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Influence of Salinity Level on the Treatment Performance and Membrane Fouling of MBRs Treating Saline Industrial Effluent

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Abstract: This study investigated the potential impact of salinity levels on the treatment performance and membrane fouling of MBR seeded with sludge from saline industrial effluent treatment plants. A pilot-scale MBR received mixed saline industrial effluents at an organic loading rate (OLR) of 1.3 g COD/L·d and a feed-to-micro-organism (F/M) ratio of 0.33 g COD/g TSS. The effects of the variable salt concentrations of 5, 10, 20, and 25 g/L were investigated. The ranges of ammonia and total nitrogen (TN) concentrations were 22.2–26.3 mgN/L and 55.1–59.2 mgN/L, respectively. The MBR achieved promising results for chemical oxygen demand (COD) and biochemical oxygen demand (BOD), with removal ranges of 95.4–97.2% and 98.3–98.8%, respectively. The system provides 93.2–96.7% and 81.6–92.5% for ammonia and TN removal. Up to a 20 g/L salinity level, there were no significant effects on treatment performance, but 25 g/L significantly declined daily and specific COD removal load. Despite this, residual values at 25 g/L were better and met the Saudi standard for effluent discharge. This is due to membrane fouling which declined the flux rate with a spontaneous reduction of OLR and F/M ratio. The MBR system inoculated with high-salinity-adapted sludge could be managed to release treated effluent that meets Saudi disposal limits by modifying the F/M ratio via reducing the flux or increasing the mixed liquor suspended solid (MLSS) concentration.

Keywords: high-salinity-adapted sludge; MBR; mixed saline industrial wastewater; specific removal COD load; F/M ratio

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1. Introduction

The discharge of wastewater without proper treatment greatly affects aquatic ecosystems [1,2] soil fertility [3], and groundwater [4], subsequently contaminating the food chain with associated human health risks [5]. Many industries generate excessive saline industrial wastewater [6–8], severely impacting ecological systems [9–11]. Saline industrial wastewater is being discharged from the coking industry [12,13], oil and gas industry [14,15], marine and fish-processing industry [1,16], textile industry [17], tannery industry [18–21], and pharmaceutical industry [22].

Saline industrial wastewater from previous sources has significant concentrations of hardly biodegradable and recalcitrant organic contaminants, which are strongly resistant to biodegradation by conventional treatment methods [13,23,24] and physicochemical processes [9]. Saline industrial wastewater has extreme pollution-inducing properties and hard resistance to biological degradation; thus, its effective treatment and safe disposal have become potentially predominant [25]. Saline industrial effluents with a salt concentration below 10 g/L are defined as low-salinity industrial wastewater and saline industrial effluents with a salt concentration between 10 and 30 g/L are defined as high-salinity industrial wastewater [26]. Generally, non-modified activated sludge plants can handle low saline effluents with a salt concentration below 10 g/L without adverse effects, but above

10 g/L, high salinity causes dehydration and cell damage [27,28]. The characterization of microbial consortium and their behaviors in two sequential batch reactors (SBR) with adapted and non-adapted activated sludge was carried out during the treatment of saline synthetic wastewater; the results indicated non-significant differences, with a 96% and 95% removal of COD in the non-adapted and the adapted sludge, respectively, at a 10 g/L salinity level [29]. Others reported detrimental effects of high-saline industrial effluents on the biological activities of conventional activated sludge [28], due to the absence of halophilic (salt-tolerant) bacteria and salt-adapted micro-organisms [27,30]. Impacts of high saline industrial effluent on the anaerobic–anoxic–oxic treatment process have been investigated, and the data revealed the significant inhibition of the microbial growth of the activated sludge communities and structural and functional changes; all these suppressed nitrogen transformations have associated nitrite accumulation and enhanced greenhouse gas emissions [26].

Conventional bioreactors (aerobic and anaerobic) can effectively treat high-saline industrial wastewater via sludge adaptation and sludge enrichment with salt-tolerant microbial consortia [25]. Gradual increase in salinity levels during the treatment of saline industrial effluent enables the system to grow well-adapted sludge that works effectively at high salinity levels [31]. The acclimation of biological sludge to elevated salinity generates salt-tolerant micro-organisms [27,32], and this enables the system to produce higher-quality effluent than the ordinary activated sludge without adaptation. Di Trapani et al. [33] reported good adaptation for biological sludge to treat high-saline wastewater by using a gradual increase in the salinity. An SBR was applied for treating tannery effluent with 35 g/L salt content, and the system successfully declined COD by 95% from the 3000 mg/L initial concentration after sludge adaptation; however, both the adaptation period and hydraulic retention time (HRT) seemed too long [18]. The adaptation and acclimation of activated sludge biomass to salinity has been proven to be feasible up to a salinity level below 50 g/L [18]. On the other hand, the authors of [27] reported a possible treatment of hypersaline wastewater (up to 100 g/L NaCl) in the MBR systems after inoculation with halophilic bacteria.

The inoculation of the biological treatment system with halophilic bacteria limits the detrimental effects of salinity on the treatment process [9]. The inoculation of the biological treatment system with salinity-tolerant (halophilic) micro-organisms can boost sludge growth and treatment performance [34], and provide excellent treated effluent. The process was successfully applied to high-saline wastewater treatment with the significant alleviation of the inhibitory effects of a high-salt concentration [35,36]. An SBR-inoculated halophilic bacteria isolated from an activated sludge system treating effluent of the fish-canning industry at 24 h HRT and 23 d sludge retention time (SRT), was used to effectively treat diluted effluent from the fish-canning industry with 30 g NaCl/L [16]. Studying the effects of salinity fluctuation on halophilic bacteria within aerobic granular and flocculent sludge indicated that granular sludge was superior to flocculent sludge in treatment performance and the ability to recover performance after long-term shock loads; this is because granular sludge retrieves a stable performance of BOD removal (90%) after 18 days, whereas flocculent sludge retrieved performance (82%) after 27 days [16]. Similarly, after long-term shock loads, the loss of the nitritation process was higher in the halophilic flocculent sludge, which experienced a decline from 4.5 to 0.24 mg N/g VSS·h compared to a decline from 3.8 to 0.73 mg N/g VSS·h in the granular halophilic sludge [17].

The MBR process is a well-known innovative technology for wastewater treatment. It is characterized by a long SRT, which potentially improves the treatment performance of saline industrial effluent because the high concentration of salt increases the lag times of saline-tolerant micro-organisms [37], and makes them slow growers. Working at long SRTs prevents the washout of the salt-tolerant micro-organisms and other slow-growing micro-organisms from the system. Alighardashi et al. [38] reported that a long SRT and the formation of a large floc size in the MBR system make it work reliably under different operating conditions. Compared to conventional activated sludge, the MBR process had better treatment performance and showed very high efficiency during the treatment of saline effluent contaminated with organic matter and ammonia [32,33]. In general, typical MBRs without adapted sludge or halophilic bacteria can be adjusted to treat low-saline effluent (up to 10 g/L salt concentration) [27]. The operation of nitrifying MBRs indicated possible operation without negative impacts up to a salinity concentration below 10 g/L [39,40]. Nitrifying MBRs show stable nitrification performance without any significant effects during the treatment of ammonia-rich (30–250 mgN/L) effluent with a salinity range of 5.6–9.1 g/L [41], providing a final treated effluent with a residual ammonia concentration of less than 1 mg/L. However, the rise in salinity above 10 g/L negatively affected the nitrification process, with drastic changes in nitrifying community structure [41–43]. Zou et al. [41], reported a reduction in the activities of ammonia oxidizers and nitrite oxidizers at an influent salinity range of 9.1–13.2 g/L in reactors inoculated with activated sludge without salt-tolerant inoculum. However, sludge adaptation enabled the MBR system to experience good nitrification performance without deterioration, and the system achieved a nitrification rate of up to 1.71 g N/L·d at 21.3 g/L salinity. Effective nitrification performance was obtained at a high ammonium loading rate (ALR) of up to 3.43 kg NH4+-N/m^3·d, which corresponded to an influent NH4+-N concentration of 2000 mg/L. This outcome demonstrated the anticipated capability of membrane bioreactors (MBRs) for treating wastewater with high ammonium concentrations [43].

Similarly, an MBR system was adapted to treat influent wastewater with salinity concentrations up to 35 g/L [44]. Halophilic bacteria play a significant role in the treatment of hypersaline wastewater [26]. Ringleben et al. [37] investigated the specific growth rate of *Bacillus spizizenii* within a salinity range of 4.7–54.7 g/L, and the results indicated non-inhibition effects within a salinity range of 4.7–24.7 g/L; above 24.7 g/L, and more specifically, at 34.7 g/L, growth was negatively affected. The excessive elevation of salinity to 54.7 g/L extremely inhibited the specific growth rate and increased the lag phase times [37]. Shokri et al. [45] reported the successful operation of halophilic MBRs for the treatment of hypersaline wastewater using 50 g/L without negative impacts; however, a decline in COD removal was observed after short-term extremely high salinity in 130 and 200 g/L shock loads. An SBR-MBR process was utilized for the successful treatment of petrochemical industrial effluent using an initial concentration of 2250 mg COD/L and 35 g/L salinity [46]. The system achieved 97.2% COD removal at 1.124 g COD/L·d OLR [46]. Results of an anoxic–oxic reactor inoculated with marine sediment that underwent 75 and 48 h of HRT in an anoxic and oxic compartment for urea and COD removal from high-salinity laboratory-prepared wastewater (20–60 g/L salinity concentration) indicated that oxic microbial communities rapidly and more effectively adapted to a 20 g/L salinity level within 10 weeks; the removal efficiency for urea was 93% compared to 18 weeks in the case of anoxic compartment with a 56% removal efficiency [47]. As salinity elevated from 30 to 60 g NaCl/L, removal of urea steeply increased from 44 to 90%. The oxic microbial community reached over 96% urea removal at 30 g/L salinity and continued constant until 60 g/L salinity. Chen et al. [47] reported COD removal efficiency between 63 and 96% for the oxic community and from 51 to 84% for the anoxic community within a salinity range from 20 to 60/L. Thus, oxic micro-organisms are better adapted to salinity; the adaptation period is shorter, and the removal efficiency is better.

Membrane fouling represents one of the limiting factors for the widespread use of MBRs for industrial effluent treatment. It increases the excessive rise in trans-membrane pressure (TMP) and reduces permeate flux. High salinity induces membrane fouling by enhancing excessive production of soluble microbial products (SMPs) and extracellular polymeric substances (EPSs). A significant rise in the TMP was detected with salinity shock load due to a significant release of SMP and EPS [45]. In addition, a significant elevation in the resistance of the cake layer was detected with a consequent rise in the contribution of the cake layers to the whole membrane resistance [45]. Increasing salinity from 0 to 40 g/L was found to reduce the abundance of filamentous bacteria and the single microbial flocs of the activated sludge, enhancing sludge granulation by increasing its compactness and density, which significantly reduces the sludge volume index and improves its settle ability [48]. Similarly, Song et al. [22] reported that inorganic particulates (salt) enhance sludge granulation and improve sludge settling properties by working as nuclei for microbial communities. On the other hand, other publications reported the significant deterioration and disintegration of granular activated sludge by rising salinity levels between 0 and 60 g/L, and the sludge volume index (SVI) increased from 50 mL/g to 97 mL/g [29,42]. Mutlu and his colleagues [14] concluded that salinity above 40 g/L induced the disruption of biological flocs and caused the acceleration of membrane fouling in MBR containing sludge seed from municipal sewage treatment plant.

Most of the published studies on MBRs for treating highly saline wastewater utilized synthetic wastewater and few of them used real saline industrial wastewater from one source. In this study, mixed saline industrial wastewater was treated using MBRs with an emphasis on the impacts of salinity level on the treatment process and membrane fouling. This is the first-ever research study in Saudi Arabia to investigate the real application of MBRs seeded with active biomass from treatment plants receiving saline industrial effluents to treat mixed high-saline wastewater and explore the effects of different salinity levels on process performance.

2. Materials and Methods

2.1. Experimental Setup

This experiment was done at the Al-Hasa wastewater treatment plant using a pilot-scale MBR containing a hollow-fiber membrane module with 1 m^2 effective filtration area and 0.06 µm pore size (ultra-filtration; UF). The module was made of polyvinylidene fluoride by HINADA Water Treatment Tech. Co., Ltd. (North of Junya Road Number 3, Huangpu District, Guangzhou, China). The membrane module was submerged in the second compartment of the two-partition aeration tank with a 240 L effective working volume (Figure 1). The aeration tank has a float valve at the inlet point or influent point, which is connected to a high-level feed tank. The feed tank receives wastewater from a 6 m^3 influent storage tank via a submerged pump, which continuously works to recirculate wastewater between the online feed tank and the influent storage tank. The MBR is connected to a suction pump or vacuum pump that drives permeate from the MBR (permeate flow out/in direction) to the treated effluent or permeate storage tank. A pressure measuring device was fixed just before the permeate vacuum pump for daily monitoring of TMP. The influent storage tank was equipped with a mechanical stirrer to keep wastewater homogenously mixed and prevent settlement and anoxic conditions in the tank. The influent wastewater gravitationally flows from the feed tank to the aeration tank under the effect of the permeate suction pump. Additionally, the membrane was linked to a backwash pump that periodically enforces clean water (30 $L/m^2 \cdot h$) from the backwash tank to the membrane for cleaning membrane pore blocking. Additionally, the membrane surface was air-purged at a rate of 0.6 m^3/min. Two air blowers (on and standby) were installed to continuously supply airflow to the reactor. Sludge was recirculated from the MBR compartment back to the aeration compartment using a sludge recirculation pump.

2.2. System Start-Up and Operations

Mixed saline industrial effluent was collected from the effluent of dissolved air flotation (DAF) at a full-scale industrial wastewater treatment facility in Al-Hasa, Saudi Arabia. The initial COD of the mixed saline industrial wastewater was regulated to be 1300 mg/L either via enrichment with glucose or dilution with clean potable water provided by the industrial city, if needed. After COD adjustment, NH_4Cl and NaH_2PO_4 were added to adjust the COD:N:P to 250:5:1 [49,50], if required; then, the influent in the storage tank was adjusted to the required salinity level (5, 10, 20, and 25 g/L) by adding NaCl (sodium chloride). Before feeding raw wastewater into the system, active biomass from the Al-Hasa saline industrial sewage treatment facility was inoculated to the aeration tank at a 4 g mixed liquor-suspended solid MLSS/L to control membrane fouling during the initial operation, since flocs of the activated sludge adsorb colloidal particles of the influent wastewater and prevent pore clogging in the membrane [51,52]. The system was fed pre-adjusted mixed saline industrial wastewater with 5 g/L of total dissolved solids (TDSs) concentration (run 1). The permeate pump was adjusted daily to drive permeate at 10 L/h, which provides a flux rate of 10 L/m^2·h; this matches the recommended flux rate (10–25 L/m^2·h) of the MBR producer and provides 24 h total of HRT in the system. After a steady state condition, the full monitoring of the treatment system was carried out for 2 weeks. The salinity level (run 2, 10 g/L; run 3, 20 g/L; and run 4, 25 g/L) was elevated every 15–20 days with full monitoring of the system to evaluate the impacts of salinity level. The SRT was kept constant at 12 days during the treatment runs during the whole experimental period by wasting excess sludge from the MBR tank.

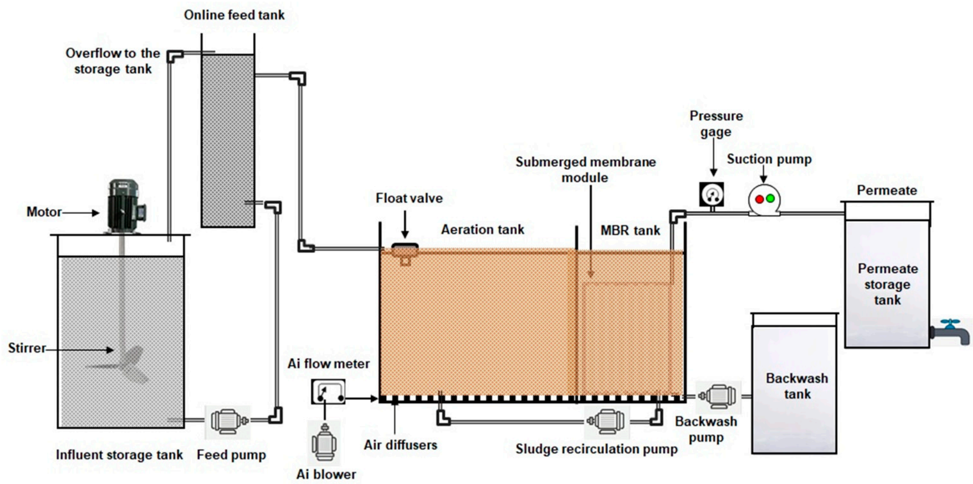

Figure 1. Setup of the MBR system.

2.3. Evaluation Parameters and Analytical Techniques

Full characterization of the influent feed before and after being supplemented with glucose, nutrients, and salt was carried out according to [53]. Permeate from the treatment system was analyzed daily for selective parameters and bi-weekly for other parameters. The analytical parameters include pH (pH meter), electrical conductivity (EC) (HACH MP-6, Colorado, USA), salinity (HACH Salinity meter, Colorado, USA), COD (APHA 5220 B), BOD (APHA 5210 B), total suspended solids (TSS) (APHA 2540 B), turbidity (Hach Turbidity Meter, Colorado, USA), TDS (APHA 2540 C), total Kjeldahl nitrogen (TKN) (APHA 4500-N), ammonia (APHA 4500-NH3), total phosphate (TP) (APHA 4500 PB), and total coliform (APHA 9221 B). All the analyses were carried out following the standard methods (APHA, 2021) {Formatting Citation}. In addition, NO_2 and NO_3 were analyzed in the treated effluent using a HACH Spectrophotometer DR 3900, Colorado, USA. Activated sludge samples were collected from the two compartments and laboratory analyzed for the volume of settled sludge after 1 h and measuring concentration of MLSS and MLVSS (APHA 2540 D, APHA 2540 E) and the subsequent calculation of the sludge volume index (SVI). TMP was measured 24 h a day using the pressure gauge. The flux rate was hourly measured using a graduated cylinder and stopwatch with readjustment to 10 L/m^2·h. Average values of TMP and the flux rate for each day were calculated. Samples from the membrane before operation and after fouling were subjected to a scanning electron microscope (SEM) following the procedure described by [54].

2.4. Statistics

Data are presented as average ± standard deviations. To check the significance of the salinity level on treatment performance, the results of each treatment parameter at different salinity levels were statistically analyzed using a one-way analysis of variance (one-way ANOVA). Similarly, data on TMP and permeate flux rate were statistically analyzed using one-way ANOVA. A probability level of 95% (p = 0.05) was selected to indicate significant and non-significant differences between the results.

3. Results and Discussion

3.1. Quality of Influent Wastewater

The analysis data of the mixed saline industrial effluent before and after enrichment with glucose (COD source), nutrients, and NaCl is depicted in Table 1. The data indicate a low level of COD concentration, which could be defined as low strength, according to [55]. This could be attributed to the positive impact of DAF in reducing the COD concentration for most particulate COD, since a good correlation (73.4%) between TSS and turbidity indicates the colloidal nature of the TSS [52]. Similarly, the BOD concentration was low and represented 32.5% of the COD; this is acceptable for mixed industrial wastewater with significant amounts of petrochemical, textile pharmaceutical, rubber, and plastic industrial wastewater [56]. The correlation between COD and BOD is negative, which means a direct correlation in some samples and an indirect correlation in others, and this is mostly attributed to the daily quantitative and qualitative variations of mixed industrial wastewater. A good correlation between ammonia and TKN was detected at 66%. This is normal since ammonia is part of TKN; however, it is not always like this because the nature of industrial activities, sources of wastewater, and weather conditions, may affect this correlation. The most interesting aspect in the characterization of influent wastewater is the significant concentration of nitrite and nitrate during this period of the study. This could be attributed to the possible high concentration of nitrate in the industrial effluent and denitrification with an accompanying low dissolved oxygen concentration in the sewer pipes, manholes, or storage tanks. Takahashi et al. [57] reported a significant accumulation of nitrite due to denitrification at low oxic conditions. In this study, TDS concentration varied between 5430 and 7980 mg/L in the influet before enrichment.

Table 1. Salinity levels before and after enrichment with NaCl.

	Unit	Salinity 5 g/L		Salinity 10 g/L		Salinity 20 g/L		Salinity 25 g/L	
Parameters		Before	After	Before	After	Before	After	Before	After
Temperature	°C	25.8	26.8	23.1	22.5	23.3	23.5	27.3	28.9
pH	-	7.6	8	7	6.9	7.2	7.4	7.7	7.5
Turbidity	NTU	95	145	126	175	111	139	121	165
TDS	mg/L	5430	6000	6050	11,090	7980	20,850	6900	25,450
COD	mgO_2/L	400	1380	375	1320	316	1399	324	1325
TSS	mg/L	73.7	122	84	110	71	112	78	135
Nitrate	mgN/L	15.35	14.92	12.67	10.32	11.31	8.96	9.51	7.72
Nitrite	mgN/L	15.93	16.66	15.13	15.63	15.83	16.32	16.43	17.35
BOD	mgO_2/L	117	744	112	717	120	810	112	752
EC	mS/cm	5.64	6.22	6.3	12.6	8.73	24.5	7.41	31.2
Ammonia	mgN/L	9.5	22.2	7.8	22.4	8.7	26.3	9.8	22.9
TKN	mgN/L	14.9	27.6	14.7	29.1	15.6	33.1	17.1	30.1
TN	mgN/L	46.18	59.18	42.5	55.05	42.74	58.38	43.04	55.17
TP	Mg P/L	4.20	5.70	2.50	5.60	4.60	5.80	3.90	5.60
Salinity	mg/L	4500	5020	5040	10,080	6890	19,680	5880	24,960
Total coliform	cfu/100ml	1.4×10^5	1.5×10^5	4.3×10^4	4.5×10^4	4.03×10^4	4.5×10^4	4.5×10^4	4.8×10^4

Notes: Before—analysis results before adding glucose, ammonium chloride, sodium monohydrogen phosphate and NaCl; after—analysis results after adding NaCl.

3.2. Organic Matter Removal and Effects of Salinity Level

The ability of the MBR treatment to remove polluting parameters is presented in Table 2 as % removals. The data show the potential removal of COD and BOD, with % removal ranges of 95.3–97.2% and 98.3–98.8% for COD and BOD. Except in advanced oxidation and related technologies used for the treatment of hardly biodegradable organic matter, other treatment technologies, especially biological treatment, always provide a higher % removal for BOD compared to COD. Micro-organisms in the biological systems degrade and consume the BOD fraction of COD, and the non-biodegradable fraction plus residual BOD constitute the residual COD. The % removal of COD is >78% as reported by [21], during the treatment of tannery effluent with 14.8 g/L salinity at 1.3 kg COD/L·d in MBR. The lower % removal of COD reported by Vo et al. [21] was attributed to a very low concentration of MLVSS since most of the MLSS originated from the TSS of the influent wastewater, which is mostly composed of non-biodegradable matter. Ferrer-Polonio et al. [49] reported only a 80.3% removal of COD during the treatment of hypersaline pickling wastewater at a low F/M ratio (0.133 kg COD/kg MLVSS·d), which could be attributed to higher salinity compared to the current (5–25 g/L) range. On the other hand, Alighardashi et al. [38] reported higher efficiency, with 98% COD removal after treatment of tannery effluent in MBRs at an OLR of 5 g COD/L·d; however, the source of MLSS was municipal-activated sludge without adaptation, and this is questionable. During treatment of synthetic sewage with 50 g/L salinity and 1500 mg/L influent COD, the halophilic MBR working at 12 h HRT and 3 g OLR was able to reduce COD by 91.1% [45], which is less than the current study, and this is mostly attributed to the low salinity level (5–25 g/L) in the current study. Reported results of MBRs treating saline organic wastewater revealed a tremendous decline in treatment performance, with a decrease in the removal efficiency of DOC from 77 to 10% after increasing the salt concentration from 0 to 30 g/L [44]. The better results of the current MBR system compared to Johir et al. [44] are attributed to the source of the MLSS. The sludge inoculum of this study was collected from a treatment plant (Al-Hasa) that receives saline industrial effluent; this sludge could be considered salt-tolerant compared to the utilization of municipal-activated sludge seed in the other study.

Table 2. Percentage removal of the treated parameters at different salinity levels.

Parameter	Salinity 5 g/L	Salinity 10 g/L	Salinity 20 g/L	Salinity 25 g/L
COD	95.4 ± 0.4 ^{a}	96.6 ± 0.7 ^{a}	95.3 ± 0.2 ^{a}	97.2 ± 0.7 ^{a}
BOD	98.6 ± 0.9 ^{a}	98.8 ± 0.2 ^{a}	98.3 ± 0.1 ^{a}	98.8 ± 0.5 ^{a}
Ammonia	95.7 ± 3.8 ^{ab}	93.2 ± 0.45 ^{b}	96.7 ± 1.3 ^{a}	95.4 ± 0.9 ^{a}
TKN	92.3 ± 0.6 ^{a}	93.1 ± 0.7 ^{ab}	93.3 ± 0.2 ^{b}	95.1 ± 1.7 ^{ab}
TN	81.6 ± 4.8 ^{a}	81.6 ± 9.6 ^{ab}	85.1 ± 0.9 ^{a}	92.5 ± 1.1 ^{b}
TP	87.7 ± 5.0 ^{a}	95.8 ± 2.7 ^{b}	84.1 ± 13 ^{c}	76.8 ± 8.2 ^{d}
Turbidity	99.6 ± 0.2 ^{a}	98.3 ± 0.5 ^{bc}	97.8 ± 0.8 ^{c}	98.3 ± 0.3 ^{b}
TSS	98.5 ± 0.7 ^{a}	97.2 ± 1.4 ^{b}	95.8 ± 1.0 ^{c}	97.9 ± 0.4 ^{a}

Notes: Analysis of variance (ANOVA): single row values with similar superscript letters are non-significantly different ($p < 0.05$).

Song et al. [25] reported possible application of conventional bioreactors for the effective remediation of saline industrial effluents via sludge adaptation and sludge enrichment with salt-tolerant microbial consortia. Thus, an activated sludge unit inoculated with municipal activated sludge and working under 1 d of HRT and 10 d of SRT was adapted to lower the COD of fish processing wastewater with 25 g/L salinity from 3400 mg/L to 490 mg/L with a corresponding range of 570–1710 mg/L·d for the removal load [58]. This value is better than the results of this study, which provides a daily volumetric COD removal load of around 1.33 g COD/L·d, and this is attributed to the better biodegradability of fish processing wastewater compared to the mixed industrial wastewater of this study, which contains petrochemical, textile, pharmaceutical, and other hardly biodegradable wastewater [56].

An MBR system inoculated with seed sludge from an MBR system receiving produced wastewater from an oil and gas field was able to achieve 70% average COD removal from an average of three phases (1880 mg/L, 860 mg/L, and 390 mg/L) of concentration at a gradual salinity increase of 10–40 ms/cm; 8–32 g/L [14]. This is extremely lower than the results of this study due to different sources of influent since produced wastewater mostly contains refractory organic pollutants that are hard to degrade and need more HRT; the mixed industrial wastewater enriched with glucose in the current study has more biodegradability. Moreover, an SBR was utilized to treat tannery sewage with 35 g/L salt content, and the system successfully achieved 95% COD removal from a 3000 mg/L influent concentration at 5 d HRT (0.6 g COD/L·d) after sludge adaptation [18]. This is comparable to the current data despite the different nature of the wastewater. Gradual elevation of salinity (0, 5, 10, and 20 g/L) did not decline organic carbon removal (maintained above 95%) of an MBR inoculated with municipal activated sludge [32]. During the remediation of synthetic sewage with 50 g/L salinity and 1500 mg/L initial COD, a halophilic MBR process employed HRT of 12 h and an OLR of 0.75 g and was able to reduce COD by 91.1% [45], which is less than the reported value of the current research and this is due to the high salinity applied by Shokri and his colleagues [45]. The carrier MBR system hosting halophilic bacteria effectively treated saline pharmaceutical effluent and declined influent COD by >75% [25], at 30 h HRT. Similarly, moving bed MBR inoculated with halophilic bacteria effectively treated disinfectant-contaminated saline wastewater [59]. Song et al. [22] achieved 89.2% COD removal during the treatment of saline (TDS 26.2 g/L) antibiotic wastewater using an MBR at an OLR of 5.5 g COD/L·d, which is tremendously better than the current results, and this is due to system inoculation with halophilic sludge.

Statistically, no significant difference for COD and BOD % removal during the treatment runs of this study; however, residual values left in the final treated effluent show significantly low residual COD at 10 and 25 g/L salinity (Table 3). This significantly low residual COD and BOD is not attributed to higher microbial activity but mostly to a low F/M ratio at 10 and 25 g/L salinity levels (Figure 2). The average F/M ratios were 0.351, 0.322, 0.337, and 0.307 g COD/g MLSS·d and 0.252, 0.249, 0.276, and 0.239 g BOD/g MLVSS·dfor the treatment process from run 1 to run 4, respectively. The low F/M ratio at a 10 and 25 g/L salinity concentration is due to the low influent COD at 10 and 25 g/L and low permeate flux at run 4 (Section 3.4), and both low influent COD and low flux decline the OLR and F/M ratio. The low flux at 25 g/L (run 4) is attributed to membrane fouling, which additionally acts as a secondary filter that improves permeate quality [52]. This explanation is confirmed by the data of the specific COD and specific BOD removal loads (Figure 3), which provide average specific COD removal loads of 0.334, 0.311, 0.321, and 0.299 g COD/g TSS, with significantly low values during run 4, run 2, and run 3, consequently. The volumetric COD removal loads were 1.32, 1.22, 1.27, and 1.18 g COD/LL·d during treatment runs from 1 to 4, respectively (Figure 4). The improvement in COD removal loads from run 2 to run 3 could be attributed to sludge adaptation or change, such as with industrial wastewater. By increasing salinity from an initial concentration of 0.5 g/L to 5 g/L, the SBR system potentially increased the removal of COD from 90 to 99%, and this is due to the adaptation of the microbial population to the gradual rise in salinity during long-term operation [29]. According to Aloui et al. [58], salinity inhibition is significant above 40 g/L NaCl, and this revealed the possible acclimatization of the bacterial consortium to the effective treatment of saline industrial wastewater up to 40 g/L salinity and 0.86 g COD/L·d OLR. All reactors working below a 40 g/L salinity level were able to attain COD removal above 97% at an OLR of 0.86 g COD/L·d. Results of TSS and turbidity are presented in Table 2 and indicate potential removal of TSS and turbidity, with an average value exceeding 97% for TSS and 95% for turbidity. The residual values of TSS (Table 3) have a range of 1.8–4.83 mg/L, with a lower value at 5 g/L salinity. However, there were significant differences between the values during the treatment runs, and there was no clear relation with salinity level. Moreover, residual turbidity at 5 g/L was significantly low, whereas, at 10, 20, and 25 g/L, the value at 10 g/L had no significant difference with the value at 20 and 25 g/L, with the lowest value at 25 g/L indicating the absence of a clear relationship between turbidity and salinity levels at 10–25 g/L. In an MBR system, the residual concentration of TSS and turbidity mostly depend on the physical characteristics of the membrane, such as pore size and the type of membrane module. Some studies reported the absence of any relationship between the operating conditions and performance of the MBR in removing TSS and turbidity [52,56]. Shokri et al. [45] reported turbidity of 2.9 ± 1.5 NTU in the permeate of MBRs during 49 days of operation, even during and after saline shock loads, which shows the nonsignificant impacts of salinity on effluent turbidity. The formation of internal biofilm with intermittent sloughing could be the reason for high turbidity in the effluent of the MBR, and this mostly depends on the HRT of the system.

Table 3. Effluent quality at different salinity levels.

Item	Salinity 5 g/L	Salinity 10 g/L	Salinity 20 g/L	Salinity 25 g/L
COD	63.3 ± 5.9^{a}	45.0 ± 8.3^{b}	66.2 ± 2.3^{a}	37.3 ± 8.8^{c}
BOD	13.8 ± 1.2^{a}	8.6 ± 1.2^{b}	13.6 ± 0.4^{a}	8.9 ± 3.5^{ab}
Ammonia-N	1.45 ± 0.35^{ab}	1.53 ± 0.10^{b}	0.87 ± 0.35^{b}	1.05 ± 0.21^{b}
NO_3-N	8.3 ± 3.5^{a}	7.2 ± 4.62^{a}	6.69 ± 0.73^{a}	2.8 ± 0.7^{b}
TKN	2.13 ± 0.15^{a}	2.00 ± 0.20^{ab}	2.17 ± 0.06^{b}	1.49 ± 0.52^{ab}
TN	10.90 ± 2.86^{a}	10.15 ± 5.27^{ab}	8.67 ± 0.55^{a}	4.15 ± 0.63^{b}
TP	0.70 ± 0.28^{a}	0.23 ± 0.15^{b}	0.92 ± 0.76^{ac}	1.3 ± 0.46^{c}
TSS	1.82 ± 0.79^{a}	3.05 ± 1.49^{b}	4.81 ± 1.17^{c}	2.71 ± 0.55^{b}
Turbidity	0.54 ± 0.29^{a}	2.99 ± 0.89^{bc}	3.05 ± 1.09^{b}	2.73 ± 0.53^{c}

Notes: Analysis of variance (ANOVA): Single row values with similar superscript letters are non-significantly different ($p < 0.05$).

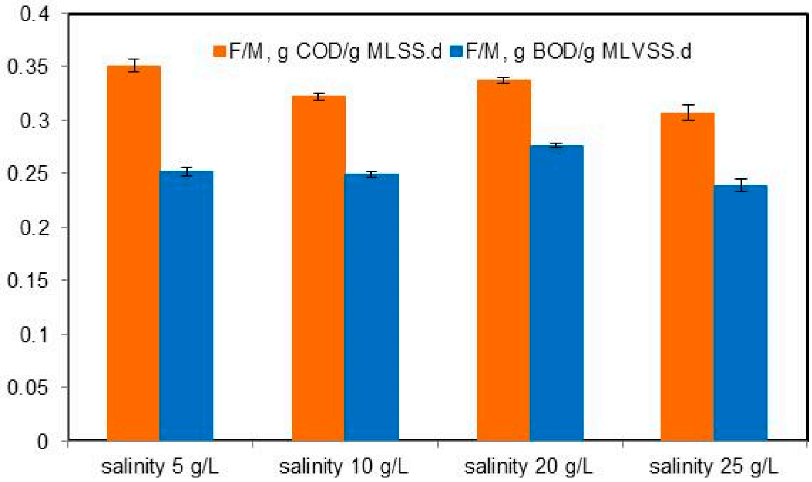

Figure 2. F/M ratios at different salinity levels.

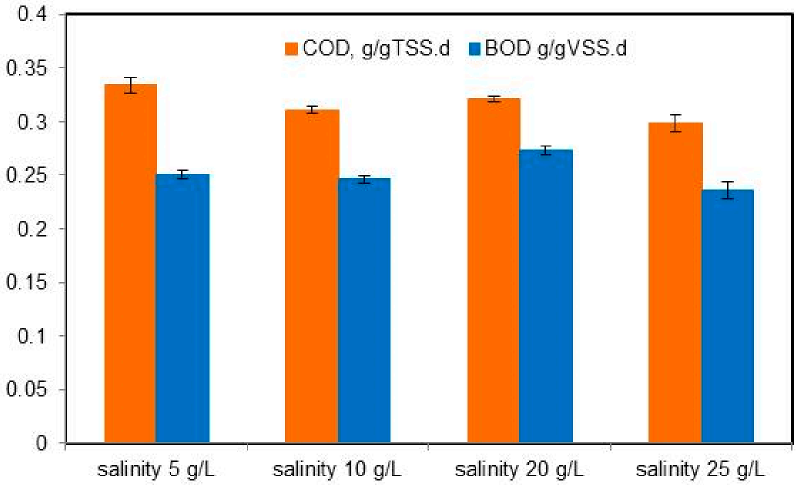

Figure 3. Specific COD and BOD removal loads at different salinity levels.

3.3. Nutrient Removal and the Impact of Salinity Level

The results presented in Table 2 show average ammonia % removal between 93.2 and 96.7%, with a lower value at 10 g/L salinity; the values at 20 and 25 g/L salinity were non-significantly different from values at 5 g/L. The residual values (Table 3) were 1.45, 1.53, 0.87, and 1.05 mgN/L in the treatment runs from 1 to 4. This indicates no significant effects of salinity increase on ammonia removal since the initial ammonia concentrations were close to each other. The presence of high NO_3 and NO_2 concentrations in the influent might be the reason for enhanced ammonia and TN removal, especially in the presence of salt-tolerant sludge. Lai et al. [60] achieved 90.1 and 83.5% for ammonia and TN removal by using marine anammox bacteria to treat saline wastewater with 30 g/L salinity. Similarly, Yan et al. [61] were able to treat hypersaline, nitrogen-rich wastewater with a 30 g/L salinity level. Additionally, a novel salinity-tolerant species of Vibrio sp. (LV-Q1) was used exceptionally to achieve 98 and 100% removal efficiency for ammonia and nitrate at 50 g/L salinity [62]. An SBR containing aerobic halophilic granular sludge effectively reduced COD and ammonia by 91% and 72.6%, respectively, from hypersaline wastewater (30 g/L) at an OLR of 0.36 g COD/L·d [63]; however, ammonia nitrification ended at nitrite, which exceeded 30 mgN/L, while the nitrate was always below 3 mgN/L. Microbiologically, Moussa et al. [48] detected only *Nitrosomonas europaea* and *Nitrosomonas mobiles* as ammonia oxidizers at a salinity level of between 10 and 30 g/L, and none of the nitrite oxidizers were detected. In the current study, no nitrite accumulation and very few traces were detected in the effluent, and this could be attributed to the presence of salt-tolerant sludge. An et al. [64] reported a possible adaptation of denitrifying sludge from a normal sewage oxidation ditch to a salinity level above 25 g/L and provided good nitrogen removal; however, salinity above 35 g/L deteriorates the denitrification process and nitrogen removal, and the system failed to adapt to this high concentration. Marine anammox inoculum was used for nitrogen removal from nitrogen-rich hypersaline wastewater and the results indicated a good removal efficiency, exceeding 90% at 35 g/L salinity [65]. A biofilm MBR inoculated sludge biomass from a conventional human sewage treatment facility was applied to treat hypersaline (20-30 g/L) wastewater [66]. The system achieved 96.8, 91.2, and 13.7% ammonia removal at three different OLRs (0.5, 1.0, and 1.5 g COD/L·d, respectively) from an initial concentration of 133.6–142.9 mgN/L which correspond to removal loads of 66.6, 130.3, and 23.6 mgN/L·d. The lowest removal load reported by Yang et al. [66] is higher than the current results; this indicates the dependence of the treatment efficiency on the composition of wastewater and treatment conditions.

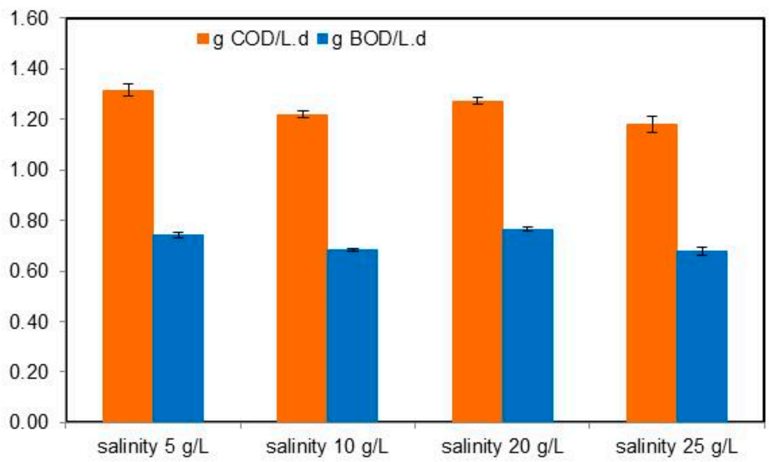

Figure 4. Volumetric COD and BOD removal loads for different salinity levels.

During the operation of a four-step SBR for the treatment of synthetic influent with initial total N and total P concentration of 60 mgN/L and 18 mg P/L at variable salinity levels, the system achieved estimated ammonia removal loads of 9.0, 8.5, 7.5, and 7.0 mg/L·h at a 5, 10, 20, and 25 g/L salinity concentration, respectively [42]. These values are extremely higher than the results of this study, which provided ammonia removal loads of 20.8, 19.2, 25.4, and 21.9 mg N/L·d. Moreover, the gradual increase of salinity (0, 5, 10, and 20 g/L) in an MBR increased the removal load of ammonia from 3.1 mgN/L·h to 4.32 mgN/L·h [32] after sludge adaptation. The lower ammonia removal loads of the present study are attributed to low influent ammonia concentration. P removal was recorded to be 87.7, 95.8, 84.1, and 76.8% at 5, 10, 20, and 25 g/L salinity concentrations, and the average residual values are 0.7, 0.23, 0.92, and 1.3 mg P/L. These results show the highest % removal occurred at 10 g/L salinity and then significantly reduced at 20 g/L and 25 g/L. The high % removal of P, especially at 5 and 10 g/L salinity, is attributed to low initial concentration since the P removal loads were 5.0, 5.37, 4.88, and 4.3 mg P/L·d. These removal loads are lower than the values reported elsewhere. Uygur and Kargi [42] reported that P concentration in the effluent of an MBR decreased from 18 mg/L to 8.3 mg/L at 60 g/L salinity, which means a P removal load of 23.3 mg P/L·d, which is nearly four-fold the values in the current study. It is well-known that conventional activated sludge systems, including activated sludge MBR, cannot remove P, and only modified systems designed for biological phosphorous removal have this ability. Zahid and El-Shafai [52] reported a P removal load of around 13.6 mg P/L·d on average during utilization of MBRs for the treatment of municipal sewage. The low P removal load at 25 g/L salinity may be attributed to the negative effects of salt on biological activity. A high salt concentration may force micro-organisms to move out of excess salt; P. Uygur and Kargi [42] reported that salt inhibition was more distinct in the removal of P when compared to COD and ammonia due to the sensitivity of P-accumulating micro-organisms to salinity; this adverse effect was severe at a salinity concentration above 40 g/L.

3.4. Permeate Flux and TMP During the Treatment Runs

Results of continuous monitoring of the permeate flux and TMP are presented in Figure 5, which shows an average permeate flux of 9.99 ± 0.17, 9.54 ± 0.11, 9.55 ± 0.08, and 9.17 ± 0.21 L/m^2·h, with significant low values at 10, 20, and 25 g/L salinity concentrations, and the lowest value at 25 g/L. There is no significant difference between 10 and 20 g/L salinity. The measurements of TMP are 0.084 ± 0.016, 0.042 ± 0.008, 0.044 ± 0.008, and 0.103 ± 0.029 bar, with significantly lower values at 10 and 20 g/L. The match between permeate flux and TMP indicates a non-significant effect of salinity on membrane fouling and flux at 5, 10, and 20 g/L salinity, whereas, at 25 g/L, there was a significant effect with a reduction in the flux and an increase in TMP, which is mostly attributed to membrane fouling. SEM of the new membrane and after exposure to 25 g/L salinity (end of the experiment) is presented in Figure 6. Most of the foulants are fine particles which can be observed with the smooth surface of the cake or fouling layers with little coarse precipitates.

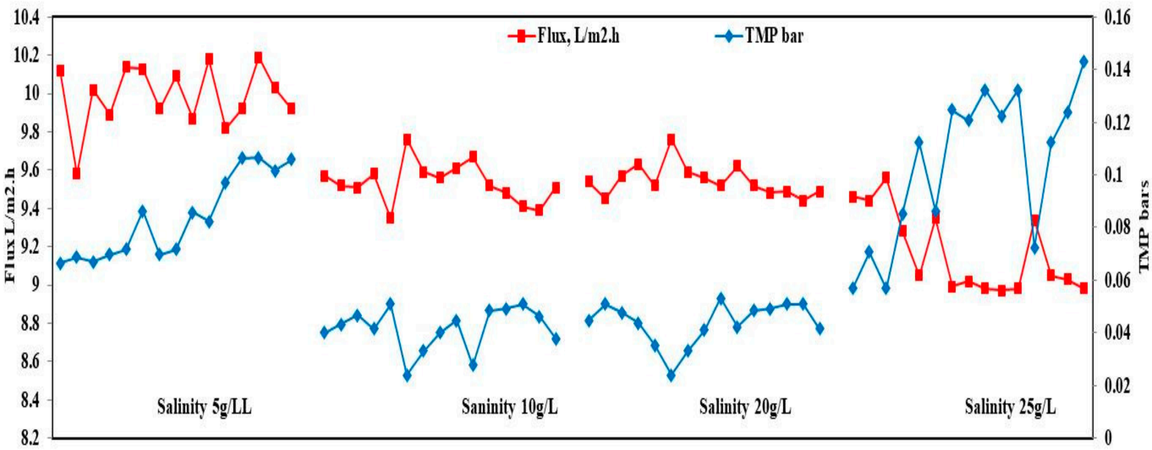

Figure 5. Permeate flux and TMP during the treatment runs.

Membrane fouling at 25 g/L could be attributed to the direct salinity effects on the physical properties of the MLSS, including wastewater and sludge flocs. Increasing salinity to 10, 15, and 50 g/L could increase the density of the MLSS by 0.7, 1.1, and 3.4%, respectively [42]. This higher density increases the buoyancy characteristics of the MLSS, which makes biological flocs weaker and ready to create tiny flocs [18,30]. These tiny flocs could adhere to the membrane surface and block the membrane pores, thus enhancing both cake fouling and internal fouling of the membrane. Xie et al. [67] reported an extreme reduction in sludge particle size, with small particles deposited in the cake and on the membrane surface with a subsequent increase in filtration resistance. When compared to small particles, larger particles enlarge the void ratio in the sludge cake with less filtration resistance. Moreover, the data of the specific removal load (g COD/g MLSS·d) and volumetric removal load (g COD/L·d) indicated significantly lower activity at 25 g/L salinity, and this may be accompanied by the high release of SMP and EPS. Jang et al. [32] reported a tremendous acceleration of membrane fouling from pore blocking at high salt concentrations (0, 5, 10, and 20 g/L) due to a substantial rise in EPS. The author added that the contribution of fouling resistance due to solutes adsorption and pore blocking increased from 7 to 22% due to salt increase; cake resistance decreased along with salt increase due to biomass detachment. Additionally, Xie et al. [67] reported an increase in SMP and EPS by salinity increase from 0 to 35 g/L, and the increase was higher in EPS with a significantly higher contribution of protein than carbohydrate. The results of a fixed-bed biofilm MBR operated to achieve COD removal and denitrification at 0–30 g/L salinity range indicated a considerable acceleration of membrane fouling beyond 1% salinity [64]. Finally, the decline in permeate flux and rise in TMP at 25 g/L salinity may be due to the direct effects of salinity on water density and physical characteristics of MLSS in addition to physiological and biochemical changes of the MLSS with a substantial increase in EPS.

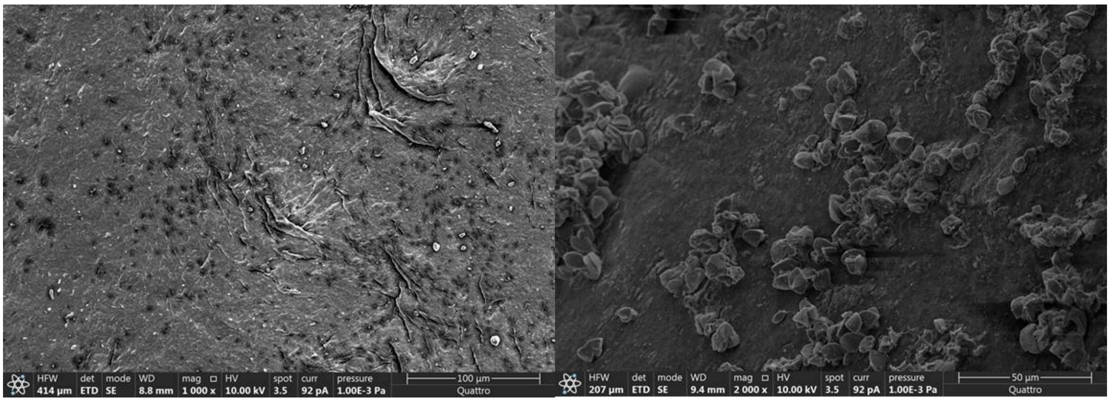

Figure 6. Shows SEM of the new membrane (**left**) and membrane after fouling at 25 g/L salinity (**right**).

4. Conclusions

The characteristics of permeate flow from MBRs inoculated with high salinity-adapted sludge indicated potential application of the system for the treatment of mixed saline industrial effluents. The operation of MBRs inoculated with high-salinity-adapted sludge at OLR 1.33 g COD/L·d and salinity up to 25 g/L is feasible and provides good quality effluent that meets Saudi discharge standards. However, variation in the characteristics of mixed saline industrial effluents requires a flexible design of the MBR process to match high salinity and high loading rates required to keep the F/M ratio within $\leq$ 0.31 at a salinity concentration of 25 g/L. A salinity of 25g/L reduced removal efficiency; however, keeping the F/M ratio $\leq$ 0.31 enables the system to produce an effluent quality that complies with Saudi disposal limits. A salinity of 25 g/L enhances membrane fouling, with an observable rise in TMP and a decline in permeate flux; however, the fouling at this salinity range is controllable. More research is required to explore the possible application of MBRs inoculated with high-salinity-adapted sludge for the treatment of mixed saline industrial wastewater at a salinity concentration above 25 g/L.

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