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The Role of Coagulation and Microfiltration in Seawater Pre-treatment

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Abstract

Power industry needs make-up water in power plant processes to compensate for the constant water loss during work. Mediterranean Seawater samples are studied to obtain pretreated water to use as a feed for the desalination stage. A comparison of coagulation with two coagulants, namely Poly aluminum chloride and ferric chloride in a one wt% stock solution, followed by microfiltration, was evaluated to obtain an efficient pre-treatment method. The lowest dosage of PACl positively affected all the measured parameters, especially the total suspended solid content was reduced to below 10 mg/L. The addition of $FeCl_3$ resulted in 2.5-times higher total suspended solid content (23.5 ± 4.58 mg/L) than the initial value measured for seawater (10.8 ± 1.03 mg/L). Considering the flux values, two-steps of microfiltration and the lowest dosage of PACl followed by microfiltration resulted in the same high flux (3500 L/(m^2 h) at the initial stage and 2500 L/(m^2 h) at the 90% recovery rate). The flux after $FeCl_3$ dosage seemed to be the highest, but it should be emphasized that the flocs were filtered via two consecutive 5–13 µm microfiltration stages, so this flux is not entirely comparable with those measured in other cases. Considering the chloride concentration, after microfiltration without coagulation, it remained constant; using coagulants it showed a slight reduction (~4%). From environmental point of view, the two-steps of microfiltration is recommended to be used since no chemicals are required for the operation; it can provide a steady flux of the cleanest pretreated water based on total suspended solid content.

Keywords

water treatment, make-up water, microfiltration, coagulation, pre-treatment technology

1 Introduction

Due to the limitation of the alternative sources of water needed in power generation, researchers agreed to give more interest to the use of non-conventional water sources [1]. Seawater (SW) characteristics are very complex; it comprises a variety of foulants, including suspended particles, colloids, and a variety of organic debris, in addition to dissolved salts. These contaminants can degrade membrane performance in the desalination step [2]. Therefore, it requires more thorough pre-treatment procedures [1]. The primary goal of the pre-treatment stage is to reduce the levels of total dissolved solids, silt density index, and turbidity of the feed water to have an efficient process with a high-quality permeate that meets the acceptable level of the feed for the following treatment step [3]. Water quality requirements are stringent; therefore, the water treatment process for thermal power plants is very significant in meeting these requirements, especially from an environmental point of view [4, 5]. Environmental trouble is associated with the chemicals used in the pre-treatment stage and the others used for membrane cleaning. This issue can be reduced or at least controlled by using membrane-based pre-treatment technologies (membrane filtration technique) rather than the conventional techniques such as coagulation and flocculation [6]. Membrane filtration techniques are used in process- [7], drinking- [8], and wastewater treatment [9]. Pre-treatment with low-pressure membranes, particularly microfiltration (MF), has become increasingly popular and established its efficacy in the pre-treatment step, creating high-quality permeate with a more steady flux and high resistance to fouling, making it appropriate for the subsequent desalination step [10]. The disadvantages of the conventional treatment process are that it is sensitive to changes in source water characteristics and

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requires different dosages of chemicals [3]. Membrane-based technology, particularly MF, exhibits many advantages over coagulation-flocculation, such as high permeate flux. The permeate flux continuously decreases with filtration time; this flux decline is related to the pores clogging and plugging caused by the fouling phenomenon. This problem can be controlled by controlling the pre-treatment step correctly [11]. Coagulation for seawater blended with brackish water was studied by Park and coworkers, and the appropriate dosage of coagulants was tested. PACl and $FeCl_3$ were compared to determine the most effective one with an optimum dosage. It was found that PACl was effective at a dosage between 20–30 mg/L compared to $FeCl_3$ with 30 mg/L to remove total organic carbon (TOC), turbidity, and dissolved organic carbon (DOC). PACl was better regarding the costs and low chemical dosages [12]. Febrina and Mesra studied the seawater pre-treatment by coagulation and determined the optimum dosage for the used coagulants. They found that 70 mg/L of PACl was appropriate at pH 6.9 [13]. Al-Mashharawi and coworkers reported the effect of $FeCl_3$ dosages on the filtration step. The permeate flux was more stable as the coagulant concentration increased while the pressure was constant over the process. Although the high concentration of coagulants increased the flow rate, less chemical usage during the coagulation step is critical from an environmental and economic standpoint. Therefore, coagulants with low concentrations to create high-quality permeate and control membrane fouling is a viable option, especially for membranes with tiny pores [10]. According to Wilf and Bartels, Al- and Fe-salts are probably the most commonly used coagulants in the pre-treatment step of seawater. They concluded that aluminium is not preferable for seawater prior to membrane filtration due to damage to the membrane in the desalination system [1, 14]. Edzwald and Haarhoff studied PACl as a coagulant used in seawater pre-treatment and tested it in the laboratory, and they found that it is not preferable in full-scale plants. Due to aluminium-based coagulants having a strong charge opposite to that of the membrane surface and the relatively high solubility of Al, it can be concentrated, producing aluminium hydroxide and aluminium silicate solids, causing precipitate scaling for the membrane in the following desalination step. Consequently, $FeCl_3$ is the most preferable for coagulation in the case of salty water treatment [15, 16]. Yang and Kim had evaluated the effect of coagulation on the performance of MF for the removal of particles under various coagulant dosages and pH. They obtained that the flux drop throughout the membrane declined after adding the coagulation before MF, compared to membrane filtration alone. In the case of choosing the optimal dosage of the used coagulant, coagulation with further MF can be considered an effective pre-treatment method in seawater desalination with higher permeate quality compared to MF alone [17]. This study compares the microfiltration and the coagulation technologies as a pre-treatment step for the seawater and their effectiveness in producing pure water based on the total suspended solids content (TSS) and chloride concentration measurements. In the case of using $FeCl_3$ and PACl as coagulants, it is worth measuring chloride concentration to monitor the coagulant efficiency and their load on the environment by increasing the chloride content of the feed water. Previous works have not dealt with the consequences of chloride and just focused on the metals as a coagulant. Therefore, experiments for the pre-treatment stage will be conducted on a laboratory scale to figure out the most appropriate technology to obtain pure water with less harming the desalination membrane. During the experiments, the efficiency of the two stages of microfiltration working alone without coagulation will be examined to check if it can be considered a sustainable and clean technology. Meanwhile, the effects of coagulants on the MF membrane will be studied to determine whether this addition will improve the treatment efficiency. The optimum dosage of the coagulants will be determined depending on the previous literature and our experiments.

2 Materials and methods

Microfiltration experiments were performed on a universal bench-scale membrane filtration apparatus in cross-flow mode. Transmembrane pressure (over pressure) of the first microfiltration step was 0.407 bar for (5–13) µm particle retention membrane and 2 bar for the 0.45 µm pore-size membrane, respectively. According to the manual, pump delivery rate is 1.81 L/min at 8 bar. Membrane with particle retention (5–13) µm was used for the first microfiltration step, and a 0.45 µm pore-size membrane as a primary filtration step. The effective area of the membranes were 28 cm^2. Mediterranean seawater was used as a salty water source; samples were collected from nearby locations (Isola, Slovenia) on 21st July 2021. Water samples were stored at room temperature (~25 °C) before use. The parameters of seawater are summarized in Table 1. A series of flat sheet microfiltration membranes were purchased from VWR Hungary Company, comprising two representative membrane pores; in decreasing size, these were (5–13) µm and 0.45 µm, correspondingly. Qualitative filter paper no. 413 with particle retention of (5–13) µm was used as the first microfiltration step. Supor®-450 trademark membrane with a pore-size of 0.45 µm was used as the primary microfiltration step. The experimental plan is illustrated in Fig. 1. Depending on the pre-treatment method and the number of samples to be analyzed, the feed volume of 0.45 µm MF step varied between 122 mL and 246 mL (Table 2). Coagulation experiments were conducted with two types of coagulants, purchased from VWR Hungary Company, PACl (purchased as $Al_2Cl(OH)_5$ (M= 174.45 g/mol)) and $FeCl_3$ (purchased as $FeCl_3 \times 6H_2O$ (M= 270.3 g/mol)). One wt% stock solutions of these chemicals were prepared and added to 30 mL of seawater samples in different dosages. Quick stirring (400 rpm) was applied for 30 seconds after adding coagulant in the jar test method. After that, stirring was stopped to provide flocs formation. Water characteristics, such as pH and specific electric conductivity (later conductivity) K, were measured with a 340I type WTW combined pH/conductivity meter. TSS content in [mg/L] was measured by a portable UV analyzer (PASTEL-UV). Chloride concentration was measured according to the classical titration method; $AgNO_3$ was used as a reagent. All samples were measured five times, and the average values and standard deviations were calculated.

Table 1 Parameters of the seawater sample

Seawater characteristics	Value
pH	7.18 at 26.1 °C
Chloride concentration [g/L]	19.53 ± 0.37
TSS [mg/L]	10.80 ± 1.03
K [mS/cm]	52.60 at 25.4 °C

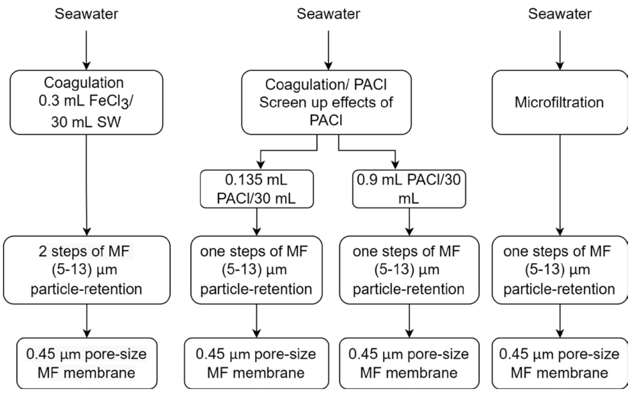

Fig. 1 Outlines of the experiments

Table 2 Experiments' description

Experiment	0.45 µm pore-size membrane MF Feed [mL]
Seawater + MF (1st batch)	230
Seawater + MF (2nd batch)	200
Seawater + MF (3rd batch)	246
Seawater + $FeCl_3$ (0.3 mL) + MF	122
Seawater + PACl (0.135 mL) + MF	148
Seawater + PACl (0.9 mL) + MF	150

3 Results and discussion

Conventional and membrane-based pre-treatment have been compared based on the permeate quality regarding TSS content, conductivity, pH, and chloride ion concentration.

3.1 Effect of two-steps microfiltration on the seawater pre-treatment process

Three rounds of MF (5–13 µm) were performed initially to remove the suspended solids from the seawater in order to avoid the fouling of the 0.45 µm MF. Residues measured are given in Table 3.

Firstly, the voidage was measured before starting the MF rounds to provide a proper mass balance. Then, around 250 mL of seawater in each batch was filtered, and flux was calculated based on Eq. (1):

$$J_v = \frac{1}{A} \times \frac{dV}{dt} \qquad \left[\frac{L}{m^2\ h}\right], \tag{1}$$

where:

- A: membrane surface [m^2],
- dV/dt: Flow rate [L/h].

According to the results, a stable flux could be reached, and experiments ended at a recovery rate of 85–98% (the volume was gained as permeate). Recovery rate is defined as a ration of the permeate volume and feed volume (Vp/Vf).

To check reproducibility, parallel experiments were carried out and showed similar values and the same trend; a considerable decline during the first 20% of the recovery rate was observed, followed by a moderate slope till the end of the experiments as expected for batch experiments (Fig. 2). Mass balance was calculated, and the

Table 3 Residues from 500 mL SW after the first step of MF

MF membrane	Mass [g]
Clean membrane	0.4718
Membrane after MF (5–13 µm)	0.5339
Residues	0.0621 (i.e., 0.01%)

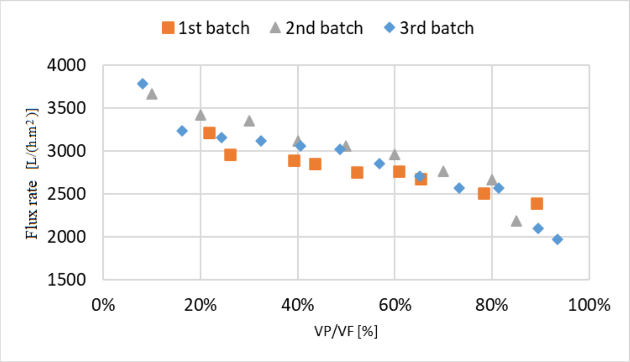

Fig. 2 The permeate flux through the 0.45 pore-size MF membrane

average error was 1.02%, an acceptable value (< 2%). Due to the variation of the feed volume (between 200–246 mL) and the requirements of the circulation pump, the recovery rate was between 85–95%. Typically, in the case of MF, the typical recovery rate must be kept within 90–95%. After two steps of MF, the parameters of filtered water were measured and summarized in Table 4.

3.2 Effect of coagulation on the seawater pre-treatment process

The coagulation process was conducted with two types of coagulants, PACl and $FeCl_3$, in the form of stock solutions of 1 wt%. First, five initial dosages of PACl were tested separately to reach a coagulant concentration range (7–100 mg/L). Based on the TSS measurements, the higher the dosage higher the TSS content and the turbidity in the treated water. By visual evaluation, the 0.2 mL dosage of PACl was efficient in forming flocs. In the case of blended seawater (seawater+ brackish water), the addition of 20–30 mg/L PACl was proven to successfully remove TOC and lower the turbidity [12]. Our results for seawater experiments show that the concentration recommended by Park and colleague's study [12] was not efficient, higher dosage should be used to form visible flocs. Thus, samples from all dosages were summed up into one flask, and the average sample was evaluated (Table 5).

In the case of freshwater, the minimum dosage of PACl and $FeCl_3$ was 3.2 mL and 2.3 mL, respectively, when using a stock solution of 1 wt% as a coagulant. Depending on these results, higher dosages were also tested for seawater experiments, such as 0.135, 0.9, and 2.1 mL of PACl, referred to as 44.7, 300, and 700 mg/L. It was also demonstrated that PACl was a more efficient coagulant than $FeCl_3$; thus, in the latter case, the addition of coagulant was continued from drop to drop until reaching visible flocs. It was obtained that a 0.3 mL dosage of stock solution was sufficient for 30 mL of SW, see Fig. 3.

Table 4 Parameters of the purified water after microfiltration

SW + MF	K [mS/cm]	T [°C]	C_{Cl^-} [g/l]	TSS [mg/l]
Average of 3 batches	50.77	28.07	19.52±0.73	<10.00

Table 5 TSS content after coagulation with different dosages of PACl

PACl dosage [mL]	PACl concentration [mg/L]	TSS [mg/L]
0.023	7.64	< 10.00
0.050	16.62	< 10.00
0.100	33.23	< 10.00
0.200	66.47	12.00
0.300	99.70	12.50
The mixture (average) 0.135	44.73	14.80

TSS content, conductivity, and pH were measured after the coagulation processes and summarized in Table 6.

In the case of adding a high dosage of PACl, flocs were visible in the sample. The turbidity was increased, along with the increased TSS content. These flocs could capture some parts of the seawater, which lowered the overall conductivity, but it was not a significant change. Additional PACl, such as 2.1 mL, can adsorb the hydroxide ions, and when H^+ and OH^- ions are imbalanced, the solution is shifted to the acidic range, resulting in lower pH. However, a high dosage of PACl decreased the conductivity; it increased the TSS and the turbidity. Thus, it is not recommended to use high dosages of PACl in agreement with the reference [12].

Fig. 3 Coagulation process; 1) 0.3 mL $FeCl_3$; 2) 0.135 mL PACl; 3) 0.9 mL PACl, 4) 2.1 mL PACl

Table 6 Parameters of the purified water after coagulation at temperature range between 25–26 °C

Batch	TSS [mg/L]	K [mS/cm]	pH
0.3 mL $FeCl_3$	71.00 ± 5.16	53.12	6.53
0.135 mL PACl	14.80 ± 3.42	53.51	7.76
0.9 mL PACl	37.50 ± 5.32	52.64	6.87
2.1 mL PACl	64.00 ± 7.87	50.68	4.77

As the dosage increased (from 0.135 mL to 2.1 mL), TSS increased (12–60 mg/L), and pH declined (7.7–4.7), but it did not affect the conductivity, see Fig. 4. Referring to Febrina and Mesra recommended value, i.e., 70 mg/L concentration of PACl at a pH of 6.9 for seawater [13], it should be emphasized that coagulant addition influences the pH. As the 0.2- and 0.3-mL dosages (66–100 mg/L) were tested, the pH was lowered with increasing dosages while the TSS content was increased. According to the results, lower dosages of PACl are preferable, and even coagulation could be replaced by MF. After coagulation with PACl, particles formation was not observed; no further filtration step was required prior 0.45 μm MF step. Big flocs were noticeable after the coagulation with $FeCl_3$, which might harm the 0.45 μm pore-size membrane in the following filtration step. Therefore, another microfiltration step was applied using a 5–13 μm particle retention membrane; these membranes are recommended for particles filtration. Due to the flocs, the membrane was fouled; thus, it must have been replaced several times; altogether, four pieces of 5–13 μm particle retention MF membrane were used as a pre-filtration step. Then permeate was collected in the same flask and stored at 26 °C to check further flocs' formation. During the storage, visible flocs formed again; thus, a second filtration step via the same type of membrane must have been inserted to have a particle-free suitable permeate as a feed of 0.45 μm pore-size MF membrane, see Fig. 5.

Some improvement has been observed concerning TSS content after using two consecutive steps of 5–13 μm particle retention MF membrane after coagulation, see Table 7.

3.3 Coagulation with further microfiltration

The influence of coagulant dosage on the membrane filtration stage was studied. As illustrated in Fig. 6, the flux after $FeCl_3$ dosage seems to be the highest, but it should be emphasized that the flocs were filtered via two 5–13 μm MF stages, so this flux is not entirely comparable with those measured in other cases.

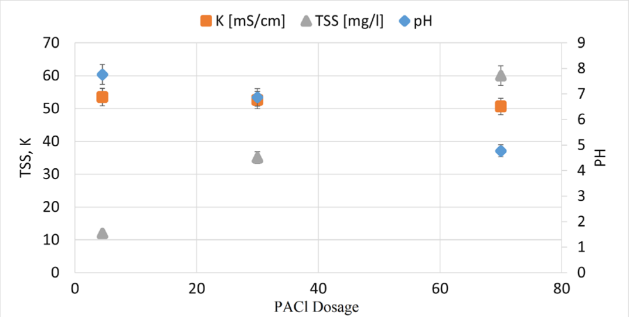

Fig. 4 Permeate parameters after the coagulation step using PACl as a coagulant

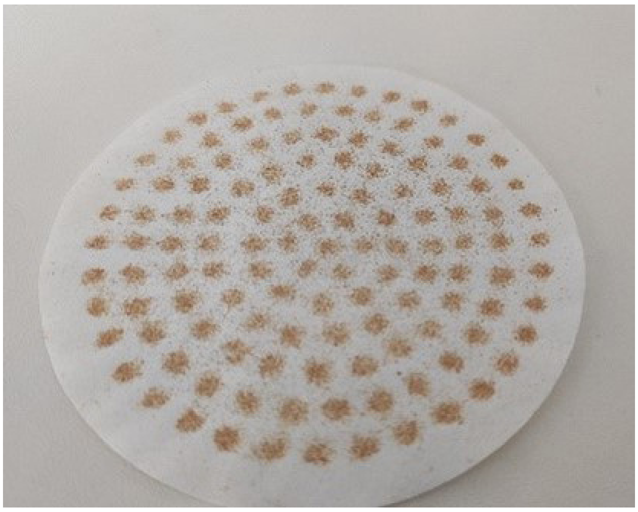

Fig. 5 5–13 μm particle retention MF membrane after two filtration steps of SW + coagulation with $FeCl_3$

Table 7 Parameters after coagulation (with $FeCl_3$) + (5–13) μm MF

Batch	TSS [mg/L]	K [mS/cm]	pH
0.3 mL $FeCl_3$+2 steps of (5–13) μm MF	43.00	52.50	7.74

A high dosage of $FeCl_3$ results in huge flocs, which generally would settle. Still, 5–13 μm particle retention MF membrane offers a time-saving solution for their removal instead of gravity settling on a laboratory scale. It can be seen that the higher dosage of PACl (i.e., 0.9 mL) resulted in lower flux, about 500 L/(m² h) lower than the flux obtained for the lower dosage (i.e., 0.135 mL). Using MF only (5–13 μm MF followed by 0.45 μm MF), comparable flux values could be reached to those obtained at 0.135 mL PACl which were, on the other hand, higher than in case the of high dosage of PACl. Mass balance calculations were

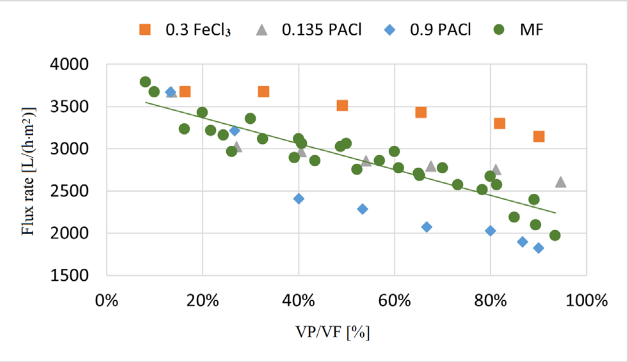

Fig. 6 Flux through the 0.45 μm pore-size MF membrane after adding coagulation prior to MF (green line represents the average of three replicates MF)

carried out; the error varied within 2–10%, indicating the significance of the pipes' volume of the test equipment.

3.4 Comparison of the permeate of different pre-treatment technologies

Previous works studied the effects of coagulation on turbidity and the SDI. According to this study, it was found that TSS is similarly important to turbidity, but it has not been measured before. TSS can provide a prognosis for the further desalination step. Characterizations of permeate were performed, including TSS content, conductivity, pH, and chloride concentration. Regarding the coagulation with PACl, it is noticeable that the high dosage of the coagulant negatively affected the parameters; TSS slightly increased, and pH decreased. Therefore, coagulation with a low dosage of PACl is preferable. The addition of $FeCl_3$ resulted in 2.5-times higher TSS (23.5 ± 4.58 mg/L) than the initial value measured for SW, see Fig. 7. This coagulant made the solution more acidic, but the pH returned to the standard value by filtration. Because the temperature influences both K and pH, and due to the differences in the outside temperature (the temperature of the samples varied between 25–26 °C), a clear conclusion cannot be made on the effect of the coagulant on K and pH. Based on freshwater experiments, coagulation with PACl is suitable in the pre-treatment step regarding the TSS content in permeate.

Meanwhile, there is no need for a coagulation step in seawater experiments because there is no improvement in the TSS. Therefore, two steps of MF without coagulation seem to be sufficient as a pre-treatment step for SW. Considering the chloride concentration, after microfiltration without coagulation, it remained constant; its value (19.517 ± 0.73) g/L was almost the same as measured for SW (19.534 ± 0.37) mg/L (Table 8).

4 Conclusion

Usually, the coagulation-flocculation process results in a large amount of used chemicals and residuals, requiring a large tank for the sedimentation step. Meanwhile, there is no need for chemicals during the membrane treatment technology, limiting the environmental effect. Therefore, when the pre-treatment step operates appropriately, and the water is pretreated well, it can be considered as an efficient, economical, and environment-friendly technology. During this research, the main focus was on microfiltration as an environmentally benign separation technique, whether it can replace the conventional coagulation pre-treatment methods with high chemical consumption, to prepare the appropriate make-up water treatment of thermal power plants. Since both TSS and chloride concentration are suitable parameters helping to choose the best pre-treatment technology, these parameters were the

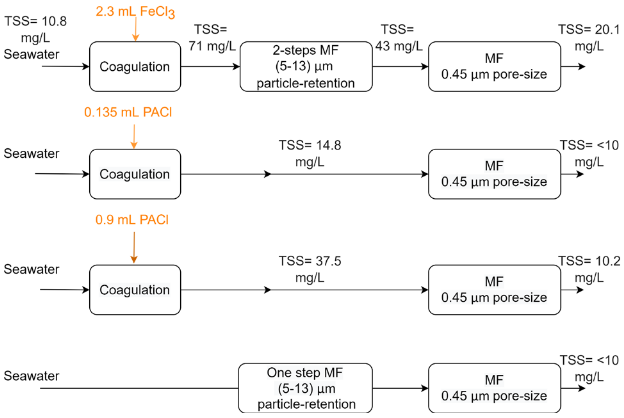

Fig. 7 Comparison between pre-treatment technologies at a temperature range between 25–26 °C

Table 8 Comparison between pre-treatment technologies at temperature range between 25–26 °C

Sample/Parameters	TSS [mg/l]	K [mS/cm]	pH	C_{Cl^-} [g/l]
SW	10.80±1.03	52.60	7.18	19.534 ± 0.372
SW+ MF	< 10.00	50.77	8.00	19.517 ± 0.729
SW+ $FeCl_3$ + 2 steps MF	23.50±4.58	52.81	7.74	18.671 ± 0.087
SW + 0.135 PACl + MF	< 10.00	52.94	8.01	18.873 ± 0.350
SW + 0.9 PACl + MF	10.20±2.55	52.60	7.50	18.772 ± 0.151

basis of the experiments. Based on the experiments conducted so far, it was figured out membrane technology can also protect membranes used in desalination procedures from fouling, extending their lifetime. Although coagulation may be needed when the seawater contains very high levels of natural organic matter, microfiltration seems to be a desirable pre-treatment technique increasingly used in seawater applications because it can remove particles as fine as 0.2 µm without coagulation. Also, MF can be considered a suitable pre-treatment technology for seawater regarding the excellent quality and quantity of permeate.

Moreover, using MF alone as a pre-treatment step requires no chemicals in the water treatment process and does not influence the water's chloride concentration compared to coagulation with PACl or $FeCl_3$. Therefore, MF can lower the TSS content without changing the ion content of water and can replace the chemical reaction with coagulants for seawater pre-treatment. Two steps of MF without coagulation seem to be sufficient as a pre-treatment step for SW. Considering the chloride concentration, after microfiltration without coagulation, it remained constant; its value (19.517 ± 0.73) g/L was almost the same as measured for SW (19.534 ± 0.37) mg/L. Although 0.135 mL dosage, i.e., 45 mg/L concentration of PACl, seems to provide less than 10 mg/L TSS content in the permeate, this coagulation step can be replaced by a 5–13 µm particle retention MF membrane resulting in the same TSS content (< 10 mg/L) without chemical usage in the following desalination step. Thus, it was proven that two stages of microfiltration as a benign environmental step could replace coagulation.

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