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Nanofiltration of oil field-produced water for reinjection and optimum protection of oil formation

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ABSTRACT

In view of the increasingly stringent norms of disposal of effluent from petroleum activities and interest in water conservation, the treatment of the oilfield-produced water (PW), the largest wastewater (WW) stream associated with oil and gas industry in the Suez Gulf, was investigated for injection in the oil formation to enhance oil production and other environmental-friendly reuse purposes. Long-term laboratory and pilot testing of treatment of PW and Gulf water (GW) by coagulation/filtration and coagulation/filtration/nanofiltration (NF) aimed to avoid blocking of the porous oil formation upon injection and to inhibit the detected corrosion and biomass accumulation on the internal wall of the injection piping, which was shown to re-contaminate the treated injection water. Analysis of PW showed the concentrations of TDS, organics including hydrocarbons, oil droplets, sulfate, silica, Boron, and suspended solids (SS) of 96472.6, 268.2, 120.4, 7087.5, 134.4, 29.3, and 20.2, respectively. The high sulfate content of both PW and GW would explain the observed hardness scale, on the well casing and pipelines. Only trace concentrations of U_{238} as complex carbonates and heavy metals as copper, vanadium, nickel, and lead were detected in GW. The thick biofilm detected inside the injection pipes consists of biomass of 92% water, extracellular polymer substance (EPS) of mainly anaerobic sulfate-reducing bacteria (SRB) of 1.8×10^9 MPN/gm, and iron compounds due to steel corrosion. The dry film includes high concentrations of iron, sulfur, and a remarkably high radioactivity of uranium 238 of 6,740 pCi/gm, heavy metals such as copper, chromium, lead, and vanadium at concentrations much higher than in GW. Results confirmed that SRB enzymatically reduced the trace uranium and the other soluble cations in PW and concentrated them in the biofilm with parallel depolarization of the cathodic-controlled corrosion of steel to produce ferric sulfide and other iron compounds. Coagulation of PW efficiently removed SS, organics including hydrocarbons and oil. Only partial removal of uranium took place, which was too pH dependent. However, since coagulation did not suppress the biofilm formation and the related phenomena of microbial corrosion and accumulation of radioactivity, the release of these components recontaminated the treated PW. On the other hand, the proposed process of "intermittent chlorination/coagulation/NF" of PW efficiently rejected sulfate, uranium, and other metal cations and polished the removal of SS, bacteria, and organics. This process inhibited the formation of scales and biofilm as well as the related undesirable phenomena and, therefore, stopped the recontamination of the PW prior to injection. Only poor

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accordance was observed between the experimentally determined percent rejection of PW components by NF and that computed according to the solution of the extended Nernst–Planck equation.

Keywords: Nanofiltration; Produced Water; Biofouling; Microbial Corrosion; Microbial Deposition of Uranium

1. Introduction

Extensive oil production takes place in the Gulf of Suez from both inshore and offshore operations. Oil field-produced water (PW) is the largest wastewater (WW) stream associated with oil and gas extraction at a rate quite higher than that of the oil. It increases with the oil formation age to attain up to 98% of the product near the end of the formation production life [1]. In the USA, e.g. this stream attains three billion ton per year [2]. Options to handle the PW are mainly disposal or treatment for reinjection in the oil formation for enhancing the productivity, or for other industrial or agricultural reuse purposes [3]. A daily production of 210 million barrels of PW in the world was reported [4]. In addition, WW produced in oil fields through the different stages of crude oil extraction and refining represents an environmental and processing problem to petroleum industry, which requires cost-effective and environment-friendly methods of treatment [5,6]. While disposal to the sea with use of fresh Gulf water (GW) for injection seems to be the most simple and cost-effective approach, the increasingly stringent norms of disposal of effluents from petroleum activities in environmental regulations and the increasing interest in water conservation favored the treatment of PW for reuse. Treatment of PW prior to reinjection was shown to conserve the porous structure of the oil formation and give better performance [7].

The European standards for effluent from onshore petroleum activities require <5 mg/L total hydrocarbons and <10 mg/L suspended solids (SS), which would not be attained by conventional separation methods and drive attention to application of membrane methods for better performance and minimized cost [8]. According to the recommendations of the American Petroleum Institute for the best available technologies for PW management, reverse osmosis (RO) and nanofiltration (NF) treatment have the advantages of effective removal of particles, dispersed and emulsified oil, small footprint, low energy requirements, and high throughput rates. However, they have the disadvantages of membrane fouling by oil, sulphides or bacteria, and the reject stream, which would contain radioactive material [9].

Our previous results of WW treatment and those of other authors [10,11] have shown the progressive development of the membrane methods NF and RO towards higher process efficiency, cost effectiveness, environmental safety, and ease to use, in addition to high permeate quality and reduced sludge, which made these methods competitive alternatives to conventional technologies.

A NF-based procedure is employed in the UK sector of the North Sea for treatment of sea water to be used for dilution of the high-salinity PW so as to avoid the formation of hardness scales upon injection [12]. NF treatment of PW was reported to enable high sulfate rejection before reinjection [13,14]. NF is also applied as pretreatment for sea water prior to desalination by RO. Such a pretreatment removes organics, colloids, hardness components, micro-organisms, and SSs [15]; therefore, it eliminates fouling problems in the downstream RO [16].

The present work is aimed to investigate cost-effective alternatives for treatment of PW from oil fields and WW produced during different stages of oil production in the Suez Gulf for purposes of reinjection in oil formation or other environmental, industrial, or agricultural reuse purposes. In fact, the present Suez Gulf PW treatment by coagulation/filtration was reported to be insufficient and would lead to blocking of the porous structure of the oil formation. Site inspection revealed the formation of a viscous fouling layer on the internal surface of the injection steel piping, which was shown to cause corrosion of steel piping and re-contaminate the treated PW through the release of SSs, organics, iron compounds, and radioactive isotopes. Furthermore, solid scales were detected on the internal well casings, pipelines, and pumps. The present work includes the rehabilitation of the site coagulation system and the introduction of adequate sanitization and a polishing NF treatment.

2. Experimental

2.1. General characteristics of PW

According to the site inspection in the Suez Gulf region, and the performed analysis of the PW, which is mainly a blend of oil formation water and injection water, its composition varies considerably with the nature of the oil extracted, the age, and geology of the formation.

PW contaminants include SS as silt and sand; free oil as suspended oil droplets; dissolved oil; organics including hydrocarbons as benzene, toluene, ethyl benzene Xylene, (BTEX), and polyaromatic hydrocarbons; dissolved gases such as H_2S, and Rn_{222}; traces of heavy metals such as copper, nickel, and chromium; hardness components such as calcium, strontium, barium, and sulfate; and traces of radioisotopes as uranium, U_{238}.

An important aspect of the observed PW composition is the continuous modification due to re-injection of treated PW or GW, reservoir stimulation, introduction of production chemicals, and bacterial activity. Such modifications should be taken into consideration in the design of the treatment process.

Fig. 1 shows a schematic representation of the mobile pilot plant designed so as to enable conduction of NF runs over a wide range of operation conditions of feed pressure, flow rates, and pretreatment steps. Percent recovery ranged from 55 to 70. Both permeate and reject streams were recirculated back to the feed tank, which was thermostated at 25℃. Feed pH was adjusted to the range of 7.5–8.5.

A laboratory experimental system composed of six test cells with circular turbulent agitation at the level of surface of membrane coupons and a test circuit, including a pump, pressure gauge, cartridge filter, flow meter, and thermostated feed tank, was used. Membrane samples were stored dry and thoroughly rinsed with deionized water before use. They were compacted in distilled water at 120 psi, prior to testing until steady flux is obtained, then conditioned by soaking in the testing PW sample for one hour. Tangential cross-flow velocity ranged from 0.005 to 0.1 m/s and feed flux from 4 to 6 GFD.

Thin-film composite NF membranes (HL4040F) of polyamide chemistry (GE/Osmonics) were used

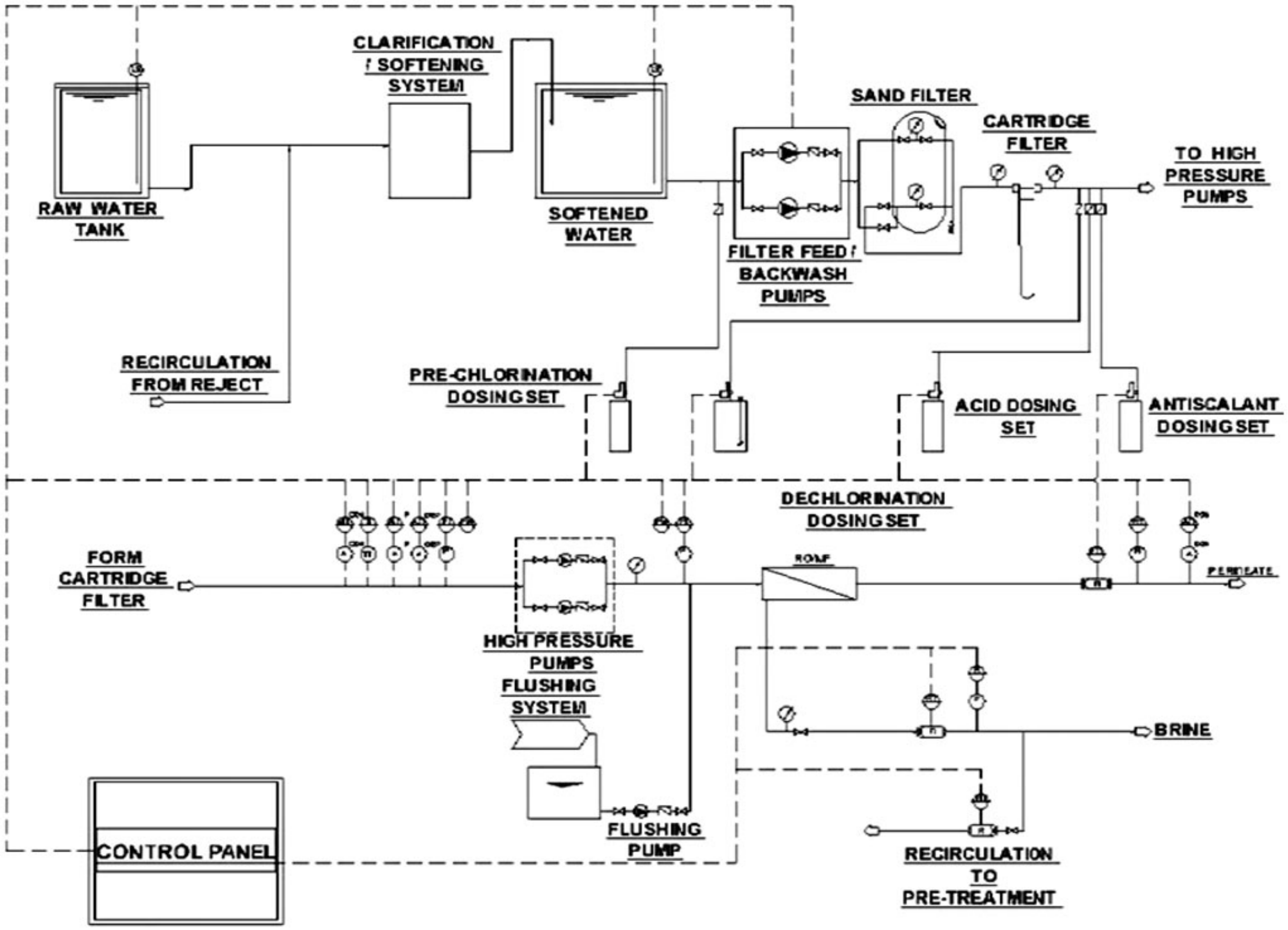

Fig. 1. Schematic flow diagram of the pilot RO/NF unit.

throughout the present study in coupons for cell testing and in 4 inch elements for pilot testing. Rejection of the trace concentrations of radionuclides was calculated using the measured activities according to:

$$\text{Uranium \% rejection} = \frac{(\text{pCi/L})\text{feed} - (\text{pCi/L})\text{permeate}}{(\text{pCi/L})\text{feed}} \times 100 \quad (1)$$

Ionic concentrations were determined by ICP-AES (Perkin–Elmer, Boston, USA). The radioisotopes —UO_2Cl_2 and $Ra(NO_3)_2$—were supplied and analyzed by radio counting or chemical analysis, courtesy from the Regional Middle East Centre of Radioactive Isotopes (Cairo, Egypt). PW was screened at the intake for the removal of coarse suspended matter. The subsequent coagulation/multimedia filtration lowers SS till SDI <10. Table 1 shows a typical analysis of PW.

In order to evaluate the removal of organics and free oil droplets, heavy metal cations and uranium 238 by coagulation jar testing was conducted using either ferric or aluminum coagulants $FeSO_4$, $Fe_2(SO_4)_3$, $FeCl_3$ and alum over the dosing range of 0.5–30 mg/L. Testing was realized as a function of PW, pH of 4, 6, 8, and 10, coagulant type, and dose.

3. Results and discussion

3.1. Coagulation/filtration/NF of PW as compared to only coagulation/filtration

According to Fig. 2, rather efficient removal of organics by coagulation is observed including hydrocarbons and oil, whether dissolved or as suspended droplets. Dosing of 13 mg/L ferric sulfate reduced TOC by 70.5% at a PW salinity of >96,000 ppm. As for the removal of uranium by coagulation, it is shown to be highly pH dependent. Highest removal was 80% measured at pH 10 with a dose of 12 mg/L of $Fe_2(SO_4)_3$. At pH values of 4 and 8, on the other hand, much lower removal was recorded at 15 and 38%, respectively, with up to 17 mg/L of coagulant. This is attributed to the high reactivity of uranium and its affinity to form complex ions with the species available in water at the different pH values. In fact, in the pH range 7–9, the principal complexes of uranium are the negatively charged carbonates, $UO_2(CO_3)(CO_3)_2^{2-}$ and $UO_2(CO)_3^{4-}$ [17] and since the metal hydroxide precipitate of coagulation at the adjusted pH of PW would also be negatively charged, lower uranium removal takes place as the interacting species have similar charges [18]. At pH 4, a similar situation exists. Both the U species, the uranyl cations $(UO)_2^{2+}$, and the metal hydroxide precipitate have the same positive charge. At pH above 10, on the other hand, the positively charged uranium hydroxide $(UO_2)_3$, $(OH)_5^+$, is the predominant complex, which might then coagulate with the negatively charged metal hydroxide to result in higher removal [18]. However, upon additional NF under similar conditions, Fig. 3 shows efficient and steady removal of uranium across the studied pH range.

As for the removal of metal ions, Fig. 4 compares the removal by coagulation to that by coagulation/NF from the high-salinity PW at pH 10 with 15 mg/L ferric chloride. However, lower removals of Cu^{2+} and Pb^{2+} by coagulation of only 47 and 68%, respectively, were realized at lower pH values of 4 and 6 upon dosing of 20 mg/L of $FeCl_3$. Chromium and radium were not at all removed by coagulation.

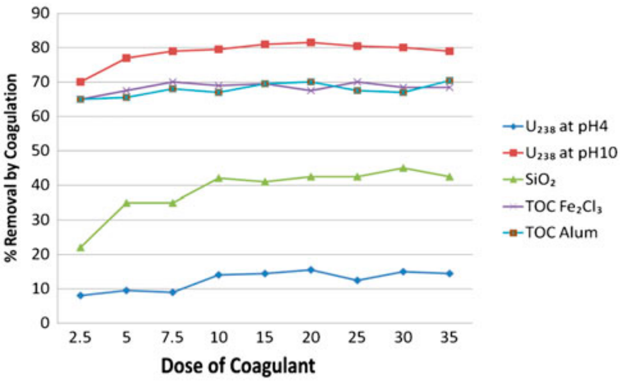

Fig. 2. Variation of the removal of contaminants as function of the coagulant dose.

Table 1
Typical analysis of PW (ppm)

Components	Concentration	Components	Concentration
K	472.86	NO_3	–
Na	30,807.64	Cl	52,190.39
Mg	2,908.47	SO_4	7,087.5
Ca	874.57	Suspended solids	20.2
Sr	9.51	Free oil droplets	120.4
Ba	5.371	Dissolved organics (including hydrocarbon and soluble oil)	268.2
Cu	0.74	SiO_2	134.36
Ni	1.13	pH	8.7
CO_3	530.71	TDS	96,472.6
HCO_3	954.08	Uranium 238	12.5 ppb

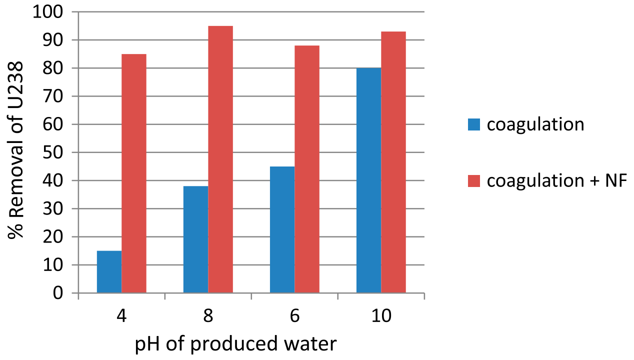

Fig. 3. Removal of U_{238} by only coagulation and by coagulation + NF.

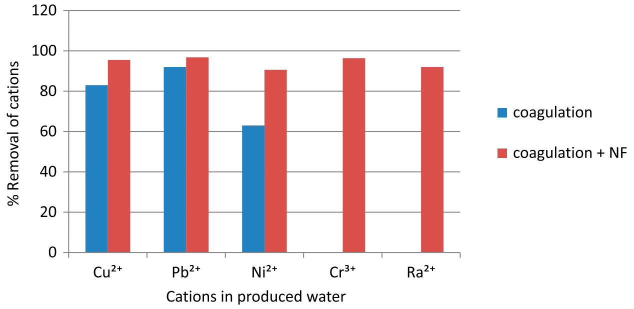

Fig. 4. Removal of HMC's by only coagulation and by coagulation + NF.

These results show that the removal of radioisotopes and other metal ions by conventional coagulation does not have a general trend of behavior. It is too dependent on the testing pH, valency state of ionic contaminant, PW salinity and composition, type and dose of coagulant, and other process details. On the other hand, additional NF showed efficient, predictable, and practically pH-independent removal, Fig. 4. Furthermore, coagulation did not modify the TDS or remove the hardness components SO_4^{2-}, Ca^{2+}, Sr^{2+}, and Ba^{2+}. Consequently, rectified coagulation did not suppress scale deposition. The collected and analyzed scale deposits on the internal well casings and pipelines are attributed to the release of dissolved carbon dioxide as the PW comes to the surface in view of decrease of pressure. The resulting pH increase shifts the equilibrium HCO_3^-/CO_3^{2-} towards decomposition of bicarbonate to form carbonate. Increase of pH and carbonate leads to the deposition of carbonate scales.

On the other hand, the rather high sulfate content in PW is not compatible, upon injection, with the formation of water hardness cations Sr^{2+} and Ba^{2+}, which explains the observed sulfate scales that would extend to damage the porous structure of the oil formation.

Control of scale deposition through dosing of antiscalants in PW is impractical, expensive, labor intensive, particularly for the important PW rates, and environment unfriendly in view of daily production of sludge to be disposed [13,14]. Use of antiscalants would also lead to adverse effects if they are hydrothermally or chemically unstable as the hexametaphosphate, which would hydrolyze to orthophosphate and form insoluble scales with hardness cations [19]. According to our previous results, similar scaling problems were, however, successfully treated by NF of WW. NF was shown to be quite advantageous in comparison with chemical softening and other conventional methods [20].

3.2. *Biofilm formation inside the injection piping, composition, and inhibition*

Analysis of PW conducted near the injection wells revealed the deterioration of the quality of PW, which was already treated by (coagulation + filtration) through increase of contents of SS, iron compounds, organics, and radioisotopes to different levels according to the seasonal temperature variations. This is attributed to the release of these pollutants from the thick fouling film detected on the internal surface of the injection piping. The significant impact of this film on corrosion of steel pipe wall and on purity of PW was confirmed upon scrapping of the film, which is periodically conducted on site, where the treated PW maintained its purity.

In order to inhibit the formation and accumulation of this film and the related negative effects, its composition was investigated in some detail. The gray–black viscous biomass layer of about 1´´ thickness formed after an incubation period of 10–14 d, has the odor of hydrogen sulfide. Analysis revealed a high bacteria content, Table 2, mainly of sulfate reducing bacteria (SRB), their life product, extracellular polymer substance (EPS), including >92% of water, ferric sulfide, and hydrous ferric oxide due to corrosion of carbon steel pipes. The film was vacuum dried, followed by ignition at 350 °C, and then acid digestion for the analysis of inorganic components. Results in Table 3 show a remarkably high percent by weight of iron and

Table 2
Bacterial count of biomass sample

Type of bacteria	Amount
Sulfate reducing bacteria, MPN/g	7.2×10^9
General aerobic bacteria, MPN/g	5.5×10^8

MPN = Most probable number.

Table 3
Chemical and radiological analysis of the dried biofilm

Component	Percent by Wt
Uranium	1.528
Iron	52.820
Sulfur	7.205
Vanadium	0.535
Nickel	0.125
Chromium	0.070
Copper	0.120
Lead	0.275
Total $\alpha + \beta$ activity	5,832 pCi/g

sulfur, which suggests the corrosion of the injection pipe wall according to a mechanism including sulfur compounds.

On the other hand, a strong radioactive contamination of >1.5% by weight of the dry deposit was uranite mineral, which resulted in an $\alpha + \beta$ activity of 5,832 pCi/g. Analysis also showed several heavy metals such as copper, nickel, vanadium, lead, and chromium at much higher concentrations than the trace amounts in GW, as referred to the volume of PW passed during the accumulation of the scrapped deposit.

Composition of the fouling layer is explained by the activity of SRB, which grows particularly in the bottom of the thick biofilm, where anaerobic conditions prevail. In fact, SRB is known to undergo enzymatic reduction of sulfate compounds [21]. Since corrosion of steel in near neutral water is controlled by the cathodic hydrogen evolution [22], sulfate reduction results in cathodic depolarization through consumption of the cathodic hydrogen, which enhances anodic dissolution to ferrous sulfide.

$$SO_4^{2-} + 8H = S^{2-} + 4H_2O \quad \text{(in presence of SRB)} \tag{2}$$

$$Fe^{2+} + S^{2-} = FeS \tag{3}$$

Hydrogen sulfide and hydrous oxides of iron are also formed according to:

$$H_2O = OH^- + H^+ \tag{4}$$

$$2H^+S^{2-} = H_2S \tag{5}$$

$$3Fe^{2+} + 6(OH)^- = 3Fe(OH)_2 \tag{6}$$

Then, the biofilm becomes a source of contamination of PW by release of organic compounds, sulfur, iron compounds, and SS.

In fact, the detected high concentrations of U_{238}, in the form of mineral uranite, UO_2, and the other deposited metals in concentrations quite higher than the trace ones in GW, can hardly be attributed, exclusively, to the possible adsorption or chelation by the biofilm complex organic compounds. However, uranium, chromium, copper, and vanadium cations were reported to be enzymatically reduced by certain micro-organisms under anaerobic conditions [21]. Soluble U(VI) and Cr(VI) deposit the insoluble U(IV) and Cr(III) through specific enzymatic reduction by SRB [23]. U(IV) was reported to deposit as mineral uranite, UO_2, in the biofilm upon reduction by SRB [24–26]. An additional possible source of the detected high radioactivity is the oil-bearing rock formation of limestone and sandstone [27], which would be brought up by the emerging PW.

The fact that microbial activity enabled separation of the mineral radioisotope dispersed in a large volume of water and its concentration up to the observed value, after ignition of this film, is worthy of further consideration as a technology for low-cost separation and concentration of precious minerals present in trace concentrations in sea water. This confirms previous results about microbial removal of toxic components or separation of valuable metals [28,29].

3.3. Removal of PW contaminants by additional NF

PW was treated by NF, after coagulation. Typical results are shown in Fig. 5. Only moderate rejection of both TDS and monovalent species took place, while stronger rejection was observed for the polyvalent contaminants whether anionic or cationic e.g. hardness components, SO_4^{2-}, Ca^{2+}, Sr^{2+}, Ba^{2+}, heavy metal cations and radioisotopes, Ra^{2+}, and UO_2^{2+} in agreement with our previous results of NF rejection [15].

NF permeate showed decontamination till less than the maximum contaminant level, MCL of the US

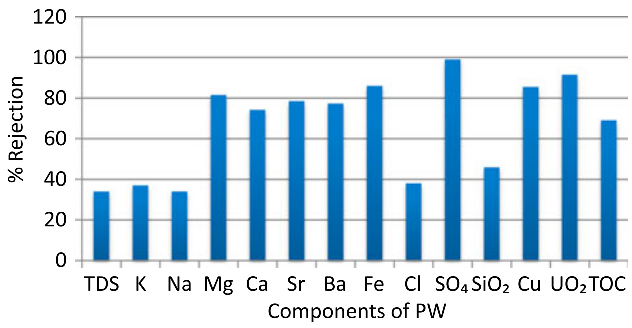

Fig. 5. Percent rejection of components in GW.

Environmental Protection Agency for drinking water or for safe environmental disposal [15].

In view of the extreme conditions encountered in NF of PW, i.e. the very high TDS and the wide variety of nature of the components, it is interesting to verify the applicability of the formulations, which predict and describe the NF behavior of solutes rejection and permeation rate. The extended Nernst–Planck equation governs the steady-state flux J_i of charged species through the charged NF membrane pores [30]:

$$J_i = -D_{i,m}\frac{\delta c_i}{\delta y} + K_{i,c}c_iJ_v - \frac{z_ic_iD_{i,m}F}{RT}\frac{\delta\psi_m}{\delta y} \tag{7}$$

This equation incorporates the combined contributions from diffusion, convection, and the electrical mobility of ions. $D_{i,m}$, being the hindered diffusivity of ions in the pores; c_i, the molar pore concentration of the ionic species with charge z_i; $K_{i,c}$, the convective hindrance factor; J_v, the permeate flux; F, the Faraday constant; R, the universal gas constant; T, the absolute temperature; and $\delta\psi_m/\delta y$, the electrical potential gradient along the pore length, which is the driving force for the electrical mobility of the ions.

The zero-current condition corresponding to constant electric field, which is assumed by the electrical mobility contribution, is given by Eq. (8), and the electroneutrality condition in the membrane pores is given by Eq. (9):

$$I = \Sigma_i F z_i j_i = 0 \tag{8}$$

$$\Sigma_i z_i c_i + X = 0 \tag{9}$$

where X represents the charge density (charge/volume) in the membrane pore matrix.

Upon combination of Eqs. (7)–(9), taking into consideration that at steady state, $j_i = J_yC_{i,p}$; where $C_{i,p}$, being the concentration of the ith ionic component in the permeate, Elimelech et al. [31] got:

$$\frac{\delta\psi_m}{\delta y} = \frac{J_v\Sigma_i\frac{z_i}{D_{i,m}}(K_{i,c}c_i - C_{i,p})}{\frac{F}{RT}\Sigma_i z_i^2 c_i} \tag{10}$$

Upon using a similar approach to that followed by these authors and solving the Nernst–Planck equation, the permeate concentrations corresponding to the feed membrane surface concentrations were computed for the various ionic species. Comparison of the experimentally determined percent rejection to that computed according to the solution of the Nernst–Planck equation revealed only poor accordance. The impact of the conditions of the present NF testing, particularly the high TDS, on the applicability of the Nernst–Planck equation is currently the subject of evaluation.

Despite their presence in trace concentrations mixed with tremendously higher ionic concentrations, contaminant species, such as radionuclides and metal ions, were strongly rejected by NF. This confirms the absence of interference or masking effects in rejection of ionic species by NF. In fact, this is an advantage of NF rejection and an additional reason of its selection for treatment of PW. NF rejection of trace contaminant ions in the presence of high TDS background of raw water was reported to be independent of TDS even in the presence of ions of similar chemical nature at much higher concentrations as those of Ra^{2+} trace contaminants in the presence of Ca^{2+}, both being alkaline earth metals [15].

Analysis of NF permeate, Fig. 5, confirmed the upgrading of PW quality as injection water through the rejection of TDS of 34%, monovalent cations of 35–37%, hardness cations from 76 to 80%, while sulfate, the main hardness anion and the material of bacterial growth through anaerobic respiration of SRB and of the consequent corrosion of steel, Eqs. (2)–(6), was practically completely removed. This is attributed to the rather high negative charge on the surface of the polyamide NF membrane, which adds electrostatic repulsion with the anion to the strong rejection of the divalent ion [32]. In addition, NF also polished the removal of organics and SS after their partial removal in the coagulation step, and remarkably lowered the bacterial count.

Application of NF in the sequence (Intermittent chlorination/coagulation/NF) resulted in the suppression of biofilm formation on steel piping; consequently, both the related steel corrosion and the accumulation of radioactivity were inhibited.

3.4. Application problems of NF in treatment of PW

Despite the presence of residual traces of oil and grease, which are known to adsorb on, and strongly foul the polyamide membrane surface [19], only very slow decline of permeation with time was observed which was controlled by just four times organic cleaning of NF membranes, using 0.5 N NaOH, over 12 months of testing. NF membrane fouling lowered the permeation rate and the rejection of hardness cations, HMC, and radioisotopes; however, cleaning restored the original rejection efficiency.

Results revealed another positive aspect of application of NF for the treatment of PW. Despite the measured strong rejection of sulfate, which contributed to the inhibition of biofouling, scaling, microbial corrosion by SRB, and the deposition of uranium and other metals, no sulfate scale was formed on the membranes. The high ionic strength of the reject stream helped in keeping the sparingly soluble salts from precipitation [10]. On the other hand, the measured modest NF rejection of HCO_3^- from PW of only 27% led to relatively small increase in pH and CO_3^{2-} concentration in the reject without the formation of carbonate scale on the NF membranes.

4. Conclusions

1. Investigation of the degradation of quality of PW used in some of the Suez Gulf oil fields for reinjection into the oil formation, which would damage its porous structure, included evaluation and adjustment of the site present treatment by coagulation/multimedia filtration.
2. Site inspection revealed the formation of a biofilm inside the injection pipes containing mainly SRB, which induced microbial corrosion of the carbon steel pipes, enzymatic reduction of heavy metal cations and uranium 238 in PW and concentration of these pollutants in the biofilm to values much higher than the trace amounts in the GW. The proposed interpretation mechanisms explained the composition of the film, the microbial corrosion of steel, and the co-present hardness scales.
3. While coagulation/filtration removed rather efficiently SS and organics including hydrocarbons and oil, it did not suppress biofilm formation and the related phenomena of microbial corrosion and metal deposition. Release of organics, SS, iron compounds, and other contaminants from the biofilm was shown to re-contaminate the PW, which was already treated for injection.
4. Intermittent chlorination/coagulation/NF, on the other hand, beside polishing the removal of SS to crystal clear injection PW, realized:
   - (i) Strong rejection of SO_4^{2-}, organics, and micro-organisms, which inhibited the anaerobic bacteria growth and the related biological corrosion.
   - (ii) Strong rejection of heavy metal cations and U_{238} and, consequently, the inhibition of their enzymatic reduction by SRB.
   - (iii) Strong rejection of hardness components, which inhibited scale deposition.

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