of the membranes. Acids and bases are considered as the most common cleaning agents used in membrane cleaning due to their low cost and high efficiencies [10].

Hydraulic cleaning [11] and ultrasonic vibration [12] can be thought as physical cleaning methods. In most cases, especially in long-term operations, physical cleaning remains incapable, and chemical cleaning becomes inevitable. Using physical and chemical cleaning in sequence is the most common approach for membrane cleaning.

An efficient cleaning depends on the concentration of the cleaning agent, the frequency of the cleaning period and the duration of the cleaning. Improper cleaning strategies, incorrect operating conditions and unstable feed flow characteristic cause rapid membrane fouling. In such cases, a membrane autopsy becomes a must to understand the nature of the problem. Therefore, membrane autopsy gives specific recommendations for pre-treatment and cleaning methods [13]. A good diagnosis and autopsy present effective strategies to control the membrane fouling [14].

There are several autopsies and foulant analysis studies in the literature considering nanofiltration (NF)–reverse osmosis [15–20] and microfiltration–ultrafiltration (UF) membrane autopsies [13,21,22]. However, there are a few researches on UF membrane autopsy, which has been used in the landfill leachate treatment in membrane bioreactor (MBR). In this study, a foulant and chemical cleaning analysis of UF MBR membrane, which belongs to Odayeri leachate treatment plant (41°13'15.2" N, 28°51'10.3" E), was performed. A chemical cleaning study was performed, and suitable cleaning strategy was found.

## 2. Materials and methods

### 2.1. MBR treatment plant and membrane description

Odayeri leachate treatment plant is located in Istanbul, Turkey. This plant treats the leachate water coming from Kemberburgaz/Odayeri sanitary landfill area. The landfill produces 2,400 m<sup>3</sup> leachate/d. Leachate treatment plant consists of primary settling, MBR (nitrification–denitrification–UF) and NF unit as seen in Fig. 1.

Leachate coming from the landfill is processed in the primary settling tank where suspended solids and chemical oxygen demand (COD) are partially removed. After primary settling tank, water flows directly to the bioreactor unit that consists of aerobic and anaerobic parts followed by membrane treatment unit.

Leachate equalization tank and pre-sedimentation tank volume was 2,000 and 285 m<sup>3</sup>, respectively. Settled solids were delivered to the sludge tank from the bottom of the tank. Supernatant leachate was sent to the biological reactor unit. Surface and jet aerators were installed for oxygen supply in order to provide homogenous mixture in the reactor. Additionally, heating system was installed for winter season to sustain required microbial kinetic in the reactor.

Characteristics of the influent leachate are summarized in Table 1. Average total nitrogen (TN) value of Odayeri leachate treatment plant was 3,548 mg/L, and the most of the TN was treated in the pre-anoxic biological treatment system. Treatment efficiencies for TN concentrations varied between 59% and 94%. Influent leachate concentrations of NH<sub>4</sub>-N varied between 1,094 and 3,595 mg/L, and average treated leachate was found as 2,713 mg/L. Average total phosphorus treatment efficiency at pre-anoxic biological treatment stage was found 79.3% in treatment system [23]. First stage is the UF unit that separates the sludge from the permeate stream. In the UF treatment unit, Pentair X-Flow Compact 33 tubular cross-flow UF membrane modules were used, and total suspended solids (TSS) were removed. The retentate obtained from the UF unit is returned back to the bioreactor. On the other hand, the permeate goes to the NF unit. UF unit works as cross-flow configuration, which decreases the fouling potential and increases the membrane flux. UF membranes permeate flow was ~90–100 m<sup>3</sup>/h, and NF membranes treating capacity was ~22–23 m<sup>3</sup>/h. In total 2,000 m<sup>3</sup> leachate was treated daily [23].

Second stage is the NF unit, which removes remaining COD, organic micropollutants, heavy metals and other components such as humic acid, color, etc. This unit consists of spiral wound membranes, which produce low retentate (<10%), and NF permeate is discharged to the sewage.

Rapid and irreversible fouling was experienced in the UF unit after 3 years of operation. Membrane autopsy was done on UF membranes within the scope of present work.

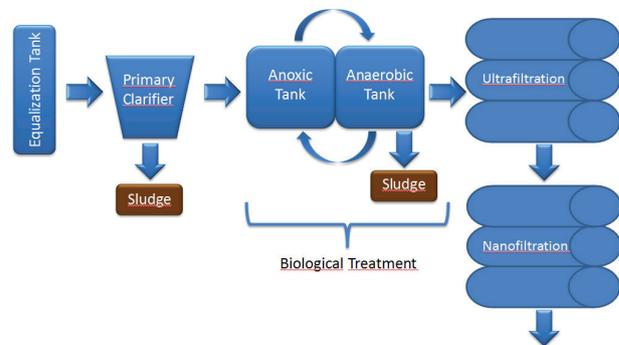


Fig. 1. Odayeri leachate treatment plant schematic.

Table 1  
Raw leachate characteristics of Odayeri treatment plant [23]

Parameter	Raw leachate	
	Min.	Max.
pH	5.5	8.5
BOD <sub>5</sub> , mg/L	3,000	13,000
Temperature, °C	15	20
TSS, mg/L	300	1,500
SO <sub>4</sub> , mg/L	5	500
Total hardness, mg/L CaCO <sub>3</sub>	1,400	2,500
Conductivity, µs/cm	30,000	40,000
Total alkalinity, mg/L CaCO <sub>3</sub>	8,000	13,000

## 2.2. Dismounting of the membrane module vessel and visual inspection

Membrane module vessel was cut down longitudinally without damaging the membrane fibers. Preventing the contamination of the membranes from the particles splattered during module opening process, a vacuum system was used. Membrane module dismounting stage can be seen in Fig. 2.

## 2.3. SEM and EDS analysis

Membrane surface was characterized by using an FEI Quanta FEG 200 SEM. Samples were coated with 5 nm of gold and palladium (Au-Pd) by using Quorum SC7620 ion sputtering equipment. Scanning electron microscope (SEM) images were taken under 24,000 $\times$ , 12,000 $\times$ , 10,000 $\times$  and 3,000 $\times$  magnifications. METEK EDAX Apollo X was used for energy-dispersive spectroscopy (EDS) analysis.

## 2.4. Analysis of foulant layer

Foulant layer structure was examined by Fourier transform infrared spectroscopy (FTIR) using Perkin Elmer universal ATR sampling equipment. Foulant deposits were extracted and then analyzed with inductively coupled plasma (ICP) and total organic carbon (TOC). Total membrane area of 75 cm<sup>2</sup> was cut from different sections of the tubular membrane module. Each piece of randomly chosen membrane samples were dipped into 100 mL solutions containing 0.8 M HNO<sub>3</sub> and 0.1 M NaOH, and awaited in ultrasonic bath for 210 min. After that, this solution was filtered using 0.45  $\mu$ m filter prior to analysis. Perkin Elmer Optima DV 3000 ICP-OES equipment was employed for inductively coupled plasma-optical emission spectrometry (ICP-OES) analysis. In the analysis, Al, Si, K, Na, Ca, Mg, Ba, Sr, Cr, Cu, Pb, Fe, Ni, Cd, Hg and S elements were scanned. Shimadzu TOC VCP-N equipment was used to find the TOC.

## 2.5. Chemical cleaning studies

Different chemical cleaning strategies were investigated in order to determine the best chemical cleaning agent. 0.5%



Fig. 2. Dismounting of the membrane module vessel.

Table 2  
The chemical cleaning strategies examined on the membrane pieces

Single	Acidic + alkaline	Acidic + oxidative	Alkaline + acidic	Oxidative + acidic
HCl	HCl + NaOH	HCl + NaOCl	NaOH + HCl	NaOCl + HCl
NaOH	HNO <sub>3</sub> + NaOH	HNO <sub>3</sub> + NaOCl	NaOH + HNO <sub>3</sub>	NaOCl + HNO <sub>3</sub>
HNO <sub>3</sub>	–	–	–	–
NaOCl	–	–	–	–

of NaOCl, 5% of NaOH, 5% of HNO<sub>3</sub> and 5% of HCl concentrations were used, respectively, for chemical cleaning strategies which were given in Table 2. Membranes were exposed to the chemicals for 6 h.

The solutions were prepared in a glass bottle, and random membrane samples were dipped in the solution. Shaking table was used to increase the contact between the chemicals and the membrane sheets.

## 3. Results and discussion

### 3.1. UF membrane foulant analysis

UF foulants can be divided mainly into four classes as particulates, macromolecules, ions and biological substances [24]. SEM, EDS, FTIR, ICP and TOC analyses revealed that membrane surface had a complex foulant layer.

SEM is a widespread method to see macromolecular foulants and depositions on membrane surface [25]. SEM is useful method for observing UF membranes as well. SEM images of UF membranes were given in Figs. 3(a)–(d). According to Figs. 3(a)–(c), the layer covering the membrane surface can be clearly seen. Microbiological and crystal structures were observed on the membrane surfaces that can be interpreted as a film layer on the membrane surface containing both organic and inorganic components. From the cross section of the membrane (Fig. 3(d)), the average cake layer thickness was predicted as about 3–4  $\mu$ m. The cake formation is related to the building up of foulant layers, and thickness formed on the membrane was not common for the membrane modules operating in the cross-flow configurations. The cake layer creates an additional resistance to the filtration and can be addressed to the increased pressure for compensating the flux decline. Also it was hard to remove thick film layers.

After the film layer on the membrane surface is confirmed, to determine the composition of the film layer, EDS analysis was conducted. EDS mapping was shown the type of the foulants and their locations in Fig. 4. According to the researchers [26,27], pH increase provides optimal conditions for struvite precipitation. Struvite solubility is pH dependent; at lower pH, struvite remains in liquid form; and at high pH values, it precipitates to solid form [26]. Apart from the operation data, pH in bioreactors is not high enough to form struvite structures. According to the results, the membrane surface were mainly covered with Na, Si, Mg, Ca and K elements as well as C and F. However, polyvinylidene fluoride (PVDF) membrane structure includes C and F; therefore, C and F elements were neglected in the results.

Some elements were found in EDS mapping results that were superimposed to understand the white particulate precipitate structures found on the membrane surface. As it can be seen in Figs. 5(a)–(d), separate Na (Fig. 5(b)) and

Cl (Fig. 5(c)) mapping results were superimposed onto the raw SEM image (Fig. 5(a)). According to this superimposing, the image in Fig. 5(d) was obtained. It can be inferred that the white particulate structures were NaCl salts shown in Fig. 5(d), which may be formed on completely dried membrane surface before SEM analysis.

To comment on the penetration depth of the foulants into the membrane, EDS mapping through membrane cross section was conducted. The results are presented in Fig. 6. Although Cl, Ca and Na elements were found on the membrane surface, the primary foulant through the membrane cross section was found as iron (Fe) element. Fe element was

dominant inside the membrane, and the fouling occurred through the membrane cross section was related to the iron.

For the removal of inorganic and organic foulants, acidic and caustic solutions were used, respectively. According to TOC and ICP results (Tables 3 and 4), it can be concluded and validated that the caustic solution was more effective than the acidic solution for the removing of organic materials. However, the amount of inorganic materials, which were removed by using caustic solution, were more than or equal to the acidic solution results. It can be interpreted as inorganic materials that were stuck in organic materials so that the use of caustic solution promotes their removal. Therefore, it can be concluded that using single chemical cleaning strategy was not effective.

FTIR analysis graph is presented in Fig. 7. Broad absorp-tion peak between 3,200 and 3,400  $\text{cm}^{-1}$  indicated the presence of polysaccharides [19]. Peaks observed at 1,630 and 1,540  $\text{cm}^{-1}$  were belonged to amide I and amide II bonds,

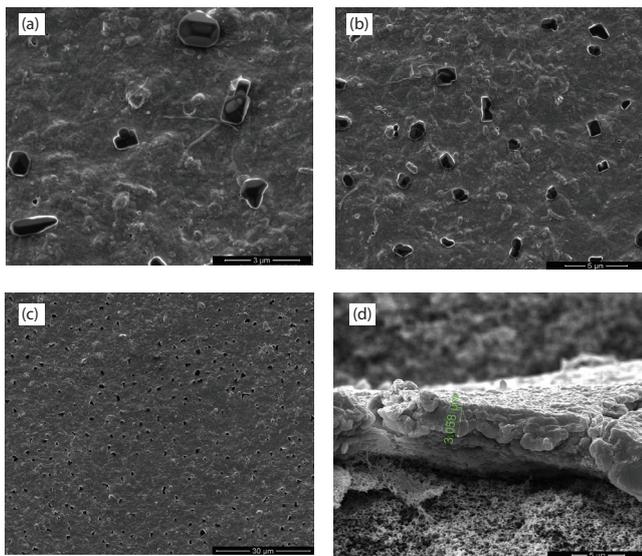


Fig. 3. SEM images of fouled: (a)–(c) membrane surface (at 24,000 $\times$ , 12,000 $\times$  and 3,000 $\times$  magnifications, respectively), and (d) cross-section image of fouled membrane, at 10,000 $\times$  magnification.

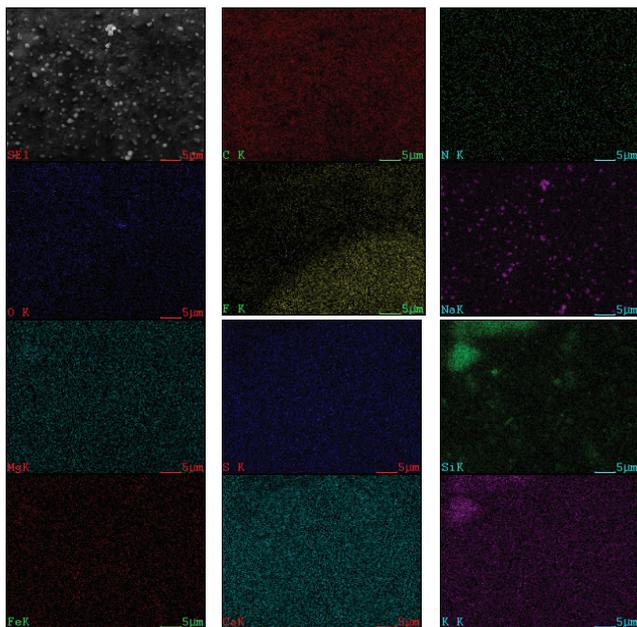


Fig. 4. EDS mapping of the membrane surface (element abbreviations can be found in lower left of the images).

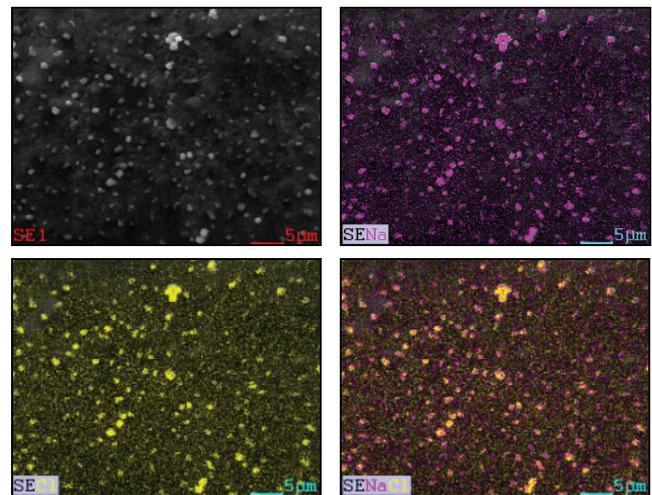


Fig. 5. EDS mapping of (b) Na and (c) Cl superimposed onto (a) a normal SEM image. (d) Superimposed EDS mapping shows NaCl salts.

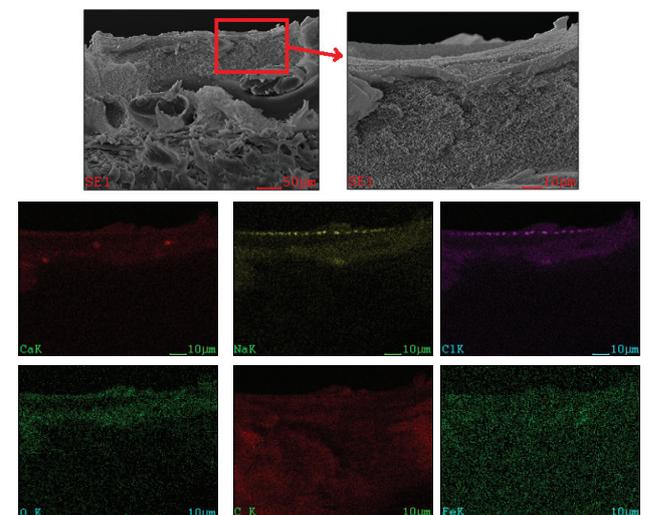


Fig. 6. EDS mapping images of the membrane cross sections.

which indicates protein presence on the membrane surface [20]. Peak observed at  $1,040\text{ cm}^{-1}$  was belonged to C–O bond, which are the indicators of carbohydrate and polysaccharides. According to FTIR analysis, a biofilm layer can be confirmed on the membrane surface.

### 3.2. Use of different chemical cleaning strategies

Different cleaning agents were applied in order to see the effects of chemical types. Digital and SEM images of chemically cleaned membranes can be seen in Figs. 8(a) and 9(a). When only HCl was used for chemical washing of the membrane, no distinct color change was obtained (Fig. 8(a)). It preserved its brown color showing that the remained fouling layer on the surface. Organic fouling and bacteria structure can be seen on membrane surface from the SEM images of the cleaned membranes with HCl (Fig. 9(a)). Acids are useful to dissolve and remove inorganic precipitates, metal oxides and hydroxides [1]. FTIR graphs prove that HCl was useful to remove inorganic foulants; however, there were still organic foulants on the membrane surface. As it seen on Fig. 7, the presence of polysaccharides, proteins and C–O bonds at  $3,200\text{--}3,400\text{ cm}^{-1}$ , at  $1,630$ ,  $1,540$  and  $1,040\text{ cm}^{-1}$ , respectively. According to FTIR analysis, remained biofilm layer can be confirmed on the membrane surface.

Additionally, no distinct color change (Fig. 8(b)) was observed when only  $\text{HNO}_3$  was used as a cleaning agent. Organic foulants can still be seen (Fig. 9(b)) on the membrane surface.  $\text{HNO}_3$  was effective for the removal of inorganic

foulants like HCl [1].  $\text{HNO}_3$  is being preferred in many plants to remove calcium precipitates. Additionally, in some cases  $\text{HNO}_3$  is a strong oxidising agent to clean many organic and biological foulants by nitration [28]. However, it has not shown the same effectiveness for organic foulants in this case. As seen on FTIR graph (Fig. 7), there are still organic foulant peaks similar to HCl FTIR results.

When only NaOCl was applied for cleaning of the membranes, the color became whiter (Fig. 8(c)). SEM images (Fig. 9(c)) and FTIR graphs showed that the organic foulants were removed but the inorganic precipitates stayed on the membrane surfaces and the most of the membrane pores were opened. Typical PVDF peaks  $841$ ,  $880$ ,  $1,072$ ,  $1,173$ ,  $1,273$ , and  $1,404\text{ cm}^{-1}$  were become much more visible after foulant layer removed on surface.

Table 3  
ICP and TOC analyses results of the membrane foulants

	Caustic average ( $\text{mg}/\text{m}^2$ )	Acidic average ( $\text{mg}/\text{m}^2$ )
Ca	56.613	75.507
Mg	15.493	16.747
Na	–	341.587
Hg	2.773	5.267
Pb	0.413	0.427
Cd	0.147	0.107
Zn	11.547	2.240
Ni	0.293	0.240
Cu	0.760	0.387
Cr	5.627	1.053
Fe	7.987	9.747
Sr	0.040	0.027
TOC (mg/L)	21.45	4.98

Table 4  
Percentage distribution of ICP analysis results of the membrane foulants

	Sr (%)	Ca (%)	Mg (%)	Na (%)	Hg (%)	Pb (%)	Cd (%)	Zn (%)	Ni (%)	Cu (%)	Cr (%)	Fe (%)
Caustic cleaning foulant distribution	0.56	55.36	15.15	–	2.71	0.40	0.14	11.29	0.29	0.74	5.50	7.81
Acidic cleaning foulant distribution	0.11	16.64	3.69	75.2	1.16	0.09	0.02	0.49	0.05	0.09	0.23	2.15

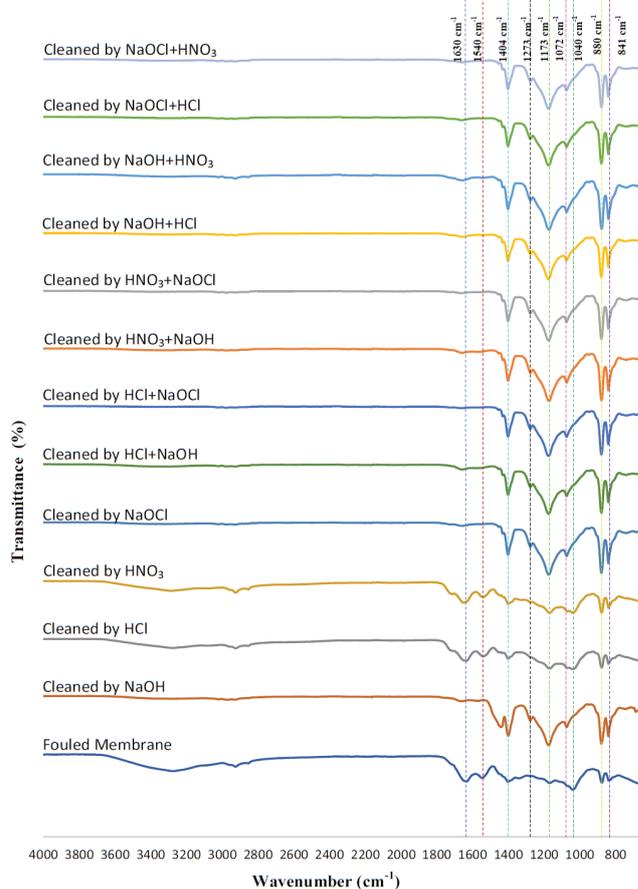


Fig. 7. FTIR graphics of the membrane surfaces.

Brownish color of the membrane was removed when only NaOH was applied (Fig. 8(d)). Hydrolysis of proteins and polysaccharides can be supported by alkalis like NaOH

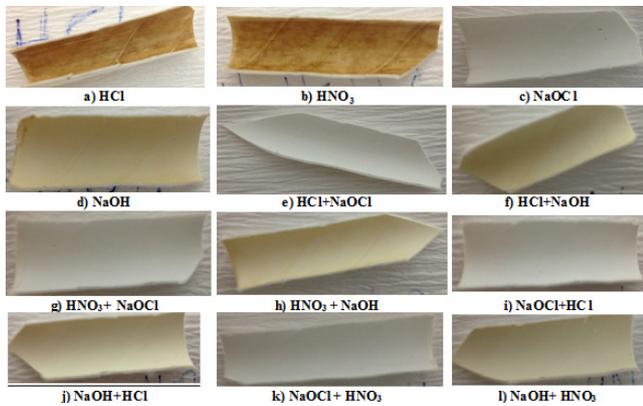


Fig. 8. Digital images of the cleaned membranes with different cleaning combinations.

and others [1]. Therefore, as it is expected, organic foulants were removed successfully from membrane surface.

After that, the cleaning chemicals were used sequentially for the cleaning of the membrane surface. First, HCl + NaOCl combinations were tried. Brownish color was removed, and the membrane was whitened (Fig. 8(e)). Pores located on the membranes surface were relatively cleaned. Although the organic foulants were removed, the inorganic foulants still exist on the membrane surface (Fig. 9(e)). The presence of the inorganic foulants after cleaning was addressed to the improper sequence of the cleaning agents. On the membrane surface, the organic fouling was dominant. The inorganic fouling was stuck between the organic fouling. Therefore, when initially acidic HCl was applied, it did not remove the organic foulants. Later, when oxidative solution was applied, only organic foulants were removed. So, partial cleaning of the membrane was obtained. When HCl + NaOH or HNO<sub>3</sub> + NaOCl were used, the result was nearly the same with HCl + NaOCl (the sequence of acidic then alkaline or oxidative).

The sequence of the cleaning agents were changed to first alkaline/oxidative and then acidic solution sequence.

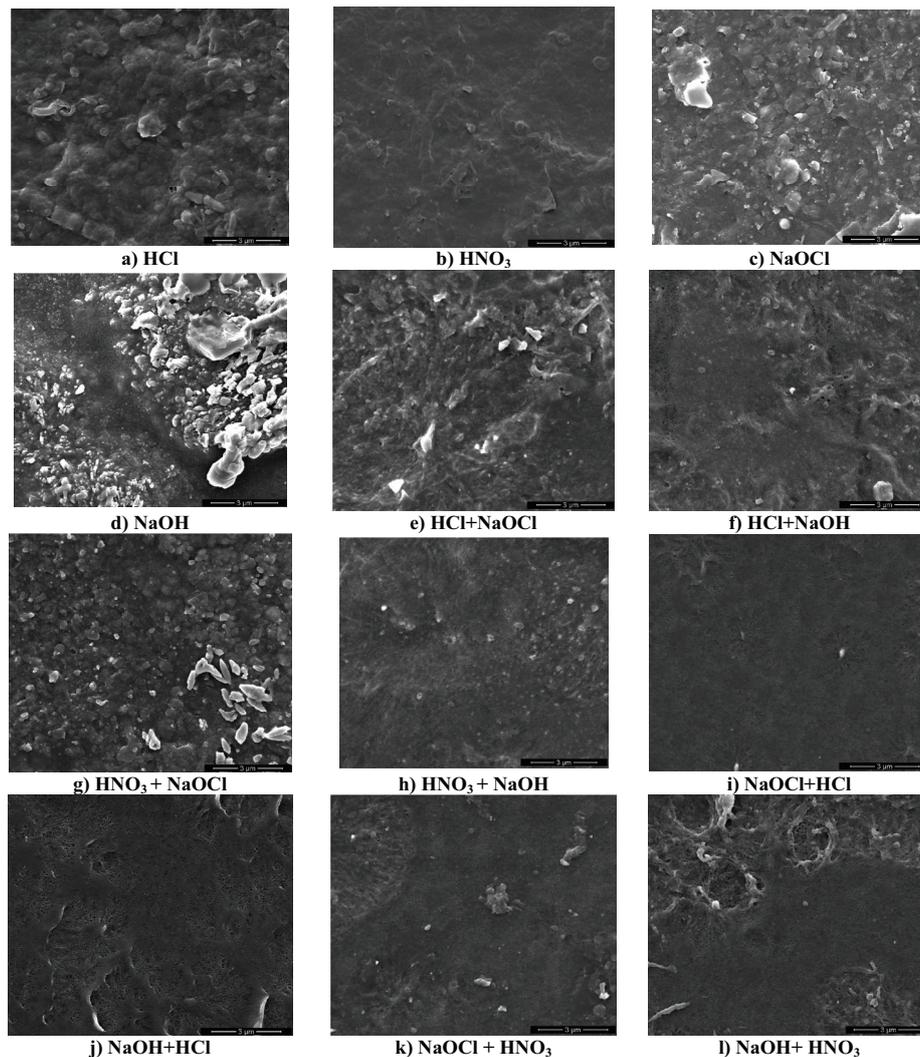


Fig. 9. SEM images of the cleaned membranes with different combinations.

Alkaline/oxidative + acidic combinations were tried by using NaOH + HCl, NaOCl + HCl and NaOH + HNO<sub>3</sub> solutions. In these cases, the membrane surface was cleaned thoroughly. Brownish color was removed from the membranes. Organic foulants were successfully removed when alkaline solution was used before the acidic solution. When acidic solution was applied, it was possible to remove inorganic foulants. The performance of NaOH + HNO<sub>3</sub> was found as effective as other alkaline/oxidative + acidic combinations.

#### 4. Conclusions

UF membrane surface was covered with a thick fouling film layer while treating landfill leachate in MBR. The film layer was consisted of both organic and inorganic foulants. These organic and inorganic foulants formed a complex structure that requires an effective chemical cleaning strategy for the removal of membrane fouling at the plant.

The application of combined chemical cleaning methods such as acidic and alkaline or acidic and oxidative sequence was found as ineffective for the removal of inorganic foulants since they were able to remove fouling partially. Alkaline solution was able to remove organic foulants in this case.

When the sequence was changed to alkaline and then acidic/oxidative and then acidic chemical agents, both organic and inorganic foulants were removed from the membrane surface thoroughly. Alkaline solution removed the organic foulants as well as the inorganic foulants stuck in organic foulants. Then, the use of acidic solution cleared the membrane surface from remaining inorganic foulants successfully. The most successful combination was found as NaOCl + HCl.

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