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Nanofiltration and Low Energy Reverse Osmosis for Advanced Wastewaters Treatment

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1. Introduction

Nanofiltration (NF) and low energy reverse osmosis (LERO) are pressure driven, selective membrane separation techniques that progressively acquire new fields of application in treatment of surface, ground and wastewater (WW) (Deraniyagala, 2006) in view of their continuous development. Trends of their development are lower energy requirements, higher efficiency and specificity solute rejection, longer working lifetime at steady performance, and consequently higher process cost effectiveness, in addition to promotion of environmental safety. In fact, according to the results presented in the present chapter, NF is expected to replace, in the near future, several conventional techniques of WW treatment (Khedr, 2004, 2008, 2009).

NF membranes are generally characterized by relatively high surface charge (Childress et al, 2000), usually negative, and pores in the range of nanometer diameter (Jiang et al, 2004). In contradiction with the usually stated oversimplified description of NF membranes as intermediate type between loose RO membranes and tight ultrafiltration ones, the permeation performance of these membranes is mainly determined by their surface charge density, pores diameter and distribution, surface roughness and fouling susceptibility depending on the simultaneously working mechanisms of solute species size exclusion and electrostatic interaction (Thanuttamavong et al, 2002).

In general, NF membranes have high permeate flux and rather high rejection of polyvalent ions at low or moderate rejection of TDS. Therefore, they have lower opposite osmotic pressure and, consequently, lower operation pressure and lower energy consumption than RO under the same operation conditions (Khedr, 2008). The moderate salt rejection in NF also maintains the stability of the water and prevents it from being aggressive to network piping.

Efficient rejection of organics by NF and LERO was reported in a wide range of applications in wastewater treatment, food, chemical and pharmaceutical industries, for purification of industrial effluents and minimizing waste discharge. NF was also applied for rejection of natural organic matter which imparts color (Jensen & Thorsen, 1995). Efficiency of NF in removal of organics, suspended solids, colloids, microorganisms, and hardness components enabled its application for pre-treatment of sea water prior to SWRO or MSF in hybrid desalination processes. Such pre-treatment promoted the percent recovery of the subsequent RO step to 65% instead of 40% and lowered the fouling potential of RO membranes so as to lower the maintenance rate (Hassan et al, 1998, 2000; khedr, 2004).

Rejection of organic compounds, (micropollutants) is mainly controlled by their physicochemical properties, membrane material, and characteristics like porosity, pore diameter, and surface charge. Compounds with higher molecular weight show higher rejection. However, other factors as static and polar effects, degree of dissociation and adsorption play an important role in the rejection mechanism (Hofman et al, 1995).

Selection of adequate membrane process for decontamination of WW or groundwater depends on the level of contamination and the possible parallel requirement of desalination. In view of the operation pressure, energy consumption in membrane processes increases in the order NF < LERO < Conventional RO.

2. Evaluation of performance of selective membrane methods, NF and LERO in treatment of WWs and contaminated groundwater in comparison with the conventional methods

Several recent WWs treatment problems revealed the low efficiency or failure of conventional methods and suggested the search of more adequate methods of higher efficiency and wider validity ranges. As an example the radioactive contamination of groundwater common to certain areas in USA and the Middle East at levels that are dangerous for drinking is due, in fact, to ultratrace concentrations of radioisotopes as Ra^{2+} in mixture with other dissolved salts of more than 10 orders of magnitude greater concentration and may be of similar chemical nature as Ca^{2+} which would farther complicate the separation process (Clifford, 1991).

As for removal of heavy metal cations (HMCs) contamination of some surface, groundwaters and industrial WWs, granular activated carbon (GAC) was reported to be inefficient adsorbent in view of the higher temperature and pH dependence of the cation uptake (Gabaldon & Gonzalez, 2000). NF is expected to enable higher separation efficiency at lower cost.

On the other hand, the increasingly stringent norms of environmental protection render the disposal of the industrial or municipal WWs or sludge a major factor in the selection of industrial or municipal waste treatment processes.

The following results are part of a long term investigation (Khedr, G, 2004, 2007) that aims to evaluate the removal of contaminants by the modern cross flow membrane methods NF and LERO, in single or hybrid processes, in comparison with some of the common conventional methods e. g. chemical precipitation, softening, ion exchange resin (IER), and Coagulation/Settling/Filtration, and to determine the optimum process conditions for promotion of separation efficiency, cost effectiveness and environmental safety.

The main undesired classes of contaminants in industrial and municipal WWs required to be removed prior to reuse of water or to safe environmental disposal are:

- Organic compounds as dyes, fertilizers, food products ...... etc.
- Inorganic pollutants as HMC's, hardness components and/or total salinity.
- Trace radioactive isotopes.
- Colloidal matter either inorganic as colloidal silica or iron, or organic like humic substances.
- Dissolved gases like CO_2, H_2S, SO_2 or the radioactive Rn 222.
- Microorganisms and their life products.

During the comparative evaluation of performance of selective membrane methods and the subsequent consideration of their application problems, our discussion will be concentrated

on the first four classes of the above mentioned pollutants. Dissolved gasses are not removed by neither the selective desalination membranes nor the mentioned conventional techniques of WWs treatment. They are mostly removed by aeration, sometimes followed by chemical scrubbing or by chemical treatment like oxidation. Rejection of colloidal substances will be considered under organic or inorganic pollutants. Microbiological contamination cannot be removed by the conventional methods of separation. In membrane processes, feed water is usually sanitized in order to avoid biofouling. In fact, NF and LERO membranes reject a high percent of microorganisms, however, they cannot be considered as absolute barrier for water sterilization. Our results of high temperature RO of WWs, to be discussed later on, have shown remarkable control of biological growth under certain conditions without interference with the rejection of the other water contaminants (Khedr, G, 1997).

2.1 Testing details

A mobile pilot unit was designed so as to enable conduction of NF and LERO runs over a wide range of operation conditions, Fig (1), feed pressures, flow rates, and pretreatment steps. Percent recovery ranged from 85–90 at feed pressures of 6 and 8 bar for NF and RO, successively. Both permeate and reject streams were re-circulated back to the feed tank which was thermostated at 25°C. Feed pH was adjusted to the range 7.5 to 8 except upon comparing membrane results with those of hot lime softening (HLS) where the high pH of the softening method was adopted. Ionic concentrations were determined by ICP–AES (Perkin- Elmer, Boston, USA). The radioisotopes Ra $(NO_3)_2$ and UO_2Cl_2 were supplied and analyzed by radio counting curtsey from the Regional Middle East Centre of Radioactive Isotopes (Cairo, Egypt).
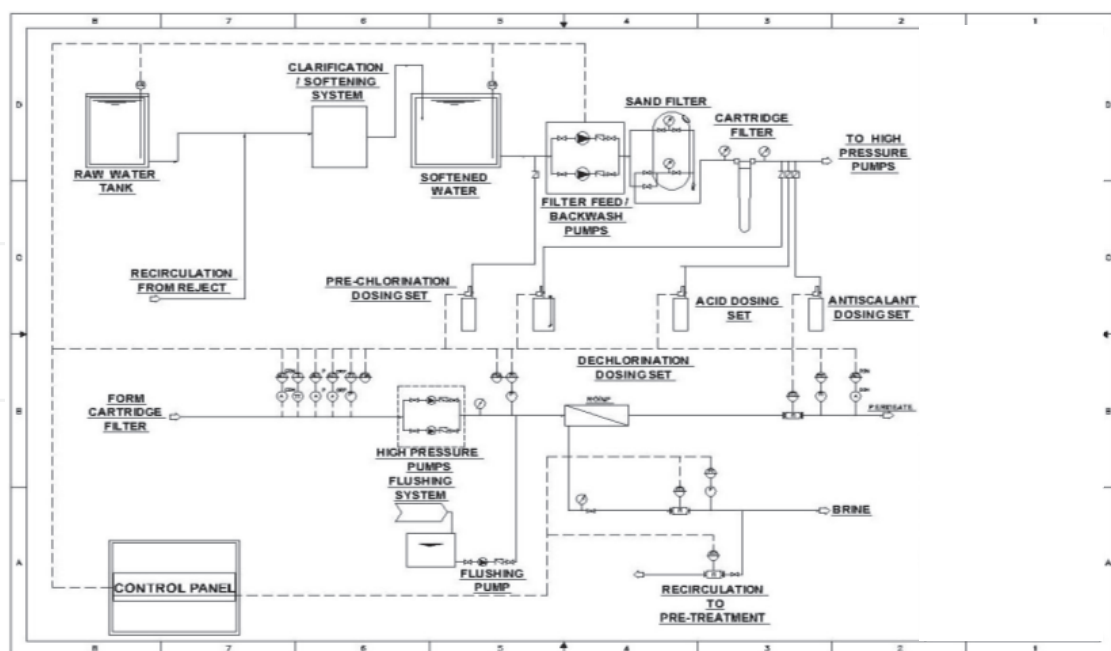

Fig. 1. Schematic representation of the mobile NF + RO pilot unit

A laboratory experimental system having six test cells with circular turbulent agitation at the level of surface of membrane coupons was used in a test circuit which consisted of a low

pressure pump, pressure gauge, cartridge filter, flowmeter and thermostated feed tank. 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 solution for one hour. The testing feed pressures ranged from 80 to 100 psi for NF experiments and from 100 to 120 psi for LERO ones. Tangential cross flow velocity ranged from 0.005 to 0.1 m/s and feed flux from 120 to 720 L/m^2.d. The synthetic contaminated feed water used was prepared by dissolution of Cu SO$_4$ to give 5.5 ppm Cu^{2+}, CaCl$_2$ to give 57.3 ppm Ca^{2+}, FeCl$_3$ to give 15.8 ppm Fe^{3+}, Na$_2$ SO$_4$ to give 88.6 ppm Na$^+$ and KSO$_4$ to complete SO$_4$ $^{2-}$ concentration to 163.4 ppm. The same procedure was used to prepare the other feed water compositions. Thin-film composite NF (HL4040F) and LERO (AK4040F) membranes of polyamide chemistry (GE/Osmonics) were used throughout the present study in coupons for cell testing and in 4" elements for pilot testing.

2.2 Performance of NF and LERO in removal of HMCs and radionuclides
Fig. 2 shows the results of treatment by RO, LERO, and NF of synthetic contaminated water containing beside usual drinking water components, Cu^{2+} as HMC as well as trace concentrations of the radionuclides Ra^{2+} and UO$_2$$^{2+}$. Rejection of radionuclides is calculated according to:

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

Only modest rejection is observed for monovalent species as is expected with RO and particularly with NF, however, rather strong rejection is recorded for polyvalent ions. LERO and NF permeate showed decontamination till less than the maximum contaminant level (MCL), for drinking water according to the norms of the US, Environmental Protection Agency i.e. 5 pC$_i$/l for combined radium (Ra$_{226}$ + Ra$_{228}$) and 20 ppb for uranium 238. The observed order of percent rejection of the solution components agrees with the expected mixed control of both Donnan and diffusion effects for a porous charged membrane (Hilal et al, 2004).
Polyvalent radionuclides, whether cationic or anionic, were strongly rejected despite their presence as trace concentrations in mixture with tremendously higher ionic concentrations of similar chemical nature, which indicates the absence of interference with rejection of the contaminants. Still higher rejection is measured for LERO, specially for monovalent species and TDS.

2.3 Effect of ion background concentration on NF and LERO contaminant rejection
Fig. 3 shows the performance of NF rejection of the TDS, Na$^+$, Cu^{2+}, and Ra^{2+} from a mixture solution of CuSO$_4$ and NaCl to which a trace of Ra(NO$_3$)^2 was added. The test runs were conducted over a wide range of background feed TDS, at constant contaminant cation concentration of 25 mg/l Cu^{2+} and 124 pCi/L Ra^{2+}.
Cation rejection decreases with concentration due to increase in concentration polarization at membrane solution interface (Khedr, 2000).This effect is much more pronounced for Na+ where, a rather linear decrease of rejection is observed. Since the monovalent ions represent the main component in the feed water, the NF rejection of TDS decreases practically parallel to that of Na$^+$.

As for divalent cations, on the other hand, stronger rejection is observed which concords with Donnan effect considerations (Albino & Donald, 1988). It is interesting to notice that the rejection of radioactivity, which belongs to a very low chemical concentration, is practically complete and independent of the background concentration over the investigated range.
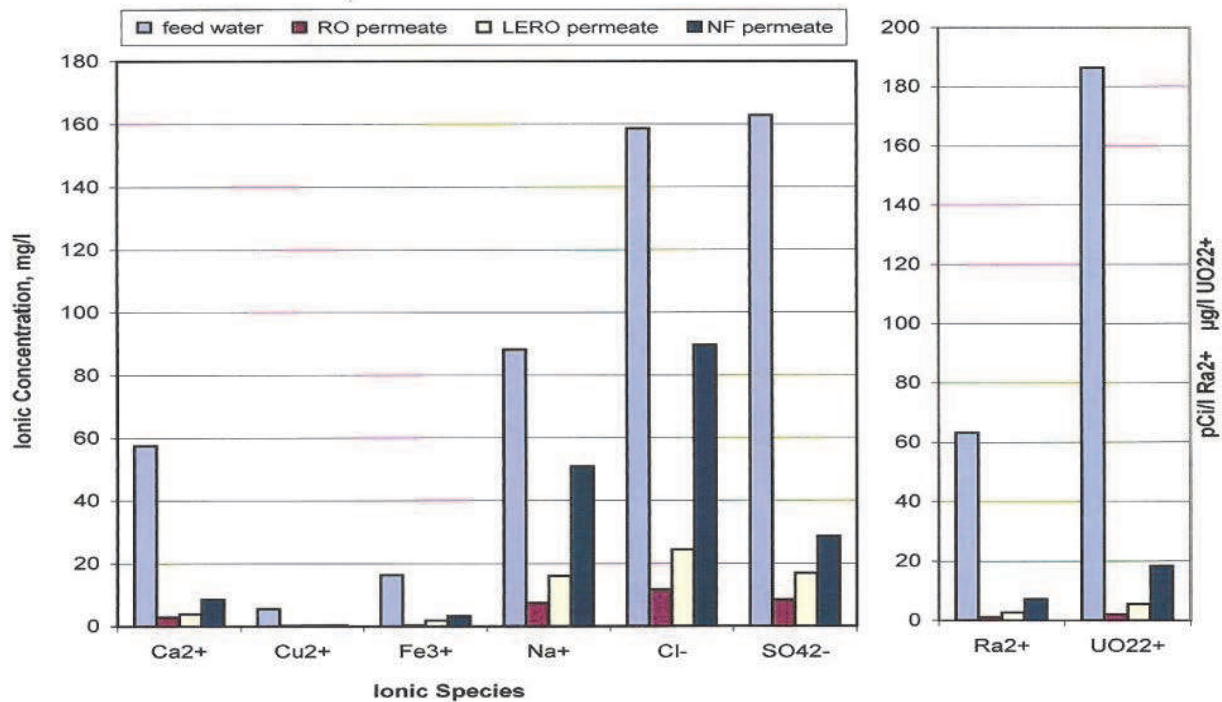

Fig. 2. Comparison of performance of membrane methods RO, LERO, and NF in treatment of contaminated feed water

These results confirm that the rejection of a trace HMC or radioactive isotope by NF is not subject to any screening or interference by the co-present much higher concentration ionic background in water. Similar behaviour was obtained with LERO membranes. However, in this case the recorded general higher rejection and lower decline of TDS and Na⁺ rejection with concentration are due to the higher selectivity of the RO membrane. Radium rejection of the order of 98% is recorded.

According to the results of Figs (2, 3), NF would be the optimum method for rejection of the considered contaminants from drinking water sources. When parallel water desalination is required in addition to the removal of HMCs or radionuclides, on the other hand, LERO is the candidate technique. Contaminant rejection takes place parallel to that of other salts in the same system without using additional equipment or process modification.

NF may also be applied as the first step in a hybrid membrane process (NF/RO). Results have shown in such case that the last polishing RO step, while it improved a lot the quality of the final product water so as to attain drinking water norms, it added only slightly to the process cost since it was performed at lower pressure and high recovery. In fact, analysis of **NF permeate confirmed that pretreatment by NF upgraded waste water quality through efficient removal of colloids, suspended solids, hardness components, microorganisms and organics (Khedr, 2008).**
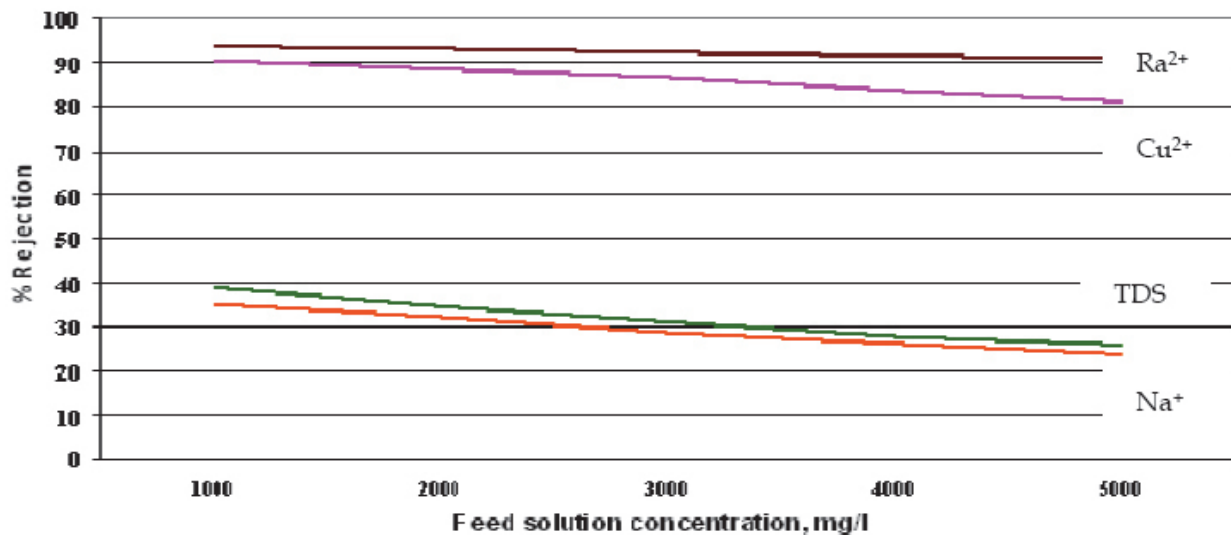
Fig. 3. Variation of rejection of Cu^{2+}, trace of Ra^{2+}, Na^+ and TDS with solution concentration in LERO

2.4 Removal of the contaminant ions by IERs

Synthetic water samples contaminated by HMCs and radionuclides were treated by chelating strong cation and anion exchange resins. Samples of product water were periodically collected from the resin columns for chemical analysis and radioactive count. Removal of contaminant ions of the order of 85 to 95% was recorded. However, results revealed the following drawbacks of this method as compared to membrane methods:

Strong interference of the polyvalent ions with ion exchange of the contaminant ions was invariably observed. This is particularly pronounced for ions of similar physico-chemical behaviour as e.g. interference of scale forming cations as Ca^{2+} with rejection of Ra^{2+}, both belonging to alkaline earth metals. Comparison of separation of Ra^{2+} in absence and in presence of Ca^{2+} confirmed that affinity of CER is much higher for the heavier cation, however, this is partially counteracted by the much higher concentration of Ca^{2+}. Since parallel Ca^{2+} removal can't be avoided, a bigger system is required to realize the required efficient contaminant removal.

The high affinity of the resin to the HMCs or the radioisotopes makes the subsequent resin regeneration quite difficult. 10% NaCl regenerant did not remove more than 23% of the retained uranium. Slightly better regeneration was observed by successive elution by 4% NaCl followed by 1N HCl as was previously advised (Sorg, 1988). However, neither process was totally effective in removing all of the uranium or radium which continued to accumulate after each run on the IER. Use of higher concentration NaCl solution and/or increasing the regeneration time improved only slightly the percent regeneration. This can be attributed to the relatively large size and poor hydration of Ra^{2+} ion which would locate it in relatively inaccessible hydrophobic regions of the resin phase. Results of the present work showed that chelating resins are more efficient for removal of polyvalent contaminant ions than conventional resins; however, they are also more difficult to regenerate. Large quantities of regenerant solution would be required if 100% removal of uranium should be approached.

According to Fig (4), at the same acid pH of feed water containing 70 µg/l U 238, the H^+ form of the CER enabled 93% removal compared to 68% for the Ca^{2+} form under the same

conditions. At intermediate pH of 6.0 lower removals was observed. At pH 7.6, AER showed much higher removal. **On the other hand, over the whole pH range NF showed higher and rather steady rejection behavior, slightly lower at pH 6.0**.

The observed high pH – dependence of U removal by IERs is due to the high reactivity of U and its affinity to form complexes to give various ionic species (Sorg, 1991). In acid Cl^- solution, UO_2Cl_2 is the prevailing form of U where the uranyl cation UO_2^{2+} is highly stable and exchanges at such high rate with H^+ ions. In slightly acidic pH in the range from 5 to 6.5 neutral carbonate molecule is forward, $UO_2CO_3^{\circ}$. In near neutral pH values, on the other hand, the uranyl ion forms stable complexes with - CO_3^{2-} which is common to drinking water supplies, the predominant anion species is $UO_2(CO_3)_2^{2-}$ and $UO_2(CO_3)_3^{4-}$. This explains the observed rather complete removal of uranium by AER and their difficult regeneration at such pH. These results confirm and add to the previous consideration that IER would probably not be practical for decontamination of drinking water supplies (Lee et al, 1982).

Separation of contaminant ions by IERs is not steady. It decreases with time with start of contaminant break through near the resin exhaustion.

Important amounts of concentrated brine, or other regeneration chemicals, are required for periodical resin regeneration (which is seldom complete). The contaminated regenerant solution has then to be disposed of.

2.5 Removal of the contaminant ions by softening and chemical precipitation
2.5.1 Removal of HMCs and radioactive isotopes by NF and LERO in comparison with Hot Lime Softening (HLS)

Rejection of the poisonous HMC Cd^{2+} and a trace of Ra^{2+} , in presence of Na^+, Ca^{2+}, Cl^-, SO_4^{2-}, HCO_3^- and CO_3^{2-} was investigated in comparison with their removal by the conventional (HLS). In order to compare the performance of these methods under similar experimental conditions, the dosing of lime and soda ash was adjusted so as to realize almost the same level of Ca^{2+} rejection by the NF. In Fig (5), for each species, the first column gives the initial concentration, the second gives the result after NF treatment and the third gives the result after HLS, independently realized.
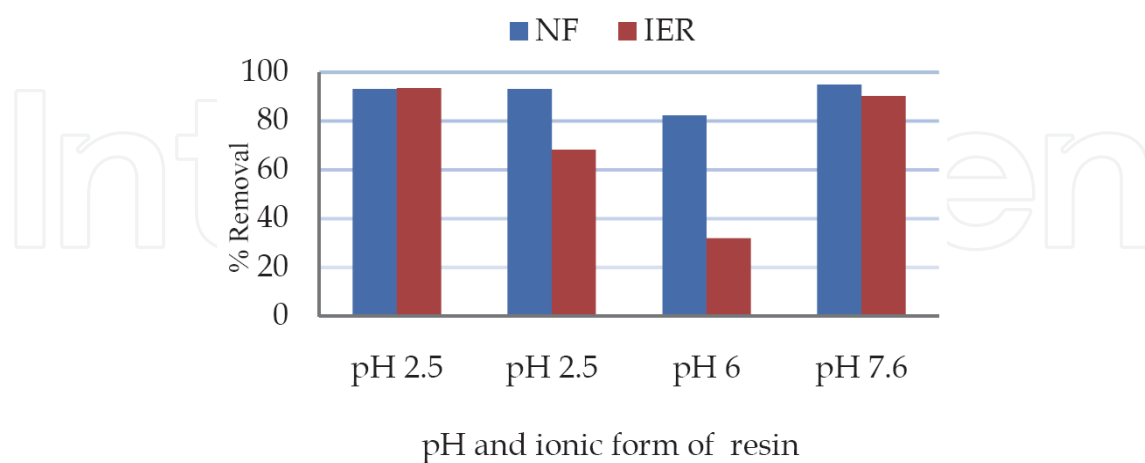

Fig. 4. Comparison between Removal of U238 by NF and IERs

While NF led to lowering the concentration of all the species to different extents and consequently to lowering of TDS, HLS lowered only the concentration of hardness components Ca^{2+}, Ra^{2+}, and HCO_3^- and of HMC. On other hand, HLS raised Na^+ and CO_3^{2-}

concentrations. Concentration of the anions which are not included in softening reactions like Cl⁻ and SO₄²⁻ were not modified. These modifications are directly explained by the softening reactions of HLS.

NF has, therefore, the advantages over HLS of (1) parallel general water desalination, (2) absence of continuous chemical dosing stoichiometric to the hardness rejection which attains several tens of tons per day in big HLS softening facilities, (3) absence of contaminated solid sludge formation which requires periodical disposal, (4) mechanism do not include slow steps like settling, (5) do not require subsequent filtration, NF filtrate being crystal clear, (6) the relative ease of operation of membrane processes, (7) NF do not require such sophisticated installations with dosing silos and agitated reactors.

Further (7), as for the process cost, results of Fig (5) show that removal of other hardness components by HLS cannot be avoided. In order to remove the radionuclide contamination to the desired low level, most of hardness components parallel to Ra^{2+} should simultaneously be removed. Such interference results in a higher rate of chemicals consumption in higher capacity softening installations, and leads to a higher amount of contaminated sludge. NF application, on the other hand, is shown to lead to co-rejection of all these components without interference. It will have, therefore, lower cost.
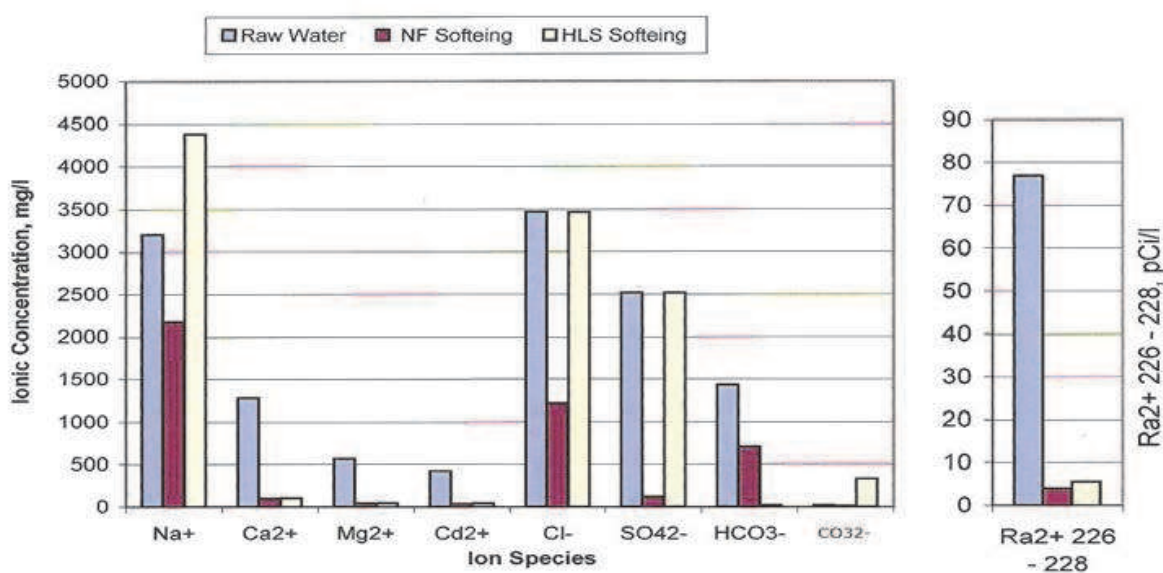

Fig. 5. Comparison between performance of NF and HLS in removal of HMC and radioisotopes.

2.5.2 Separation of HMCs and radionuclides by chemical precipitation

The most common method used is precipitation of contaminant polyvalent cations as hydroxides by means of e.g. lime dosing. According to Fig (6) pH adjustment is critical for optimal precipitation of most of cations and specifically for amphoteric ones like Cr^{3+} and Zn^{2+}, where a slight increase in pH results in a remarkable decrease in precipitation efficiency by dissolution as CrO₄²⁻ or ZnO₂²⁻. For the purpose of comparison, the separation by NF on Fig (7) was realized independently for each cation at the same concentration of 75 ppm in mixture solution with Na Cl at the same TDS from (Na₂CrO₄ + NaCl), (CrCl₃ + NaCl), and (ZnCl₂ + NaCl) mixture solutions. Steady rejection of HMCs is obtained over the whole pH range for both cationic and anionic forms. Preferential rejection is even more pronounced for

anions than for cations. CrO_4^{2-} showed higher rejection than Cr^{3+} despite the higher ionic charge, also rejection of Zn^{2+} increased in the high pH range of the line Zn^{2+} / ZnO_2^{2-}. This behavior was previously observed and attributed to the coulombic repulsion with the residual negative charge on the polyamide membrane surface (Khedr, 2000).

According to Fig (6), in case of water contaminated by more than one HMC the possible pH of co-precipitation will be at much higher solubility than the theoretical minimum value for each cation. In such case, NF and LERO enabled efficient co-rejection of contaminants, Fig (2), without measurable interference. Membrane rejection is also much more feasible than the recently proposed two-step precipitation of HMCs, first as hydroxide then as sulphide (Cort, 2005), each step being followed by independent coagulation, flocculation settling and filtration. As for chemical precipitation of radioisotopes, in the inhand work, only limited testing was preformed to deposit Ra^{2+} through dosing of $BaCl_2$ according to:

$$Ba^{2+}_{(excess)} + Ra^{2+}_{(trace)} + SO_4^{2-} = Ba\ (Ra)\ SO_4\ (s)$$
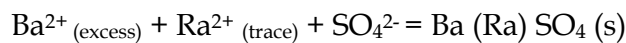
This reaction is reported to be highly specific to Ra^{2+} separation and leads to its quantitative deposition (Clifford, 1991). However, our results showed its deposition in a finely divided form which clogs in part to the walls and piping of the reactor and settling equipment causing strong radioactive contamination of the system. Also since such deposit includes highly concentrated radionuclide, it represents a waste disposal problem. In fact, it should be buried in a standard radioactive waste disposal facility with all the related potential environmental risks.

The NF radioactive waste, on the other hand, is dissolved in the reject stream. Upon drying in e.g. the evaporation ponds it is diluted in the solid state by the co-rejected, non radioactive salts and compounds so that the radioactivity of the deposit is usually lower than the level that imposes burying in a "low-level radioactive waste disposal site" i.e. 1-3 nCi/cm^3 (Hahn, 1991). Instead, contaminated NF or RO reject streams have several selections for safe environmental disposal. As per the level of contamination they may be discharged to storm sewers, surface water bodies or sanitary sewers, or blended with sanitary effluent streams so that the final stream radioactivity becomes lowered to the trace state.

2.5.3 Removal of the contaminant ions by coagulation, flocculation, and settling

In order to compare the results of rejection by membrane methods to those of conventional coagulation, laboratory testing was conducted using the same synthetic contaminated feed water under the previously described testing conditions. Jar tests were conducted using both ferric and aluminum coagulants over the dosing rate of 1 to 20 mg//l for various feed pH values covering both acid and alkaline ranges. Results of Fig (7) showed wide variation of behavior between different contaminant ions. As an example while Cu^{2+} and Pb^{2+} were removed at 95% and 97%, successively, Hg^{2+} was only 52% removed, and Cr^{3+} and Ra^{2+} were not removed.

Efficient uranium removal by coagulation was observed, however, the extent of removal is function of water pH and selected coagulant. While ferric chloride led to 82% removal over the pH range of 6 to 10, higher removal of 95% was obtained in presence of alum coagulant at pH 10. The sensitivity to pH in case of coagulation is due to its influence on the nature of the ionic form of U as was previously mentioned.

According to these results separation of such contaminant ions by conventional coagulation does not show a general trend of behavior. It is too much dependent on water composition,

pH, valency state of ionic contaminants, type and dose of coagulant, as well as other process variables. **In order to assess the validity of this method for a specific application, evaluation testing has to be conducted.**
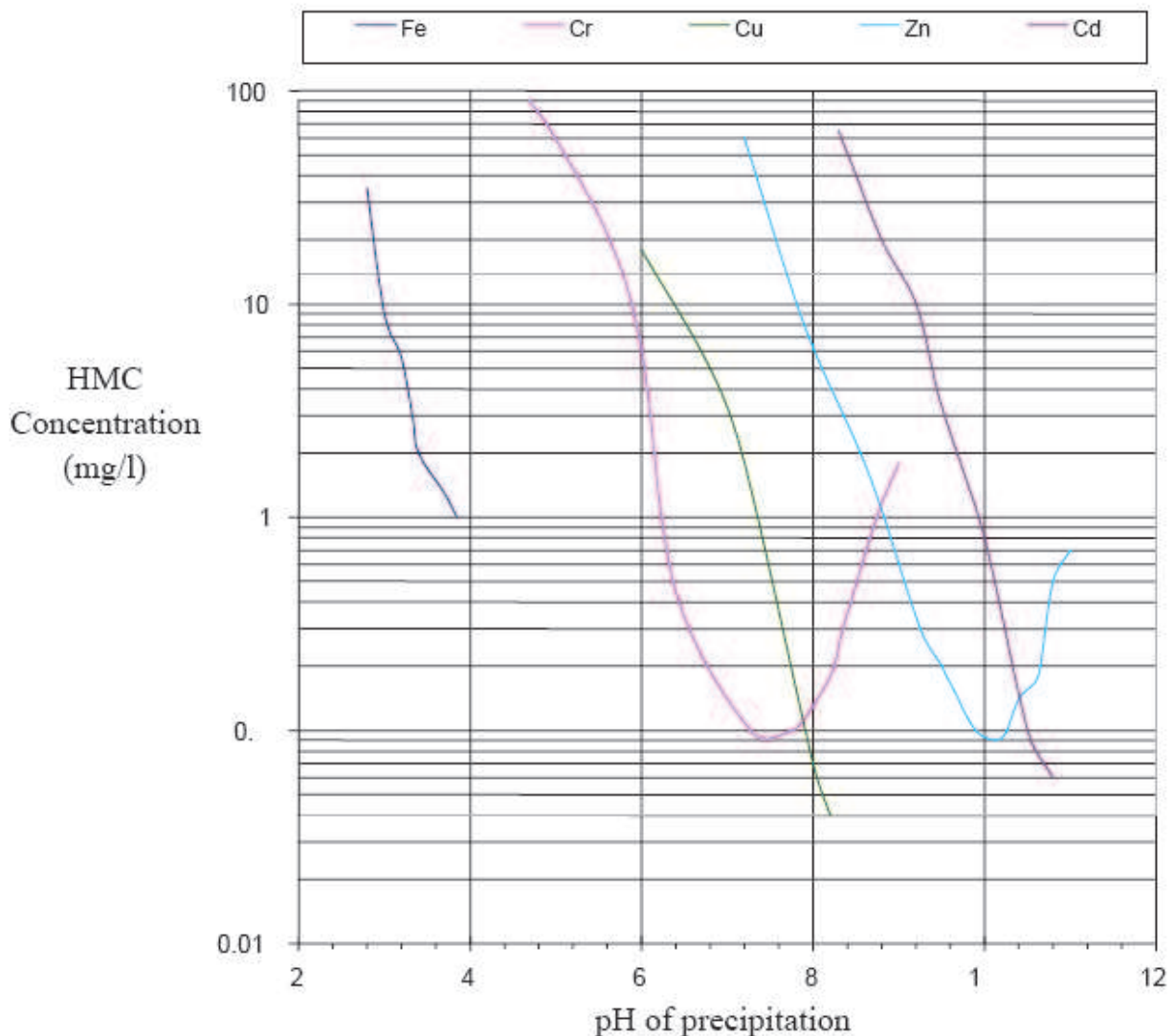

Fig. 6.

Rejection by NF and LERO, on the other hand Fig (7), is quite predictable and is mainly determined by the characteristics of the contaminant ions. Only slight effect of pH on NF separation is recorded. Membrane methods, again, do not include slow steps like settling, and do not require specific chemical dosing or subsequent filtration.

3. Problems encountered in NF and LERO treatment of WWs

The main application problems of these techniques are the phenomena of membrane fouling (khedr, 2000) where undesired rejected components accumulate on and/or in membrane and block its surface and would interfere with its function. Organics, microorganisms and their life products lead to development of colloidal fouling (Rizwan et al, 2007), organic fouling (Elimelech & Li, 2004), and biofouling (Koek et al, 2003). Saturation of inorganic

sparingly soluble salts leads to scale deposition (Bertels et al, 2008). These phenomena are far of being cases of simple mechanical surface blocking. In fact, they interfere with membrane rejection mechanism.
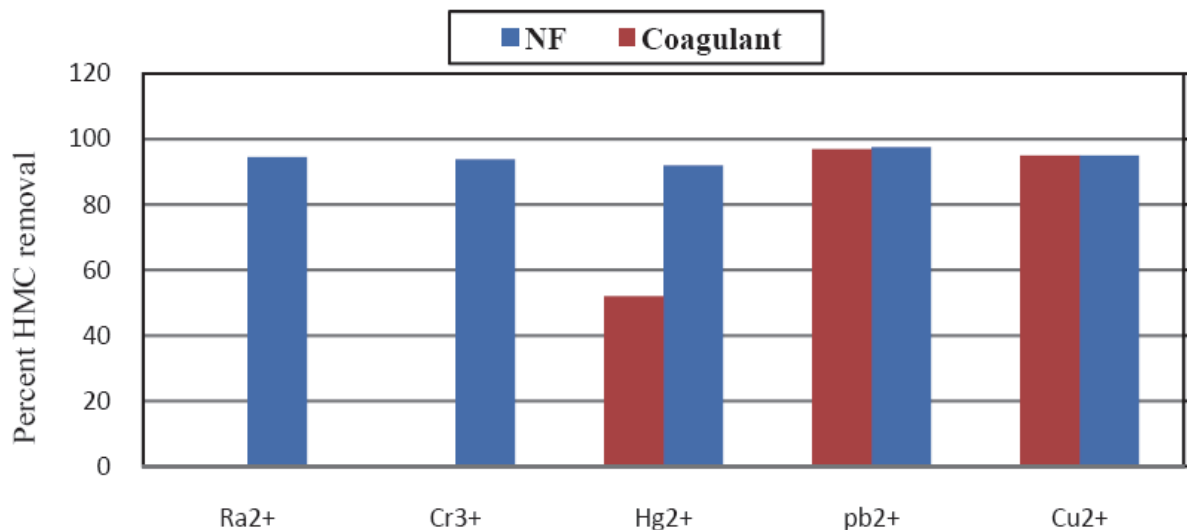

Fig. 7. Comparison between removal of HMCs and Radioisotopes by NF and by coagulation.

Fouling lowers both process efficiency and cost effectiveness through decline of the permeate rate, repeated shutdown for cleaning which would shorten the membrane lifetime, increasing the energy consumption, and modifying the rejection selectivity which decreases the product quality. Only limited works were reported about the potential interference between the various forms of fouling and its effects on NF performance (Khedr, 2000; Elimelech & Li, 2004).

Another difficulty encountered in NF system design which promotes fouling is due to its quite low operation pressure. A balanced flux throughout the system stages is sometimes difficult to achieve and maintain. Too high permeate rate would be obtained in the first stage which leads to too low brine flow rate in the last ones. This increased the potential of organic fouling in first stages and scale deposition in the last ones (Khedr 2008, 2009).

In order to characterize the development of complex fouling on NF membrane and the possible effect of interaction between the different forms of fouling, results of periodical inspection of membrane surface during advance of fouling by SEM and analysis of fouling deposit by energy dispersive X-ray and Fourier Transform Infra Red Spectroscopy, were correlated to the measured parallel decline of NF permeation and solute rejection.

Fouling tests were conducted using water from one of the collection streams of wastewater of the industrial city of Riyadh, KSA; typical analysis is given in Table 1. Composition of this stream remained rather steady over the duration of the experimental work.

Membranes used in investigation of high temperature NF are of quality characterized by high temperature resistance (Duratherm Membrane Series, Osmonics/GE). In this case, permeation rates and rejection values are measured in absence of temperature gradient i.e. they are free from effect of thermoosmosis. The reported changes in membrane performance are reversible and not related to polymer degradation due to high temperature.

Component	Concentration ppm	Parameter	Value
Ca^{2+}	122.8	TDS	1543.7 ppm
Mg^{2+}	12.5	pH	7.6
Na^+	424.8	TOC	64 ppm
K^+	17.0	Bacterial count	Too numerous to count
NH_4^+	12.8		
NO_3^-	46.0		
Cl^-	630.0		
SO_4^{2-}	160.4		
SiO_2	22.5		

Table 1. Typical Raw Wastewater Analysis.

3.1 Fouling behavior of NF membranes

Fig (8) shows the decline of NF product rate with time for the three stages of the NF system in absence of antiscalant. Different rates of decline are observed for different stages. While the first and second stages started by a gradual rate of decline which increased remarkably after the first 20 hours of operation, the flow rate of the third stage suffered a rather rapid decline to practical blocking after a much shorter time. The first stage then showed some increase of permeation to compensate for the decrease of the last one. This behavior is typical to scaling of the third stage membranes. Decline of first and second stage permeation rate indicates another form of fouling that accumulates on the membrane surface as e.g. organic and/or biofouling. The chemical analysis of the spent cleaning solutions confirmed the mentioned nature of the fouling film.

In presence of antiscalant (Genesysis International, DS, UK), on the other hand, usual decline in permeation was recorded for the three stages of the system, Fig (9).
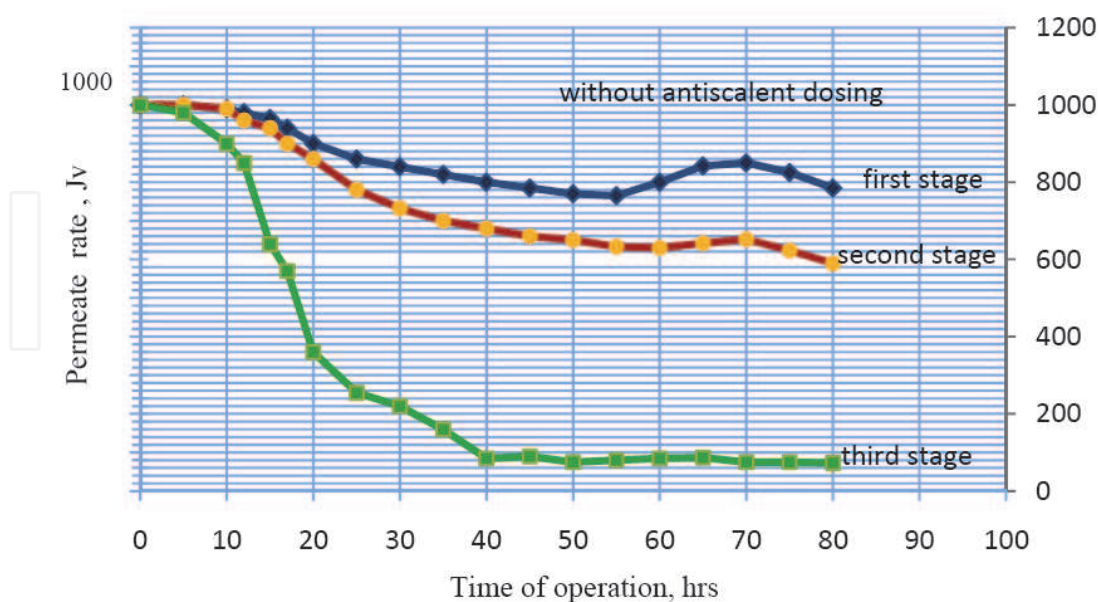

Fig. 8. Variation of permeate rate with time for the three stages of the NF system

In order to verify the relative importance of organic fouling and biofouling in formation of the fouling film similar test series were conducted in absence then in presence of UV

radiation at the level of inlet to cartridge filtration (Trojan UV Swift ECT, FL, and USA). In absence of UV radiation a much faster rate of decline of permeation is observed particularly after an incubation period of 14 days. Our results have also shown that: the use of phosphate-based antiscalent as hexametaphosphate contributed to increase of biofouling rate, specially in the high temperature condition of summer time (ambient temperature of 30 to 35 °C) higher rates of bacteria development and fouling film thickening were observed than in the case of non-phosphate based, polymeric antiscalents.

Upon testing of granular activated carbon filters for lowering the raw water content of organics, those filters became themselves contaminated with bacteria and acted as substrate for bacterial growth and source of additional contamination of feed water.

Correlation between decline in permeation and progress of fouling is confirmed by SE micrographs realized for membrane coupons collected at different time intervals over the test duration, as well as through analysis of the deposit accumulated on membrane surface.
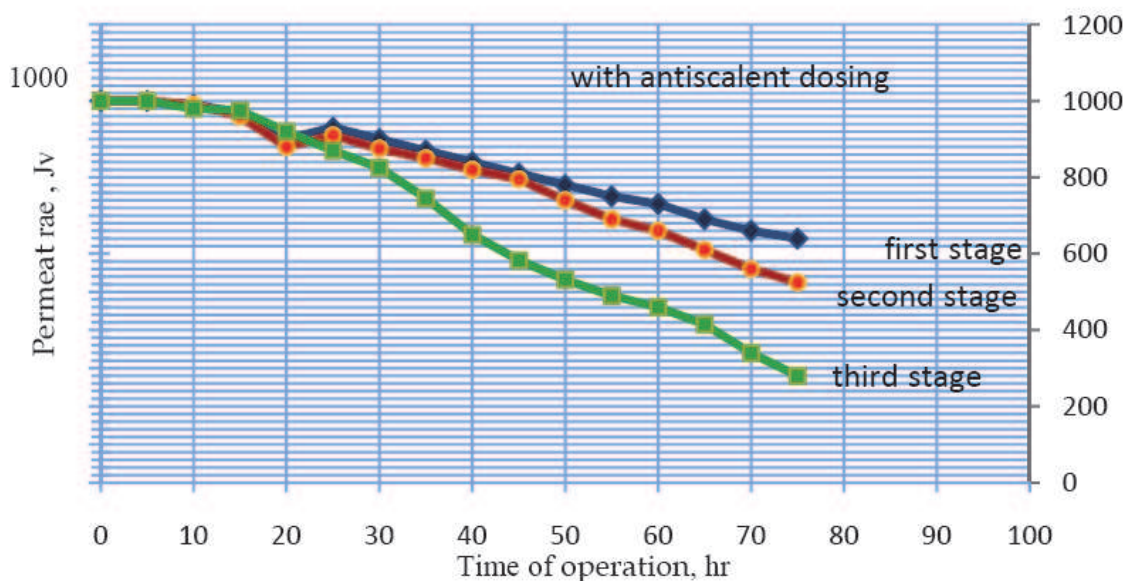

Fig. 9. Variation of permeate rate with time for the three stages of the NF system

3.2 Chemical composition of the fouling film

Table (2) shows the chemical analysis of the fouling deposit. Elemental composition of the film shows sulphur on the fresh membrane surface which would belong to the polysulphone supporting film of the membrane. The main elements in the deposit are phosphorous (P) and Calcium (Ca). P would be due to adsorption of the phosphate-based antiscalant in the fouling film. HMP may also become hydrolyzed to orthophosphate, which would deposit calcium phosphate scale. Phosphates may also be fixed by bacteria. On the other hand, calcium detected in the fouling film may be part of metal-organic complex created by interaction of the divalent cation with organic molecules. This was reported to result in highly compacted fouling layer and substantial flux decline (Seidel & Elimelech, 2002; Cho et al, 2000).

Since raw water did not contain iron, its presence in the deposit is attributed to corrosion of steel or stainless steel. In fact, a trace of some heavy metal cations e.g. in sewage water, can induce a rather severe galvanic corrosion of steel in view of cathodic depolarization through an autocatalytic displacement mechanism (Khedr, 1989).

As for the organic components detected in the film without being present in feed water, proteinacious and hydrocarbon derivatives would be metabolic products of bacteria. (proteinacious amide and long chain hydrocarbon ester). Polysulphone and polystyrene would be contribution removed from membrane surface and membrane separators along with the accumulated deposit. Analysis of samples of the fouling film and bacterial count confirmed the progressive accumulation of organics and bacteria cells in case of organic/biofouling parallel to the measured decline of permeation rate. In case of UV exposure a lower film material is accumulated which is practically free from growth and propagation of bacteria cells. While organic fouling accumulates on membrane surface since the beginning of each run, an incubation period is required for biological fouling to contribute effectively to the decline of permeation together with a higher rate of organics accumulation in the film.

3.3 Forms of membrane fouling

Membrane fouling is usually classified into different types which are attributed to presence of certain components in the feed water (Ridgway, 1988).

- Inorganic or crystalline fouling due to deposition of sparingly soluble salts of Ca, Mg, Sr and Ba or of SiO_2.
- Organic fouling, due to adsorption on membrane surface of various dissolved organic compounds.
- Particulate fouling, due to accumulation of suspended particles as silt or clay.
- Colloidal fouling, due to colloidal organic or inorganic components.
- Biofouling, due to microorganisms and their metabolic products.

Sample	Major Elements	Minor Elements	< Minor < 1%
Fresh membrane surface	_	S	
case 1 (deposit on membrane surface)	Ca, P	Si, Fe	Ti, Al, Mg
case 2 (deposit removed from membrane surface)	Ca, P	Si, Fe	Ti, Al, Mn

Concentrations are estimated from spectral lines intensities.

Table 2. Analysis of fouling deposit (Energy dispersive X-ray results)

However, such consideration of individual forms of membrane fouling oversimplifies the situation observed on NF or LERO membranes in applications which include rejection of inorganic, organic and biological contaminants from waste waters. In fact, most of, the cases of membrane fouling include combinations of these forms or even all of them in one and the same film (Khedr, 2000). The present work considered the potential interaction between these forms of fouling and its possible influence on the fouling rate, NF performance decline, and recovery upon cleaning.

3.4 Mechanism of development of fouling film

Through surface inspection by Scanning Electron Microscopy (SEM) of periodically collected membrane samples during the progress of membrane fouling, analysis of fouling deposit by EDX-ray and FTIR-Spectroscopy, and correlation to the parallel decline of permeate rate and solute rejection, the following sequence of steps was observed to determine the mechanism of development of fouling on membrane surface:

3.4.1 Colloidal fouling

The fouling film starts to form invariably under all operational conditions by the primary accumulation on membrane surface of organics, silt, and colloidal particles of organic and inorganic nature that escape filtration. These particles are entrained and adhere to membrane surface by convective forces. They may also be fixed on it by forces of electrostatic or chemical nature, e.g.:

- Through interaction between the surface charge of the membrane and the stabilizing layer of charges of the colloidal particles.

In fact, zeta potential measurements of NF membranes have shown that membrane fouling susceptibility is higher the higher the measured negative surface charges (Elimelech & Li, 2004).

- And/or the interaction between the polyvalent cations like iron or aluminum accumulated by membrane rejection in the diffusion layer with the stabilizing charge of colloidal particles, which leads to the neutralization of the latter and their consequent deposition on membrane surface.

3.4.2 Primary scale deposition

It takes place if the dosing of acid or antiscalant is unadequate to cover the supersaturation of the scale forming salts or compounds or if the related system design parameters are exceeded i.e. if NF is conducted at too slow brine flow or too high recovery.

Results confirmed what was previously observed (Khedr, 2008, 2009) that if the balance of flows in the NF testing is not precisely controlled according to system design values, fouling and scaling susceptibilities of membranes are enhanced. On the other hand, organics may interfere with scale deposition. Even at slightly positive LSI values, scale formation was not observed. In fact, surface adsorbed of organics may act as scale inhibitors (Bartels, 2008).

In presence of bacterial contamination of the feed water, membrane fouling may be further complicated by the parallel development of biofouling:

3.4.3 Formation of biofouling film

Results confirmed that particularly in hot summer time the contribution of biofouling to the decline of permeation of NF is quite more important than the other forms of fouling in absence of scale deposition. Fig (10) shows the SE micrographs of surface of membrane samples periodically collected at 3-days intervals during the progressive development of biofouling. Biofilm starts by adsorption of bacteria cells on membrane followed by film growth.

3.4.3.1 Adsorption of bacteria

A small fraction, less than 1% of bacteria cells (biocolloids) in the feed water adsorbs on the membrane to cover about 10 to 15 % of its surface. Such low adsorption is attributed to electrostatic repulsion between the residual negative charge on the thin film polyamide NF membrane and that on the bacteria cells at near neutral pH values. Adsorption is assumed to take place through intermediate connection through the extracellular polymer substance (EPS), life product of bacteria. Bacteria cells may reside on or near the membrane surface under force of permeation drag until the extruded EPS ensures strong attachment.

Electrostatic repulsion is, therefore, overcome by a combination of chemical bonding and brownian displacements of the bacteria cells. Factors like cross flow rate, permeate rate, water solutes, suspended matter, and membrane surface characteristics would also contribute to the rate of adsorption of microorganisms (Hoek et al, 2003). Possible effect of membrane surface roughness on the extent of bacterial adsorption was not evaluated in the present work.
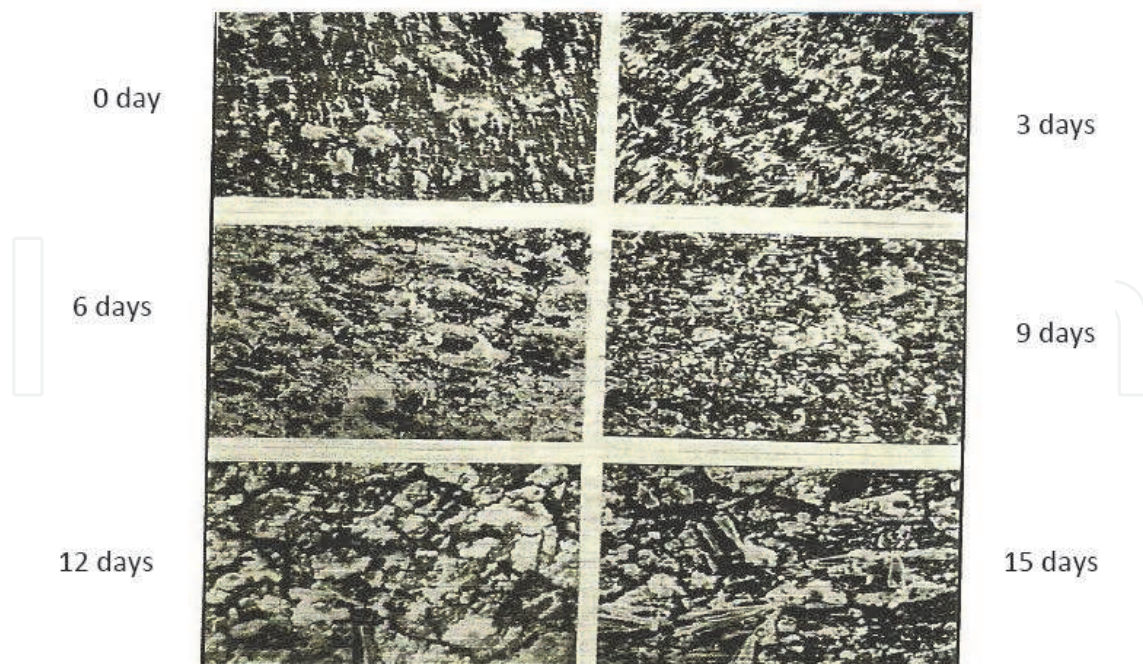
Fig. 10. SEM's of membrane samples showing the progressive accumulation of biofilm over 15 days of NF

3.4.3.2 Propagation of biofilm

The adsorbed bacteria were observed to progressively cover the membrane surface by the life products. Full surface coverage by a layer of several microns thick was obtained in about 15 days at an ambient temperature of 32° C. Rate of propagation of biofilm was observed to depend on the continuous exposure to contaminated feed water. The faster film growth and performance decline in this case is due to the role played by the slime gel matrix of the EPS of bacteria. It favors further organic and biofouling through entrapping of the organic molecules, colloidal particles, suspended particles and bacteria cells carried by the feed WW stream which we considered as (secondary adhesion). **This is an example of interaction between the different forms of membrane fouling. In fact, in contact with uncontaminated stream, the initial biofilm showed a much slower rate of growth.**

Addition of organic biocides could control the biological growth, (DOW Antimicrobial 7287 and Permatreat PC-11, Nalco). However, sometimes organic slimes were detected on the membrane surface which would be due to interaction between organic biocides with the present polyvalent cations (Yuan & Zydney, 1999; Cho et al, 2000).

3.4.3.3 Fouling film denaturation

Up to this stage in the development of NF and LERO membrane fouling results of chemical cleaning showed successful film removal and recovery of initial membrane performance. The optimum combination of mechanical and chemical washing conditions resulted in dissolution of the binding organic matrix of the film, killing of bacteria and washing these components off the membrane surface.

However, if cleaning is not timely conducted or is not successful, the accumulating fouling film undergoes, with time, a progressive change in nature. It becomes increasingly more dense and sticky, more adherent to membrane surface, and of lower permeability. Such modifications may be attributed to several factors which are either physico-chemical like

film compaction due to thickening under pressure, or increase of hydrophobic character of the EPS of the microorganism upon interaction with membrane surface, and/or biological e.g. through the development of anaerobic bacterial reaction. The characterstic black coloration of the fouled membrane elements is thought to be due to the tendency of bacteria, at certain depth in the fouling film, to become anaerobic due to the lack of oxygen and the prevailing reducing conditions. SO_4^{2-} reducing bacteria lead to the production of the black ferric sulphide in the presence of ferric ions. Only limited reference is reported in the literature (Paul & Abanmy, 1990; Kaakinen & Moody, 1984) about such modification of the nature of fouling film.

3.4.4 Secondary scale deposition

The fouling film is assumed to act as a secondary membrane parallel to the original one. The fouled membrane, therefore, will have **a higher transmembrane pressure i.e. feed-permeate ΔP**, and in view of the partial blocking of the feed flow channels through the NF membrane element the fouled membranes will also have **higher transelement pressure, i.e. feed-brine ΔP**. The hydrodynamic conditions of the feed flow at the vicinity of the membrane surface would become further impaired if the fouling deposit causes blocking of the feed spacer in contact with membrane surface. This, together with the mentioned increase in ΔP values, would induce severe localized concentration polarization at the membrane surface (Khedr & Varoqui, 1981) with the consequent deposition of inorganic scale which we considered as (secondary scale deposition). In fact, in the advanced stages of membrane fouling, inorganic scale crystal aggregates were detected in the fouling film. **Again, this represents an example of interaction between the different forms of membrane fouling which further complicates the structure and composition of the fouling film and causes higher decline in NF performance.**

3.5 Control of NF membrane fouling

Results of Fig (11) show the decline of permeation rate under fouling conditions and the success of prompt cleaning to recover the initial performance (alkaline clean followed by acid clean) which was not the case with retarded cleanings Trails were conducted to control the membrane fouling in NF through:

3.5.1 Periodical backwash of membrane coupled with intermittent chlorination

In this case the choc dosing of chlorine for 5 minutes every 6 hours of nanofiltration together with periodical backwash of the membranes at a higher flow rate and a lower pressure could control the development of organic/biofouling as shown by Fig (11). Chlorination of feed stream is accompanied by dechlorination through dosing of a reducing agent (MBS) at a dose higher than stoichiometric for chlorine reduction in order to protect the polyamide thin film of membrane from destruction through oxidation/dissolution. Success of intermittent chlorination is usually attributed to (Baker & Dudley, 1998) either overcoming the increase in secretion of life products by microorganisms that takes place in presence of continuous dosing of chlorine, or to the decrease in availability of nutrients for bacteria through feeding on non-viable bacteria or on the products of decomposition by oxidation of complicated organics which otherwise cannot be digested by microorganisms.

However, chlorine-tolerant polymers have been prepared and adapted for the production of performant RO membranes (Nita et al, 1993; Glater et al, 1994). This will enable the introduction of residual chlorine in the feed stream up to membrane surface and killing of

bacteria without risk of membrane damage. The role of chlorination will also include oxidation and depolymerization of the EPS secreted by bacteria leading to the destruction of the fouling film matrix and consequently to its detachment by the cross flow stream.
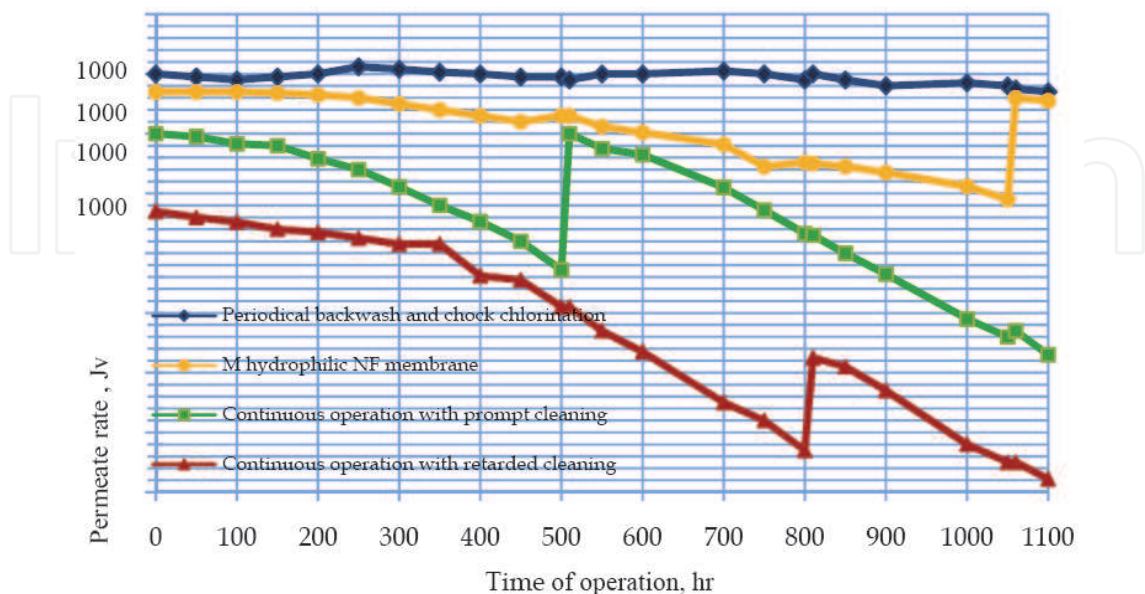

Fig. 11. Decline of product rate for various NF membranes upon fouling and remedy

3.5.2 Use the hydrophilic membranes

The use of the M membrane, based on modified PAN, previously supplied by Osmonics Corp. (currently under GE) which is characterized by high hydrophilicity, with the same fouling wastewater and under the same operation conditions, enabled to realize steady NF rate without decline of permeation and with largely spaced cleaning requirements as shown by Fig (11). Cleaning of hydrophilic membranes is shown to recover easily original performance.

When compared to conventional PAN membrane, the M membrane has a very low contact angle with water drop than that of other polymer membrane surface (Nicolaisen, 2002) i.e. much stronger hydrophilic character. This explains the much lower fouling rates observed with this membrane in view of the suppressed affinity towards adsorption of organics and colloids, including bacteria cells on the hydrophilic membrane surface. No sanitization was required for control of biofouling. In fact, much lower bacteria adhesion and biofouling were detected by SE microscopy and chemical analysis of film on M membrane surface.

3.5.3 Effect of high temperature operation on NF & LERO membrane fouling

In case of hot WW streams direct treatment by NF or RO without cooling is of both technical and economic interests. Beside acceleration of permeate rates at lower pressures i.e. saving of energy, HTNF/RO save installation of cooling facilities as well as their required chemical dosing and maintenance costs.

Thermal acceleration of Jv is attributed to the increase of water diffusivity and increase of efficiency of sweep of fouling film by the cross flow feed stream in view of decreased viscosity. Under these conditions our pervious results (Khedr & Quadri, 1997) have shown that the hydraulic permeability varies with temperature according to an Arrhenius type relation:

$$C_{HPT} = C_{HPO} \exp [- Eo / RT] \qquad (1)$$

Where the C_{HPO} and C_{HPT} are the membrane hydraulic permeabilities at initial temperature, and at temperature T, respectively.

E_o is the activation energy of the hydraulic permeability.

The product flux will vary because of these viscosity changes according to

$$J_{VT} = J_{V25}\, 1.03^{(t-25)} \qquad (2)$$

The temperature correction factor of the product rate at a given temperature "t" referred to the value at 25 ° C is

$$TFC = [J_{V25} / J_{VT}] = 1.03^{(25-t)} \qquad (3)$$

It is worthy to clarify here that the activation energy E_o, equation (1), has no kinetic implication. The Arrhenius equation models the change in hydraulic permeability with the change in temperature, however, it does not indicate that an initial energy barrier should be exceeded in order to activate a change in the hydraulic permeability.
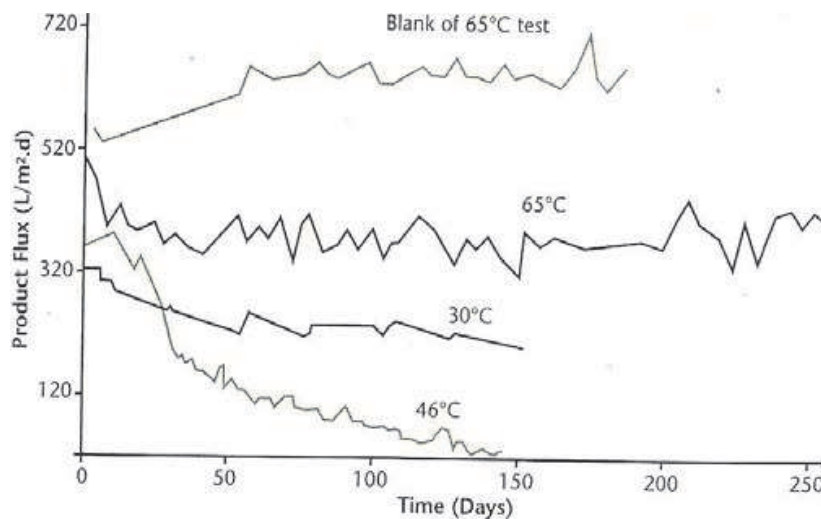

Fig. 12. Variation of permeate flux with time at various feed temperatures

It was observed that with mixed bio/organic fouling the acceleration of permeation rate upon rise of temperature (25 – 40° C) was leveled by the parallel acceleration of biofouling. At still higher temperature (45 -55 ˚ C) the influence of enhanced biofouling became predominant and progressive decline of permeation was obtained accompanied by formation of a thicker fouling film. However, at still higher temperatures, fig (12), 55 – 65 °C, the permeate flux remained steady as function of time at a value lower than that obtained in absence of fouling (blank line). In fact, at such temperatures bacteria cells are not killed and when temperature was lowered biofouling growth was reactivated. According to SE micrographs the minor film with minor bacteria adsorption and growth detected at such high temperatures suggests that bacterial growth and multiplication were just inhibited.

Increase of operation temperature, Fig (13), on the other hand, caused a rapid and rather linear decline of rejection of mono-monovalent salt while the di-divalent one showed a much higher rather steady rejection over a wide temperature range, followed by a slow decline. Rise of temperature enhances back diffusion of salt through membrane to the dilute side. This effect may be assisted, in part, by the viscous drag of salt ions due to interference with the accelerated product water flux, in the same direction, according to the friction

model of ion and water transport through selective membranes (Jansson & Boesen, 1975) It is evident that both factors i.e. back diffusion of ions and influence of viscous drag by the permeate flux exert higher effect on the lower rejection monovalent ions.

As for the rejection of organics investigated under exposure to UV radiation in order to avoid the contribution from biofouling, lower organic fouling was observed with rise of temperature in view of decreased adsorption of organics on membrane surface as well as lowered viscosity of the fouling film which facilitated its detachment by cross flow feed stream. TOC rejection showed a primary decline with temperature rise followed by increase of rejection at higher temperatures. Analysis of permeate and reject streams showed that the initial decline is mainly the case for low molecular weight organics, while the subsequent rise is only apparent due to the additional dilution of the permeate stream due to the enhanced water permeation.
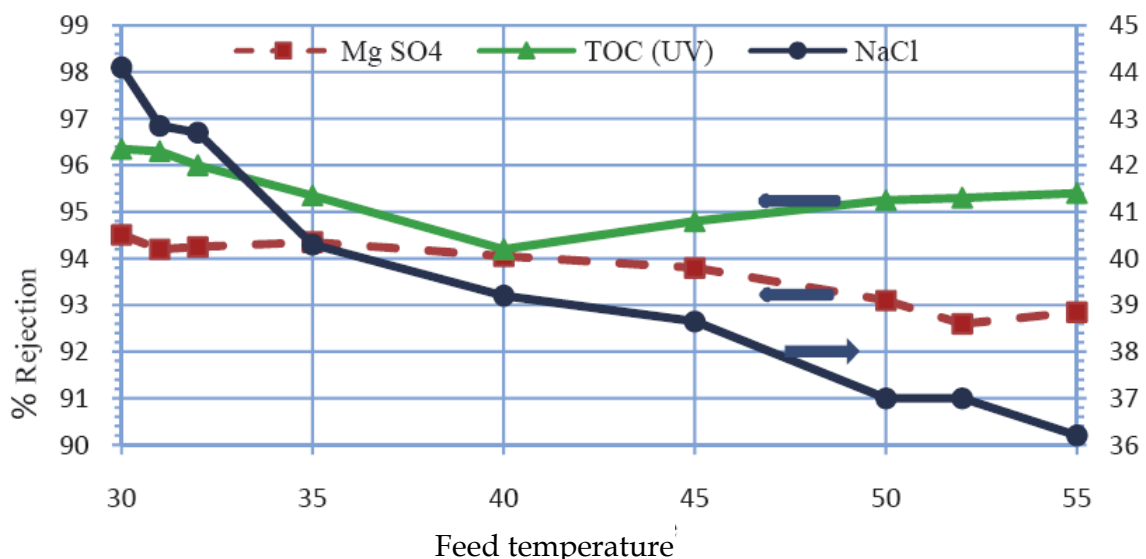

Fig. 13. Variation of NF rejection of various components with temperature

These results reveal the improvement of Nanofiltration and Low Energy Reverse Osmosis process efficiency for high temperature WW streams or groundwater through rise of permeation rate at lower pressure, better control of organic and biofouling, and increase of rejection of organics without significant loss of rejection of polyvalent contaminant ions (heavy metal cations, hardness components, and radioactive isotopes).

4. Conclusion

1. In view of the general advantages of NF & LERO of low energy consumption, cost effectiveness over a wide range of water salinities and plant capacities, and particularly their efficient rejection of wide variety of contaminants, it was decided to evaluate their performance in treatment of WW problems in comparison with the conventional methods applied in this respect. The general results of the long-term **investigation suggest that the low energy membrane processes are candidate to replace several conventional technologies of WW treatment.**

2. NF & LERO showed the following advantages for the separation of HMCs, hardness components, and radioactive isotopes from WWs and drinking water

sources over IER, chemical precipitation, softening and coagulation/ settling/ filtration methods:

2.1. Higher rejection of contaminants which include anions, cations and uncharged components with co-rejection of various trace contaminants without masking or interference.

2.2. No critical feed pH adjustment is required. Rejection is practically not pH dependent.

2.3. They are continuous processes and membranes do not require subsequent regeneration. Their mechanisms have no slow steps like settling.

2.4. They lead to general feed desalination and do not require subsequent filtration.

2.5. They do not require continuous chemical dosing stiochiometric to rejection of contaminants and do not result in formation of sludge which requires periodical disposal.

2.6. They do not require sophisticated installations or additional equipment to usual RO system.

2.7. They have a partial sterilizing effect.

3. The main factors that enabled to control the membrane fouling which is the main application problem of NF & LERO are:

3.1. Periodical backwash of membranes coupled with intermittent choc chlorination.

3.2. Hindrance of the adsorption of organics and microorganisms on membrane surface through promoting its hydrophilicity.

3.3. High temperature NF or LERO which accelerated permeate rate at lower pressure and enabled control of organic and biofouling without significant loss of polyvalent ion rejection (HMCs, hardness components and radioactive isotopes).

4. High temperature NF or LERO suggest the direct treatment of hot wastewater streams or groundwater in order to save the installation and operation costs of the cooling facilities. It also enabled lower energy consumption and higher plant availability through lowered shutdown for cleaning.

Knowledge of the wastewater composition would enable the selection of the adequate temperature range of HTNF for the optimum product rate and rejection performance.

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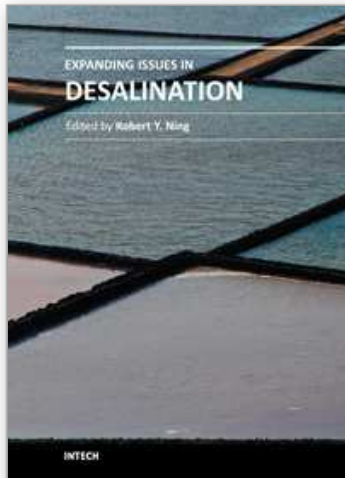
Expanding Issues in Desalination

Edited by Prof. Robert Y. Ning

For this book, the term "desalination" is used in the broadest sense of the removal of dissolved, suspended, visible and invisible impurities in seawater, brackish water and wastewater, to make them drinkable, or pure enough for industrial applications like in the processes for the production of steam, power, pharmaceuticals and microelectronics, or simply for discharge back into the environment. This book is a companion volume to "Desalination, Trends and Technologies", INTECH, 2011, expanding on the extension of seawater desalination to brackish and wastewater desalination applications, and associated technical issues. For students and workers in the field of desalination, this book provides a summary of key concepts and keywords with which detailed information may be gathered through internet search engines. Papers and reviews collected in this volume covers the spectrum of topics on the desalination of water, too broad to delve into in depth. The literature citations in these papers serve to fill in gaps in the coverage of this book. Contributions to the knowledge-base of desalination is expected to continue to grow exponentially in the coming years.

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