WASTEWATER TREATMENT BY ELECTRODIALYSIS SYSTEM AND FOULING PROBLEMS

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Abstract: Electrodialysis ED is a separation process commercially used on a large scale for production of drinking water from water bodies and treatment of industrial effluents (Ruiz and et al., 2007). ED system contains ion exchange membranes and ions are transported through ion selective membranes from one solution to another under the influence of electrical potential difference used as a driving force. ED has been widely used in the desalination process and recovery of useful matters from effluents. The performance of ED, depends on the operating conditions and device structures such as ion content of raw water, current density, flow rate, membrane properties, feed concentration, geometry of cell compartments (Chang and et al., 2009, Mohammadi and et al., 2004).

The efficiency of ED systems consist in a large part on the properties of the ion exchange membranes. Fouling of ion exchange membranes is one of the common problems in ED processes (Lee and et al., 2009, Ruiz and et al., 2007). Fouling is basically caused by the precipitation of foulants such as organics, colloids and biomass on the membrane surface or inside the membrane and fouling problem reduces the transport of ions. The fouling problems are occasion to increase membrane resistance, loss in selectivity of the membranes and affect negatively to membrane performance (Lee and et al., 2002, Lindstrand and et al., 2000a, Lindstrand and et al., 2000b).

Membrane fouling leads to high energy consumption and poor separation efficiency. Therefore some methods such as pretreatment of the feed solution, turbulence in the compartments, zeta potential control, pH and flow rate optimization, modification of the membrane properties and pulsed voltage have been studied to reduce fouling in ED systems. Also some reducing methods such as chemical methods require additional chemicals, more energy or instruments and those requirements increase the operation and investment cost (Chang and et al., 2009, Lee and et al., 2002, Park and et al., 2003). The fouling problem has been disqualified also by electrodialysis reversal systems EDR. EDR is similar to ED but the direction of ion flow is reversed periodically to break fouling progress by reversing the polarity of the applied electric current. This process is not use only for the removal of fouling but also for removing salts in desalination systems. EDR does not require additional chemicals and it increases membrane life (Bouhidel and Rumeau, 2004, Strathmann, 2010).

Fouling of membranes is one of the major problems in ED systems. For this reason there is a need to understand the fouling factors and fouling formation. This study includes brief introduction about ED systems, a literature review of the different kinds of fouling mechanisms, fouling mitigation techniques, cleaning methods to enhance the ED efficiency and advantages /disadvantages of those cleaning methods. This paper concludes with an outlook to future ED system studies.

Key Words: Electrodialysis, Electrodialysis Reversal, Fouling, Fouling Mitigation, Wastewater.

Introduction

Recycling and reuse of wastewater after the convenient treatment will reduce the use of natural water sources. Also this method provide to waste reduction. Thus a variety of treatment methods have been investigated, such as adsorption, flotation, chemical coagulation, biological degradation, electrochemical methods, and membrane filtration (Ashrafi and et al., 2015). The membrane is an interface that has selectively between in two phases. Membranes can be made from inorganic, organic and metal polymer and they are used for gas separation solid - liquid and liquid- liquid separation. Separation is depending on the membrane pore size [Figure 1].
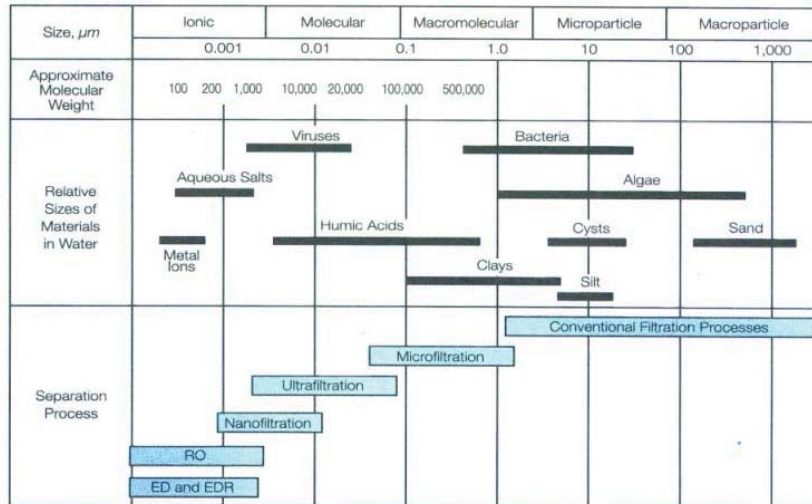
Figure 1. Membrane Processes (AWWA, 1995).

Electrodialysis (ED) is an alternative membran separation process that used in wastewater treatment. The ion exchange membranes are arranged between the anode and cathode in ED system. Ionic compounds that in the feed water, starts to move across ion exchange membranes using an electrical driving force. The cations in the solution migrate toward to the cathode and the anions migrate toward to the anode by applying electrical current between the anode and cathode. Cations pass through the cation exchange membrane but they are retained by the anion exchange membrane. Similarly, anions pass through the anion exchange membrane but they are retained by the cation exchange membrane [Figure 2]. The overall result of the process, concentrated and diluted compartments occurs in the system. The electrolyte solution used to ensure the conductivity and remove gases produced by electrode reactions in the system. The degree of electrodialysis depends on (Caprarescu and et al., 2012, Mohammadi and et al., 2004);

- Ion exchange membrane structure,
- Feed water ion concentration,
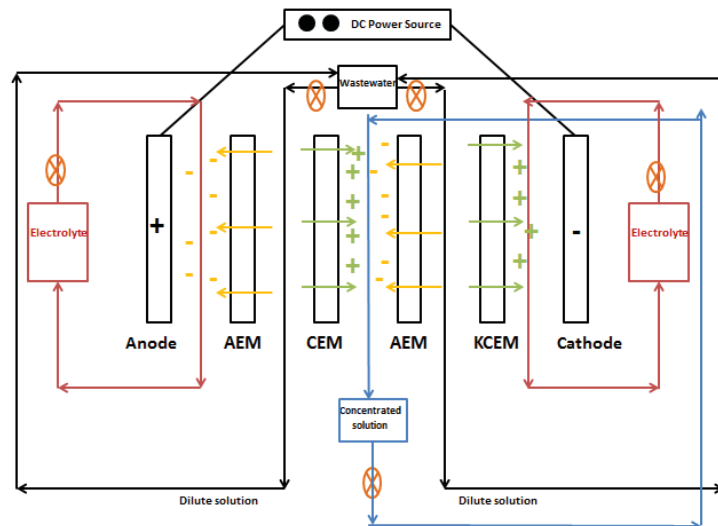- Current density
- pH
- Flow rate
- ED cell structure

Figure 2. Schematic diagram of ED system (AEM: Anion exchange membrane, CEM: Cation exchange membrane)

The most important limiting factor in electrodialysis system is fouling such as other membrane systems. Particles that larger than the membrane pore size cannot pass through the membrane in solution and causing deposition on the membrane surface. Accumulation of insoluble biomass, colloid and organic substances inside membrane or on the membrane surface is called as fouling. Fouling affects to membrane performance adversely by flux drop or increase in the electric resistance. Most of works on membrane fouling have focused on anion exchange membranes (Ruiz and et al.). Because of studies carried out that anionic membranes were more affected than cationic membranes, since most colloids present in natural water are negatively charged (Araya-Farias and Bazinet, 2006a, Ayala-Bribiesca and et al., 2006, Lee and et al., 2002).

In this research working principle of the electrodialysis system is explained. Also fouling mechanism of ED ion exchange membranes and its control methods investigated and provided information on the fouling problems. In this context this study includes a literature about fouling mechanisms, causes and removal methods.

Operating Principles of ED System

Electrodialysis consists of the combination of electrolysis and dialysis methods. This method was used by Maigrot and Sabates in 1890 for the first time. The aim of their study was demineralize of sugar syrup. The first ED system is illustrated in figure 3. The frames of ED system were made of wood, electrodes were made of carbon and membranes were made of permanganate paper. Electrical current is provided from the dynamo. The middle electrode is used as anode. The syrup was poured through the anodic compartment. Potassium, sodium, magnesium and calcium cations were migrate from the anodic compartment to the cathodic compartment by electrical current. At the end of the process the syrup has been demineralized in the anodic compartment (Shaposhnik and Kesore, 1997).
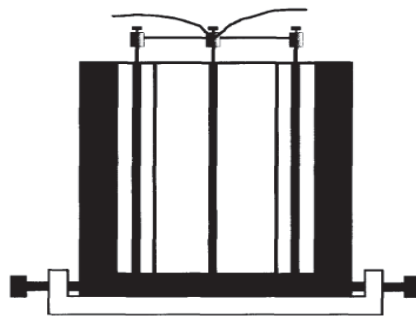

Figure 3. Scheme of the first electrodialysis system (Shaposhnik and Kesore, 1997).

ED is an electromembrane separation process that ions are transferred through ion exchange membranes under electrical current (DC voltage). When electrical current is applied (Murray and et al., 1995);

- Cations migrate to the cathode, or negative electrode.
- Anions migrate to the anode, or positive electrode.
- Water molecules break down and produce two hydroxyl ions and hydrogen gas at the cathode.
- Water molecules dissociate to four hydrogen ions, one molecule of oxygen, and four electrons at the anode
- Chlorine gas may be formed at the anode.

Electrodialysis (ED) processes are used on a large scale in commercially. ED systems, corresponding processes and their applications are summarized in table 1. ED has been widely used to demineralize, concentrate and/or convert salt-containing solutions. ED contains ion exchange membranes that ions are transported from one solution through ion selective membranes to another solution under influence of an applied electric potential difference. Performance of the system depends on operating conditions and ED cell structure (Chang and et al., 2009, Ruiz and et al., 2007).

Table 1. ED system, related processes and applications (Strathmann, 2010).

PROCESSES	APPLICATIONS
Electrodialysis	Water desalination, salt pre-concentration
Diffusion dialysis	Acid and base recovery
Donnan dialysis	Water softening, ion exchange
Bipolar membrane ED	Production of acid and base from corresponding salts
Continuous electrodeionization	Ultra-pure water production
Capacitive deionization	Water desalination, water softening
Reverse ED	Electrodialytic energy generation

Caprarescu and et al. (2012) were study, the removal of copper ions by ED system. They used two different ion exchange membranes and determined initial copper ions concentration, solutions conductivity, pH, flow rate. When applied voltage that during the ED was 7.5 V and initial copper concentration was 4 g/L, the extraction percentage of copper ions increased >85% (Caprarescu and et al., 2012).

Mohammadi and et al. (2004) studied about effects of flow rate, temperature and voltage on lead removal by ED. Their results showed that increasing voltage and temperature improved cell performance; however, the separation percentage decreased with an increasing flow rate.

Dissolved solids are selectively removed by semipermeable ion exchange membranes with their electrical charge in ED systems. The ion exchange membranes used as either anion transfer or cation transfer membranes in ED systems. Each membrane is 0.1 to 0.6 mm thick and it can be homogeneous or heterogeneous. Membranes are produced by blending an ion-exchanging resin with a polymer. The ion exchanger must involve at least half the polymer mixture (AWWA, 1995). Anion exchange membranes used in ED processes and allow only negatively charged ions to pass through. Cation exchange membranes are also used in ED processes and allow only positively charged ions to pass through. This membrane allows the transition of cations while blocking the passage of anions. Both anion and cation exchange membranes have the following qualification (Murray and et al., 1995);

- Semi-rigidity for ease of handling
- Low electrical resistance
- Insolubility in aqueous solutions
- Availability to use above 46°C
- Availability in different sizes and configurations
- Long life expectancy
- Resistance to change in pH from 2 to 9

ED plants can be planned to remove from 50% to 99% of source water pollutants or dissolved solids. When source water salinity content is less than 100 mg/L up to 12,000 mg/L, TDS can be successfully treated by ED to produce finished water of less than 10 mg/L TDS (AWWA, 1995). The system is commonly used by applications of waste treatment such as; removal of hazardous chemical, desalination of salt water, recycling and reuse of valuable compounds, whey demineralization, developing new production methods that require lower energy consumption, pharmaceutical application, recovery of pickling acid, recycling of rinsing solution (Koter and Warszawski, 2000). The main advantages of the ED system are; separation without phase changes, not additional chemicals, high permeability selectivity, low electrical resistance, good mechanical stability, high chemical stability (Nagarale and et al., 2006). One of the most important substances that affect negatively the performance of ED system is fouling of ion-exchange membranes. Industrial applications of ED and key problems are listed in table 2.

Table 2. Industrial applications and key problems of ED (Strathmann, 2010).

APPLICATIONS	STATUS of APPLICATION	KEY PROBLEMS
Desalination	Commercial	Costs
Process water treatment	Commercial	Costs
Waste treatment	Commercial	Membrane Fouling
Demineralization of food products	Commercial and pilot plants	Membrane Fouling
Table salt production	Commercial	Membrane Fouling

Membrane Fouling of ED Systems

The membrane recovery rate can be limited by the presence of certain salts that can form a foulant on the membrane surface. High concentrations of salts increase fouling potential in ED systems. The transport of ions increases through ion exchange membranes by applying electric current, and precipitation occurrence on the selective membrane surface in time. Accumulation increase on the membrane surface with time and it becomes gel layer. The precipitation on the membrane surface increases with the increase of accumulated charge and the gel layer becomes thinner and denser. The formation of a gel layer, ion movement is restricted. Consequently gel layer will have an additional electrical resistance. The concentration and the electrical resistance of the gel layer depends on the foulant and membrane structure (AWWA, 1995, Lee and et al., 2002). With increasing current density, current efficiency increases in ED system. However, polarization phenomena impede the achievement to the upper current density value. The higher current value than the limiting current is caused to fouling and reduction of the process efficiency (Koter and Warszawski, 2000). There are three types of blockages;

- The formation of divalent or trivalent ions hydroxides
- The accumulation of proteins or colloidal substances on surface
- Membrane disruption (Bazinet and Araya-Farias, 2005).

Organic substances such as humates, natural organic substances, and dedoksil sodium benzenesulfonate are fouling on anionic membranes. Colloidal materials , proteins , 2 and 3 valent hydroxyl ions do accumulate blockage of the membrane surface and inside on Cationic membranes. The chain length of the fouling molecule is a physical feature that affects the degree of the fouling. Long chain molecules cause a harder fouling. Larger molecules have lower solubility, so the rate of being adsorbed on the membrane is higher. Current density also affects the fouling among other parameters. Fouling is faster at higher current densities (Lindstrand and et al., 2000a). Suspended solids, silicates and low solubility substances such as iron hydroxide or calcium carbonate are causes of high hydrodynamic pressure loss and flow deterioration. Precipitation on the membrane surface increases the electrical resistance as well as lead to physically damage of membrane structure (Bernardes and et al., 2013).

If internal membrane fouiling have control over the membrane fouling is increase with increasing of concentration. Also surface fouling is more dominate at high concentrations. Protein foulant leads to the formation of the gel layer on the membrane surface and additional hydraulic resistance. Some studies have also been reported that the occurrence of calcium and magnesium hydroxide as well as calcium carbonate at the surface and inside the membranes in the chloralkali industry (Mondor and et al., 2009). Precipitation of inorganic salts leads to crystallization on the membrane surface and it is a reversible situation (Guo and et al., 2014).
Some compounds which cause different precipitation effect during electrodialysis are listed below (Bernardes and et al., 2013);

- Heavy Metals
- Organic and inorganic colloids
- Dissolved organic matter
- Biomass
- Dissolved gases
- Alkaline metals

Lindstrand and et al. (2000b) studied about influence of various kinds of organic solutes on the fouling in ED processes. They noticed that a remarkable difference was observed between the anion selective and the cation selective membrane. The resistance of the cation selective membrane was slightly increased whereas the anion selective membrane was fouled by all solutes. They reported that it was definitely due to the electrostatic interactive relation between the negative charges of the membrane and the negatively charged organic molecules. Also they indicated that the molecular size and the pH of the solution affect the solubility of organic acids. Fouling of larger acid molecules can be awaited to cause of their lower mobility, lower solubility, and greater adsorption affinity to the membrane (Lindstrand and et al., 2000b).

Damages of Membrane Fouling

Fouling problems in the ED system is one of the most important parameters that limit its use. Fouling, negatively affects the selectivity of membrane and will result in poor selectivity also increase energy requirements (Chang and et al., 2009). Most especially, organic anions can precipitate on the anion-exchange membranes and increase the electric resistance. Generally, anion-exchange membranes are more critical to organic compounds fouling.

Colloidal particles are a significant pollutant type for all membrane processes. Colloidal matter that in the feed water comes together with growing of concentration, salinity, flocculation, surface interactions and other physical and chemical factors and hold on the membrane surface. There are examples in about decreasing of membrane performance with colloidal foulant in the literature (Bukhovets and et al., 2010).

Araya-Farias and Bazinet (2006b) showed that in the presence of sodium carbonate fouling has not occurred in anion exchange membrane in their study. The fouling was observed when the salt concentration increases in the feed water. Recently, calcium hydroxide precipitated on the cation exchange membrane which is in contact with sodium chloride (Bazinet and Araya-Farias, 2005).

Mechanical failure, leads to increase chemical deterioration and fouling of membrane. In Ghalloussi and et al. (2011) study, they disclosed that fouled membranes lost 93% of their toughness after 2 years of operation and fracture point decreased of 87%. They reported that even today, there is not wide range of information about decomposition occurring as a result of fouling in the ED system used in the food industry (Garcia-Vasquez and et al., 2013).

Korngold and et al. (1970) specified that the fouling is originated from H^+ ions which produced by polarization at the membrane surface and it increases with decreasing salt concentration and decreasing flow velocity also with increasing current density and colloid concentration. Foulant can be regenerated with alkali solutions or under reversed electric current (Korngold and et al., 1970).

Prevention or Removal of Membrane Fouling

Many studies have focused on the need to reduce of ED membrane fouling. Some methods have been proposed in these studies such as turbulence in the cell compartments, pretreatment of the feed solution, optimization of process conditions and the membrane properties. There are several cleaning procedures, including physical and chemical methods. The selection of the chemical to be applied for membrane cleaning depends on the membrane type, intensity of the pollution and pollutants type. Chemicals that usually recommended by membrane manufacturer are; weak and strong acids, alkali (NaOH), detergents, complex agents (EDTA), disinfectants (H_2O_2, NaCl) and enzymes. Also operation conditions can be considered to reduce fouling. However, these approaches may require an additional chemicals or equipment and as a result, they increase the operating cost (Lee and et al., 2002). Forward flushing, backwashing, vibrations, air sparge and CO_2 back permeating are used for pressure-driven membrane as physical cleaning methods. But these methods are not suitable for ion-exchange membranes. Alternatively, ultrasound is an effective technique for cleaning foulants (Wang and et al., 2011).

Colloids that commonly found in natural water are clay, silica, iron, and aluminum hydroxide, organic deposits, and carbon dust. Pre-filtration, membrane cleaning with chemicals, cross- flow velocity should be provided for control of colloidal pollution. Many foulants move to ion exchange membranes under an electric field by their surface charge and electrical conductivity. After that the foulants accumulate on membranes and the electrical resistance increases. These deposits can be removed with an alternating electrical current. It was reported the pulsed electric fields can cause irregularity of the gel layer on the surface of ion exchange membrane in ED system (Lee and et al., 2002).

Humic matters have an important role in the organic fouling of ED membranes. Organic anions such as humate can be collapsed as humic acid on anion exchange membranes. Membrane fouling with organic anions small enough to penetrate into the membrane, causing these substances remain within the membrane. It causes to increase of electrical resistance vigorously. The use of certain detergents also causes the same effect which is requires to the proper pretreatment of the feed solution. There have been various studies to reduce it, for instance zeta potential control and pulsed voltage. Mechanical cleaning or acids -bases cleaning methods can disrupt the structure of the membrane (Bernardes and et al., 2013, Chang and et al., 2009). Lee and et al. (2002) worked on negatively charged humate with high molecular weight fouled anion exchange membrane by sedimentation on the surface during an ED. They used ED of NaCl solution containing 100 mg/l humate. They found that the pulsed electric fields enhanced the mobility of the charged particles in the fouling layer and decreased the electric resistance of the electrodialysis cell (Lee and et al., 2002).

Membrane fouling depends on the characteristics of membranes, specifications of feed water and the physical and chemical properties of the foulants. Foulant categories, causes of formation and cleaning methods shows in table 3 (Lee and et al., 2002).

Table 3. Foulants and cleaning methods in ED system (Lee and et al., 2002)

FOULANT TYPE	DEFINITION	FOULANTS FORMULA	CHARGE PROPERTIES	CLEANING METHODS	REFERENCE
Scale	Precipitates of less soluble salts in the solution	$CaCO_3$, $CaSO_4$, $2H_2O$, $BaSO_4$, $SrSO_4$, SiO_2	Non	-pH adjustment, -Use of citric acid or EDTA	(Korngold and et al., 1970), (Lonergan and et al., 1982, Wilson, 1959)
Colloids	Stack of suspended material on membrane surfaces	SiO_2, $Fe(OH)_3$, $Al(OH)_3$, $Cr(OH)_3$	Negative	-Pretreatment with MF, UF, higher flow rate, -pH adjustment	(Grossman and Sonin, 1972), (Schippers and Verdouw, 1980), (Rao and et al., 1989)
Organics	Adsorption of organic species to membrane surfaces	Macromolecules, proteins, whey, polyelectrolytes, humate	Negative	-Pretreatment with MF, UF, activeted carbon, -Clean with NaOH	(Korngold and et al., 1970), (Mallevialle and et al., 1996), (Kressman and Tye, 1969), (Kobus and Heertjes, 1972),

In Bazinet and Araya-Farias (2005) study, they investigated that the effect of fouling on cation exchange membranes with composition in calcium and carbonate of a model solution to be treated by ED. They reported that there was no fouling at 400 and 800 mg/L of $CaCl_2$ in the absence of carbonate but a deposit was observed at only 400 mg/L $CaCl_2$ with carbonate. They explained that this difference could be by the buffering capacity of the carbonate that affects the system with and without sodium carbonate. They also reported that this fouling formed during ED was easily cleaned by an acid procedure (Bazinet and Araya-Farias, 2005). In a subsequent study Araya-Farias and Bazinet (2006a) indicated that the presence of fouling was only observed on the membranes treated with a solution containing 400, 800, and 1600 mg/L of CaCl2 in presence of sodium carbonate. They saw that fouling on the anionic membranes was typical of a cubic and crystalline material. The crystals would be identified as calcium hydroxide. They reported that this fouling was not found to affect significantly the conductivity and the thickness of the membranes and it was easily dissolved after the soaking with HCl (Araya-Farias and Bazinet, 2006a). According to the Bernardes and et al. (2013) study, biomass is the another important problem for membrane fouling. Biomass fouling is the type of contamination in the feed water caused by microorganisms. Microorganisms such as bacteria, fungi, algae, viruses cause fouling by forming a thin layer on the membrane surface. They can damage the membrane but they can be extinguished by sterilization. Biological fouling not only increases the membrane resistance also it causes biological degradation by hydrolysis of the membrane polymer

Natural organic matter (NOM) is a well investigated another problem in the ED system especially for the brackish water desalination industry. According to some research the current and flow direction are periodically inversed to break fouling originating from NOM. This methods call as ''Electrodialysis reversal'' (EDR) system. EDR provides a continuous self-cleaning electrodialysis process that uses periodic reversal of direct current polarity. When polarity is reversed in ED system, chemical reactions at the electrodes are reversed. However, EDR can make fouling easily reversible. Some pretreatment is always necessary to complete EDR (Bouhidel and Rumeau, 2004).

Conclusions

ED system is well constituted in water treatment as a confidential process since more than half a century. Generally, ion exchange membranes are in competition with other separation techniques such as reverse osmosis, ultrafiltration, nanofiltration or conventional ion-exchange. According to some study ED processes have more advantages than the other separation techniques. The main advantages of ED are; there is no osmotic pressure, higher quality product, environmentally friendly, no additional chemicals and ion exchange membranes have long useful life. But ED has various limitations. A major disadvantage of ED system is membrane fouling because it reduces the limiting current, reduces the flux, increases the membrane resistance, decreases the ions migration

yield and lead to serious polarization problems. Fouling increases with decreasing flow velocity, with increasing current density and colloid concentration.

Electrodialysis processes are promising separation technologies for clean production. While decreasing of raw materials in the world, ED system can provide to recycling and reuse some valuable substances. For sustainable development, clean technologies are become more and more important. Electrodialysis can removes ions by electrical potential difference. Thus biomass, colloidal material or organic materials in the feed water will survive in the product stream. To prevent fouling, need for the addition of chemical products, mechanical methods or the system polarity can be reversed periodically with electrodialysis reversal (EDR).

ED based processes are preferable in some applications because of providing products of higher quality or being environmentally friendly. Development of electromembrane system is very important for multidisciplinary science and technology. Thus the researchers focused on the electrodialysis system thoroughly and obviate to current design and operating problems.

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