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RECOVERY OF WATER UTILIZED IN FOOD INDUSTRY BY INTEGRATED MEMBRANE SYSTEMS

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Esra ALTIOK tarafından Yüksek Lisans tezi olarak sunulan "RECOVERY OF WATER UTILIZED IN FOOD INDUSTRY BY INTEGRATED MEMBRANE SYSTEMS" başlıklı bu çalışma EÜ Lisansüstü Eğitim ve Öğretim Yönetmeliği ile EÜ Fen Bilimleri Enstitüsü Eğitim ve Öğretim Yönergesi'nin ilgili hükümleri uyarınca tarafımızdan değerlendirilerek savunmaya değer bulunmuş ve 23.08.2017 tarihinde yapılan tez savunma sınavında aday oybirliği/oyçokluğu ile başarılı bulunmuştur.

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23/08/2017

Esra ALTIOK

ÖZET

Bütünleşik Membran Sistemleri İle Gıda Endüstrisinde Kullanılan Suyun Geri Kazanımı

ALTIOK, Esra

Yüksek Lisans Tezi, Kimya Mühendisliği Bölümü

Tez Danışmanı: Prof. Dr. Nalan KABAY

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Bu çalışmada meyve suyu işletmelerinde ham kuyu suyunun ters ozmos ile arıtılması sonucunda ortaya çıkan atık suyu geri kazanılmasında elektrodiyaliz (ED) ve elektrodeiyonizasyon (EDI) yöntemlerinin uygulanabilirliği incelenmiştir. Bu amaç için İzmir'de bulunan meyve suyu fabrikasından alınan ham kuyu suyu ve ters ozmos konsantre örnekleri ile çalışmalar yapılmıştır.

Ham kuyu suyu ile yapılan çalışmalarda laboratuvar ortamında konsantre üretebilmek için laboratuvar ölçekli çapraz akışlı düz tabakalı SEPA CF II GE-Osmonics membran test sistemi 2 farklı nanofiltrasyon (NF-90 ve NF-270) ve 2 farklı ters ozmos membranı (BW30 ve AK-BWRO) ile kullanılmıştır. Membran test sisteminden elde edilen konsantre bileşenler ED sisteminde besleme çözeltisi olarak kullanılmıştır. Deneylerde Tokuyama Firmasının TS-1-10 model Elektrodiyaliz cihazı kullanılmıştır. Yapılan çalışmalar sonucunda optimum koşul olarak bulunan 10 V elektriksel potansiyel uygulanan ED deneylerinde oluşan seyreltik çözelti EDI sisteminde besleme çözeltisi olarak kullanılmıştır.

Ters ozmos konsantresi ile yapılan çalışmalarda bu konsantre direkt olarak ED besleme çözeltisi olarak kullanılmıştır. Ham kuyu suyu ile yapılan çalışmalarda olduğu gibi bu çalışmada da optimum koşul olan 10 V elektriksel potansiyel uygulanan ED deneylerinde oluşan seyreltik çözelti toplanılmış ardından EDI sisteminde besleme çözeltisi olarak kullanılmıştır.

EDI deneylerinin sonucunda %50 oranında su geri kazanılmış ve kazanılan üretilen bu su düşük iletkenliğe sahip yüksek kaliteli sudur.

Anahtar Kelimeler: Elektrodeiyonizasyon, elektrodiyaliz, gıda endüstrisi, konsantre yönetimi, nanofiltrasyon, suyun yeniden kullanımı ters ozmos,

ABSTRACT

Recovery of Water Utilized in Food Industry by Using Integrated Membrane Systems

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Master Science Thesis, Department of Chemical Engineering

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In this study, the feasibility of electrodialysis (ED) and electrodeionization (EDI) methods in the recovery of brine of reverse osmosis treatment of raw well water in fruit juice enterprises has been investigated. For this purpose, studies were carried out with raw well water and reverse osmosis brine samples taken from the fruit juice factory which located in Izmir.

The laboratory-scale cross-flow flat-sheet SEPA CF II GE-Osmonics membrane test system was used to produce brines in laboratory with raw well water and by using 2 different nanofiltration (NF-90 and NF-270) and 2 different reverse osmosis membranes (BW30 and AK-BWRO). Concentrated components from the membrane test system were used as feed solution in the ED system. TS-1-10 model Electrodialysis device of Tokuyama Company was used in the experiments. The diluated solution formed in ED experiments with 10 V electrical potential, was used as a feed solution in the EDI system which was the optimum condition of ED system.

When it comes to studies conducted by reverse osmosis brine, the brine used as feed solution directly ED system. In these studies, diluated solution formed in ED experiments with 10 V electrical potential, which is the optimum condition, was collected and then used as feed solution in EDI system.

As a result of the EDI experiments, 50% of the water is recovered and this water produced is high quality water with low conductivity.

Keywords: Concentration management, electrodeionization (EDI), electrodialysis (ED), food industry, nanofiltration (NF), reverse osmosis (RO), water reuse

TEŞEKKÜR

Bu çalışma, TÜBİTAK 114M551 projesi kapsamında desteklenmiştir. Yüksek lisans çalışmam boyunca 114M551 ve 214M360 projelerinden sağlanan burslar için TÜBİTAK'a çok teşekkür ederim.

Gerek yüksek lisans eğitimim ve gerekse öğrencisi olduğum tüm zaman boyunca bana her türlü imkânı sağlayarak sabırla destek olup hep cesaretlendiren danışmanım Prof. Dr. Nalan Kabay'a müteşekkirim. Değerli tecrübeleriyle katkılarını ve desteğini esirgemeyen Prof. Dr. Mithat Yüksel'e teşekkür ederim.

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Deneylerimiz için gerekli olan su örneklerini bize sağlayan Dimes'e teşekkür ederim. Deneylerimizde kullandığımız NF ve RO membranları için Dow Kimyasal, iyon değiştirici membranlar için Fujifilm ve ASTOM AŞ.'ye ve iyon değiştirici reçineler için de Purolite AŞ. ve Lewatit A.Ş.'ye katkılarından dolayı teşekkür ederim. AAS analizlerinde Kimyager Mehmet Akçay ve TOK analizlerinde Gürsel Serin'e yardımlarından dolayı teşekkür ederim.

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NOMENCLATURE

ED	Electrodialysis
EDI	Electrodeionization
ILSI	The International Life Sciences Institute
LEAD	The Livestock Environment and Development
MWCO	Molecular Weight Cut-Off
MF	Microfiltration
NF	Nanofiltration
RO	Reverse Osmosis
UF	Ultrafiltration
TDS	Total Dissolved Solids (mg/L)

1. INTRODUCTION

Water is an essential natural resource mobilized by human activities which is circulated through the natural water cycle. Large part of the world’s human population and agricultural expansion is located in water stressed regions and the increasing level of demand for water is a growing concern. The threats of increasing deficiency of water show that the situation will worsen in the next decades (LEAD, 2006) To solve the scarcity problem of water, some precaution should be taken like recharging aquifers and groundwater, besides putting into practice of water reuse and water management and effective water treatment technologies. (Eartheclipse, 2017).

1.1. Water Reuse

Around the world, water supplies are under increasing pressure due to population growth, climate change, pollution, and changes in land use affect water quantity and quality. Many communities are working to increase water conservation and are seeking alternative sources for potable water. Treatment of wastewater and water from rivers, lakes, or aquifers is one of the strategies to increase usable water supplies. The treated wastewater, or reclaimed water are used for beneficial purposes such as drinking or industrial uses, power stations, agriculture, toilet flushing, decorative fountains, irrigation for parks, golf courses, other open areas and landscaping (Bunani, et al. 2014)

In order to attain the environmental standards, membrane technologies provide an important solution in wastewater discharge, reuse and recovery of water, recycling valuable components from the waste streams (Marcucci et al., 2001) Depending on the demand of water reuse purposes, various quality of water can be obtained by using different membrane filtration techniques such as reverse osmosis (RO), nanofiltration (NF), ultrafiltration (UF), microfiltration (MF) and electromembrane processes like electrodialysis (ED), electrodialysis reversal (EDR) and electrodeionization (EDI) (Bunani, et al. 2014).

1.1.1. Water reuse in food and beverage industry

Water is an essential natural resource for the food producing industry, as large volumes are needed to meet processing and ingredient demands (Uschamber, 2017). As a result, water reuse is an attractive option to minimize the use of additional potable water and discharge of wastewater. Due to legal constraints and hygienic concerns, despite the food industry used and discharged large volume of water, a very small amount of it was treated and reused. Therefore, instead of reusing of treated wastewater in food industry, minimizing the waste of ground water treatment process is the more acceptable way due to hygienic concerns. But with growing technologies, these worries are lagging behind (Casania et al., 2005).

Technological innovations and process wastes spent in food processing plants allow for the recycling of waste wastewater treatment applications to reduce the amount of wastewater discharged to municipal sewage treatment plants.

In food processing, water management can be obtained by increased efficiency of water use and/or the promotion of water reuse. The significant demands for water in food processing are listed below: (ILSI, 2008).

- Washing / cleaning of (raw) products
- Transport of products
- Dissolving of ingredients
- Treatment of the product (e.g. alteration, separation)
- Provision of appropriate water content in the final product
- Cooling processes
- Steam generation
- Cleaning / rinsing of equipment

- Abnormal incidents (e.g. fire protection)

- Sanitation

1.2. Membrane Processes

A membrane (semipermeable membrane) is a thin layer material which is capable of separating materials in the presence of a driving force (Mallevialle et al., 1996). A membrane is also defined as a barrier that does not allow for all the species pass through but permeable to some of them (Sammon, 1973). Thus a membrane divides the medium in two different sides: a component rich side and a component poor side. The rich side of the components is called the concentrate stream and the poor side is the permeate stream (filtrate).

To perform a membrane separation process, a driving force is required to achieve the transfer of the components throughout a membrane from one side to the other side of the membrane. These driving forces can be the pressure difference~~s~~, concentration differences and electrical potential difference~~s~~. Depending on the driving force involved in the separation process, tree different membrane separation process groups can be distinguished: pressure driven membrane processes, chemical potential based processes (concentration difference based) and electrical potential based membrane processes (electromembrane processes). Typical examples of membrane separation process characteristics are shown in Table 1.1

According to the properties of the membrane used in the process, the separated species and the properties of other materials that are involved in the separation technique, each group includes various type of the process based on the same principle. The membrane processes can be categorized as;

- Pressure difference based processes.
    - Reverse osmosis (RO)
    - Nanofiltration (NF)
    - Ultrafiltration (UF)

  - Microfiltration (MF)

- Concentration difference based processes.
  - Dialysis
  - Membrane extraction
  - Forward osmosis
- Electrical potential difference based processes.
  - Electrodialysis (ED)
  - Membrane electrolysis
  - Electrodeionization (EDI) (Matsuura, 2009)

Table 1.1 Examples of membrane separation process characteristics (Bernardes, 2014)

Membrane Process	Driving force	Typical separation mechanism	Retentate	Permeate
Microfiltration (MF)	ΔP (0.5-2 bar)	Sieve	Suspended solids, bacteria MW>500000Da (0.01μm)	Water and dissolved solids
Ultrafiltration (UF)	ΔP (1-7 bar)	Sieve	Colloids, macromolecules MW>2000 Da	Water, salts and compounds of low molecular weight
Nanofiltration (NF)	ΔP (05-25 bar)	Sieve+solution/ diffusion+exclusion	Molecules 500 Da<MW<2000Da	Water, salts and compounds of low molecular weight
Reverse Osmosis (RO)	ΔP (15-80 bar)	Solution/diffusion +exclusion	All soluble or suspended material	Water, solvent
Dialysis (D)	ΔC	Diffusion	Molecules MW>5000 Da	Ions and organic compounds of low molecular weight
Electrodialysis (ED)	ΔE	Ion exchange	Macromolecules and non ionic compounds	Ions

ΔP: Pressure difference
ΔC: Concentration difference
ΔE: Potential difference
MW: Molecular Weight

1.2.1. Pressure driven membrane processes

Pressure driven membrane processes are typically classified as microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) in an order of decreasing pore size of the membrane used. As shown in Table 1.2, the pressure difference across the membrane increases with decreasing of the membrane’s pore size (Chriwa, 2017).

Table 1.2 Separation of various species by pressure driven membrane processes (Chriwa, 2017)

Membrane technology	**Microfiltration >0.2 mm**	**Ultrafiltration 0.1-0.01 mm**	**Nanofiltration 0.01-0.001 mm**	**Reverse osmosis <0.001 mm**
Retained water ingredients	Zooplankton	Macromolecules	Organic compounds	Monovalent ions
	Algae			
	Turbidity	Viruses	Multivalent ions	
	Bacteria			
	Suspended particles	Colloids		
Needed pressure difference	0.2-3 bar	0.5-5 bar	5-10 bar	10-150 bar

Based on the type of the processes employed, membranes used are categorized as polymeric and inorganic membranes. The fabrication of polymeric membranes involves many organic polymers including crystalline and amorphous, glassy and rubbery. On the other hand, oxides, ceramics, metals and carbon are the material commonly used for the fabrication of inorganic membranes. These membrane preparation methods involve various techniques such as phase inversion, interfacial reaction, coating, stretching and so forth. The most available commercial membranes are prepared using phase inversion approach (Lawrence et al., 2011).

In terms of morphologies, polymeric membranes are generally considered as isotropic and anisotropic membranes. While isotropic membranes include nonporous dense membranes, microporous membranes, and electrically charged membranes, anisotropic membranes consist of integrated asymmetric membranes, composite membranes, and supported liquid membranes as shown in Figure 1.1 (Baker, 2004).

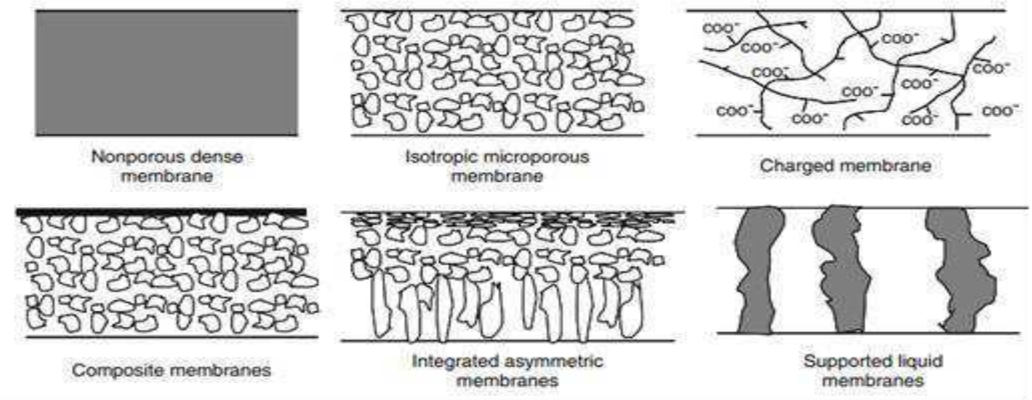

Figure 1.1 Diagram of different membrane morphologies (Baker, 2004).

1.2.1.1.Reverse osmosis (RO)

Reverse osmosis (RO), also known as hyper filtration is a pressure driven membrane process where membrane acts as the semipermeable barrier, allowing selective transition of smaller species such as water while partially or completely retaining other species (Williams, 2003).

RO membranes have dense active layers, where the preferred transport mechanisms are often attributed to solution/diffusion. Because of the high density of the active layer, operating pressures are higher than MF and ultrafiltration processes (Bernardes, 2014).

The name of RO comes from the direction of solvent flow that is opposite of natural occurring osmosis process (Sammon, 1973). In natural osmosis, saline solution has high osmotic pressure than pure water. To equalize the pressures of both sides of a semipermeable membrane, pure water transfers to saline solution side. By applying external pressure, higher than osmotic pressure the process is reversed such that water moves from saline solution to pure water side. This process is called reverse process (Williams, 2003). This phenomena is shown in Figure 1.2 (Bunani, 2013).

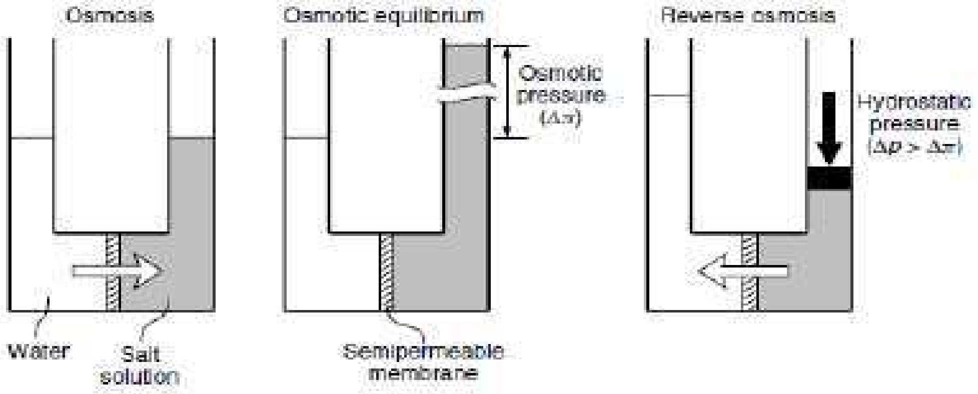

Figure 1.2 Schematic representation of reverse osmosis process (Bunani, 2013)

1.2.1.2.Nanofiltration (NF)

Nanofiltration (NF) method is an application in a range between RO and UF (Bowen and Mukhtar, 1996; Wang et al., 1995). NF has replaced reverse osmosis (RO) in many applications due to lower energy consumption and higher flux rates. The properties of NF membranes lie between those of non-porous RO membranes (where transport is governed by a solution-diffusion mechanism) and porous ultrafiltration (UF) membranes separation is usually assumed to be due to size exclusion and, in some cases, charge effects.

In NF process, even small uncharged solutes are highly rejected while the surface electrostatic properties of NF membranes allow monovalent ions to be reasonably well transmitted although multivalent ions mostly are retained. The NF technology has several applications in a number of industries such as treatment of pulp-bleaching effluents from the textile industry, separation of pharmaceuticals from fermentation broths, demineralization in the dairy industry, and metal recovery from wastewater and virus removal from water etc (Shon, et al., 2013).

1.2.1.3.Ultrafiltration (UF)

Ultrafiltration (UF) is pressure driven membrane separation process mainly based on size-exclusion mechanism. Since the membranes used in UF have pore sizes in the range of 2–50 nm, they retain species in the molecular range from 3000 to 500000 Da (Kulkarni, et al., 1992) while smaller molecules including water pass through the membrane. Both UF and MF membranes are porous in nature, but UF membranes have much denser skin layer with always an asymmetric structure. As a consequence of smaller pore size and lower surface porosity, UF membranes have higher hydrodynamic resistance. The thickness of the top layer is 1.0-3.0 µm (Mulder, 1997).

1.2.1.4.Microfiltration (MF)

Microfiltration (MF) is a pressure driven membrane characterized by a membrane pore size between 0.05-10.0 µm. MF process operates under the pressure below 3 bar. MF is used for removing particles or biological entities in the 0.1 µm to 10.0 µm range from aqueous solutions Although MF has some limitations such as the service life of the filter (Starbard, 2008), it is widely used in water and wastewater treatments, dairy, biotechnological and pharmaceutical industries, food and beverage processing, and medical applications.

1.2.2. Electrically driven membrane processes

Electrically driven membrane processes are widely used to remove charged components from feed solution or suspension. In order to establish an electrical driving force you need an electrical field. Therefore two electrodes are required; a cathode and an anode. The positive ions in a solution will migrate to negative electrode, the negative ions will migrate to the positive electrode and the uncharged molecules will not be affected by electrical field. Main application of electrically driven membrane processes is the desalination of saline water in the production of potable water (Beier, 2015).

1.2.2.1.Electrodialysis (ED)

Electrodialysis (ED) is an electrochemical separation process in which ionic species are separated from an aqueous solution under an electrical potential by the help of charged membranes. Charged membranes used in conventional ED stacks are typically cation and anion exchange membranes. The charged membranes are made of ion-exchangers similar in some ways to those used in preparation of cation and anion-exchange beads. The cation and ion exchange membranes are arranged alternatively between two electrodes (a cathode and an anode). By this membrane arrangement with salt solutions flowing between them, two different solutions called diluate and concentrate are formed in ED process. Under the driving force of a direct current (DC), electric potential that is applied perpendicular to the flow, cations are transferred towards the cathode through cation exchange membrane as well as ions towards the anode through anion exchange membrane (Rajindar and Nicholas 2016). The side where the ions move from is therefore named diluate, while the side where theses ions move to is named concentrate. The principle of ED process is shown in Figure 1.3.

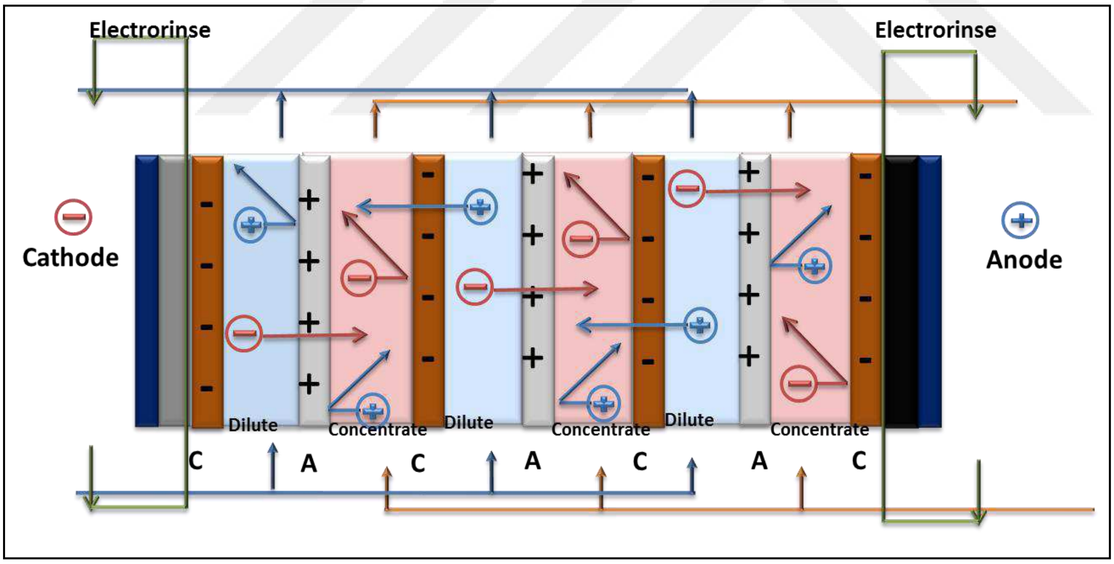

Figure 1.3 The flow scheme of ED process

The main applications of ED include desalination of brackish water with TDS less than 5000 mg/L, and treatment of municipal water and wastewater (Rajindar, and Nicholas , 2016). However, the ED process is reported to be more cost-effective for water containing TDS concentrations less than 5000 mg/L (Al-Karaghouli and Kazmerski, 2013). For solution with higher TDS such as seawater, it is not competitive with RO because of high energy consumption (Rajindar, and Nicholas, 2016). For desalination of higher TDS saline solutions, ED is sometimes preferred rather than RO because it is not affected by osmotic pressure. For example, ED was used in Japan to concentrate NaCl from seawater for the production of table salt (Strathmann, 2010, and Tanaka, 2010,). It was used to increase the TDS concentration of seawater from 35,000 mg/L to 150,000-200,000 mg/L before the evaporation and crystallization steps. In addition to desalination, ED finds its application for the pretreatment of RO feed water and for post treatment of RO concentrates, e.g. for water and salt recovery (Kabay, et al., 2016). In this case of pretreatment of RO feed water, ED with monovalent ion selective membranes is expected to selectively remove multivalent ions such as calcium, magnesium and sulfate that are responsible of RO membrane scaling. The use of ED for RO concentrate treatment contributes in minimizing impact of waste disposal on the environment, recovering chemicals and increasing usable water recovery (Kabay, et al., 2016).

1.2.2.1. Electrodeionization (EDI)

Electrodeionization (EDI) is a hybrid process that consists of combination of conventional electrodialysis (ED) and ion exchange resins together. Cation and anion exchange membranes are placed between the electrodes like in ED system but between the membranes of diluting solution, the ion exchange resins are placed either in mixed or layered mode. Ions contained in the diluate compartment solution react with the ion exchange resin between the ions exchange membranes and transfer through the resin in the direction of the potential gradient. At the same time, these ions transfer across the membranes and then maintain the neutrality in all compartments. The presence of permselective ion exchange membranes and the electrical potential, ions in diluated compartment are depleted and they are concentrated in the adjacent compartment (Kabay, et al., 2016).

The role of ion exchange resins in EDI process is that they enhance the transport of ions and can also participate as a substrate for electrochemical reactions, such as water dissociation into H^+ and OH^- ions (Wood et al., 2010). The H^+ ions and OH^- ions produced during water splitting are transported towards the cathode and anode under an applied potential field, respectively. They recombine in the diluate and/or the concentrate compartments and help the process in regeneration of the ion exchange resins without addition of chemicals. Because of the water splitting under an electrical potential field, the ion exchange resins are continuously regenerated in mixed bed ion exchange media, exchanging H^+ ions and OH^- ions in stoichiometric amounts with other ions present in the solution (Tanaka, 2007; Song 2006). EDI is mostly applied for ultra-pure water production. EDI has been used for polishing RO permeates by removing weakly ionized species, such as carbon dioxide, silica and boron which are difficult to remove by means of RO and ED. Removal of boron and silica from the RO permeate of geothermal water was studied by Arar et al. (Arar et al. 2013) where boron and silica concentrations in the product water was 1.60 mg/L and 0.20 mg/L, respectively. For high performance of EDI, however, there are many experimental strategies that have to be implemented. In addition to EDI feed solution requirements, process parameters such as electrical current applied, flow velocity in the diluate and concentrate compartments and temperature should be optimized. In EDI applications, feed water conductivity, CO_2 content and temperature are restricted so that EDI can achieve a high product water quality. Avoiding extreme temperature, pressure, and foulants such as organics and hardness that could damage the EDI performance is also required. EDI feed water conductivity is restricted to less than 50 μS/cm, CO_2 concentration $\leq$ 20 mg/L and free chlorine, silica, hardness (as $CaCO_3$) and TOC have respective maximum limit of 0.05 mg/L, 1.0 mg/L, 1.0 mg/L and 0.05 mg/L (Wood et al., 2010). Applied temperature and pressure to the system have to be between 5-45°C and 1.0-6.9 bar, respectively.

1.2.2.2.Ion exchange membranes

Ion exchange membranes are essential components in electrochemical separation processes. These membranes can be characterized in terms of their function in seperation or in terms of their structure (Strathmann, 2010).

There are three types of ion-exchange membranes; cation exchange membrane, anion-exchange membrane and bipolar membrane. While cation exchange membrane has negatively charged fixed groups attached to polymer membrane matrix, anion exchange membranes has positively charged fixed groups are attached to polymer membrane matrix (Singh, and Shahi, 2013). And in case of bipolar membrane bipolar membranes, it is a composed of an anion- and a cation-exchange layer laminated together (Strathmann, 2010).

Fixed charges present in the membranes allow to ions movements through the membrane matrix due to the exclusion of co-ions. The driving forces for the transport of ions are the electrochemical potential gradients as shown in Figure 1.4 (Singh, and Shahi, 2013).

Cation exchange membranes are often sulfonated to attach SO_3^- groups to the polymer. They permit intrusion and exchange of cations from an external source, but exclude anions. Anion exchange membranes have a similar polymer structure with attached quaternary ammonium groups (Arar, 2012).

Ion-exchange membranes can be divided according to their structure and the way they are prepared into homogeneous and heterogeneous membranes. In homogeneous ion-exchange membranes, the fixed charged groups are evenly distributed over the entire membrane polymer matrix. Heterogeneous ion-exchange membranes have distinct macroscopic domains of ion-exchange resins in the matrix of a polymer.

An ion exchange membrane should have high permselectivity, low electrical resistance, and good mechanical and chemical stability (Vogel, Meier-Haack, 2014).

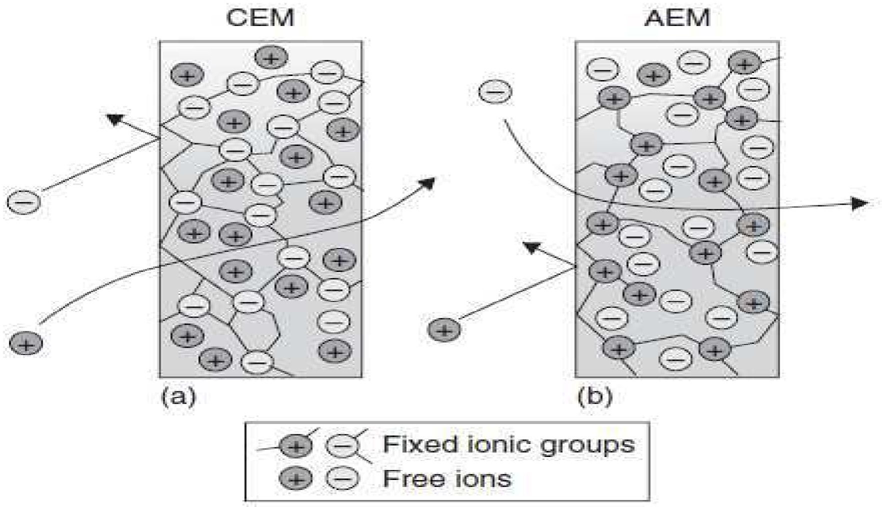

Figure 1.4 Schematic presentation of cation exchange membrane and anion-exchange membrane (Singh, and Shahi, 2013)

1.2.2.3.Ion exchange resins

Ion exchange resins are insoluble polymers which contain acidic or basic functional groups and have the ability to exchange counter-ions within aqueous solution. The material has a highly developed structure of pores on the surfaces from where the ions are trapped or released. The trapping of ions takes place only with simultaneous release of other ions; thus, the process is called ion exchange (Singh, et al., 2016).

The chemical properties of the resins basically depend on the type of functional group.

Ion exchange resins are manufactured by introducing functional groups such as sulfonic acid and quaternary ammonium into three dimensionally crosslinked polymer matrix. The ion exchangeable functional groups are called fixed ions because they are chemically bonded to the polymer structure. On the contrary, the mobile ions that are electrically neutralized with fixed ions are called counter ions.

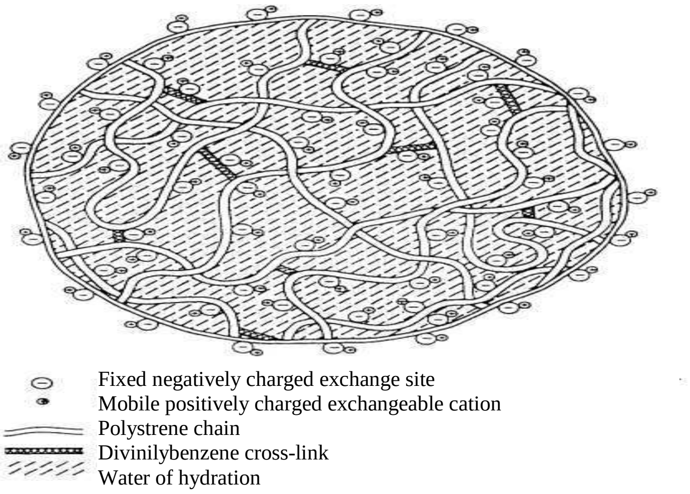

Figure 1.5 Ion exchange resin particle

Ion exchange resins are broadly classified into two main categories as cation exchange resins and anion exchange resins. Cation exchange resins, whose exchangeable ions are positively charged while anion exchange resin has negatively charged exchangeable ions (Singh, et al., 2016).

Ionizable groups attached to the resin bead determine the functional capability of the resin. Industrial water treatment resins are classified into four basic categories:

- Strong Acid Cation (SAC)
- Weak Acid Cation (WAC)
- Strong Base Anion (SBA)
- Weak Base Anion (WBA)

SAC resins can neutralize strong bases and convert neutral salts into their corresponding acids. SBA resins can neutralize strong acids and convert neutral salts into their corresponding bases. These resins are utilized in most softening and full demineralization applications. WAC and WBA resins are able to neutralize strong bases and acids, respectively. These resins are used for dealkalization, partial demineralization, or (in combination with strong resins) full demineralization (Gewater, 2017).

1.3. Aim of This Study

The aim of this thesis is the purification of ground water which is used for fruit juice production by NF and RO filtration methods. Recovery of water from concentrated streams of NF and RO processes discharged during membrane filtration step by means of ED and EDI methods is another target of this thesis. For doing this, optimal conditions such a membrane type, electrical potential and flow rate applied during ED/EDI operations are investigated and obtained.

2. EXPERIMENTAL

2.1. Materials

To perform experiments in laboratory, ground water and RO treatment plant concentrate samples were taken from a fruit juice located in İzmir. The characteristics of the water taken from the plant are given in Table 2.1.

Table 2.1 Characteristics of ground water and RO concentrate water from the fruit juice plant

Parameters	Unit	Ground water (min-max)	RO Treatment plant concentrate
pH	-	7.62-7.82	7.53
Conductivity	µS/cm	771-873	5450
TDS	mg/L	377-428	2870
Salinity	‰	0.38-0.43	2.94
Resistance	Ωcm	1298-1146	1835
TSS	mg/L	0.00	1.00
Color	mg/L (Pt)	3-7	19.0
Turbidity	NTU	0.05-0.36	0.12
Ammonium	mg/L	0.026-0.086	0.15
Chlorine	mg/L	29.8-44.1	1407
Nitrite	mg/L	<0.015	<0.015
Nitrate	mg/L	13.1-17.4	89.8
COD	mg/L	5.74-10.8	20.80
Sulfate	mg/L	34.30-52.4	345
Silica (SiO_2)	mg/L	18.70-21.3	87.8
Phosphate	mg/L	<0.05-0.024	13.5
Bicarbonate	mg/L	0.54-0.68	2.95
TOC	mg/L	2.49-3.01	168

During the NF/RO membrane filtration tests 2 different NF and 2 different RO membrane were used. The characteristics of these membranes were shown in Table 2.2.

Table 2.2 Characteristic of NF membranes

Membrane	**NF-90**	**NF-270**	**AK-BWRO**	**BW30**
Manufacturer	Dow FilmTech	Dow FilmTech	GE Osmonics	Dow FilmTech
Membrane Type	Polyamide thin film composite	Polyamide thin film composite	Polyamide thin film composite	Polyamide thin film composite
Maximum Operating Pressure (bar)	41	41	28	41
Maximum Temperature (^{o}C)	45	45	50	45
Operating pH Range	3.0-10.0	3.0-10.0	4.0-11.0	2.0-11.0
Minimum NaCl rejection (%)	> 85	> 97	98.0	99.0
MWCO (Dalton)	200	400	Dense	Dense

Table 2.3 shows Neosepta AMX and Neosepta CMX ion exchange membranes characteristics which used in ED cell and EDI cell.

Table 2.3 Properties of ion exchange membranes used in ED system

Membrane	**NEOSEPTA CMX**	**NEOSEPTA AMX**
Type	Strongly acidic cation permeable	Strongly basic anion permeable
Characteristics	High mechanical strength (Na-form)	High mechanical strength (Cl-form)
Electrical Resistance (Ω -cm^2)	2.0 - 3.5	2.0 - 3.5
Burst Strength (kgf/cm^2)	3.5 - 6.0	4.5 - 5.5
Thickness (mm)	0.16 - 0.20	0.14 - 0.18
Application	Demineralization of whey, Purification of organics, Concentration of inorganics, Demineralization of sucrose, Desalination of ground water	Demineralization of whey, Purification of organics, Concentration of inorganics

In EDI cell between ion exchange membranes ion exchange resins were placed in mixed bed form. For this Lewatit or Purolite ion exchange resin pair were used in EDI experiments. The properties of these resins were shown in Table 2.4 and Table 2.5.

Table 2.4 Properties of ion exchange resins used in EDI system (Lewatit)

	Lewatit® UltraPure 1213 MD	**Lewatit® UltraPure 1243 MD**
Ionic form as shipped	H^+	OH^-
Functional group	Sulfonic acid	Quaternary amine, type I
Matrix	Crosslinked polystyrene	Crosslinked polystyrene
Structure	Gel type beads	Gel type beads
Appearance	Brown, translucent	Light brown, translucent

Table 2.5 Properties of ion exchange resins used in EDI system (Purolite)

	Purolite® CT175	**Purolite® A500**
Application	MTBE & TAME – Highly Crosslinked	Uranium and Gold Mining - RIP Grade
Polymer Structure	Macroporous polystyrene crosslinked with divinylbenzene	Macroporous polystyrene crosslinked with divinylbenzene
Appearance	Spherical Beads	Spherical Beads
Functional Group	Sulfonic Acid	Type I Quaternary Ammonium
Ionic form	H^+	Cl^-
Moisture Retention	50 - 57 % (H^+ form)	53 - 58 % (Cl^- form)
Specific Gravity	1.19	1.08

2.2. Equipments

2.2.1. NF/RO Tests

In the NF/RO test experiments, a lab-scale cross-flow flat sheet membrane test system (SEPA CF II GE Osmonics) (Figure 2.1) was used. Before NF/RO processes, ground water taken from fruit juice plant was pre-filtrated with a filter paper. The characteristics of water taken from the plant are given in Table 2.1.The performances of two NF membranes (NF-90 and NF-270) and two RO membranes (AK-BWRO and BW30) performances were investigated at 10 bar for NF membranes and 20 bar for RO membranes. The concentrate stream flow rate was kept constant at 96 L/h. The characteristics of the membranes used in the experiments and the flow diagram of the membrane filtration system are given in Table 2.2 and Figure 2.2, respectively.

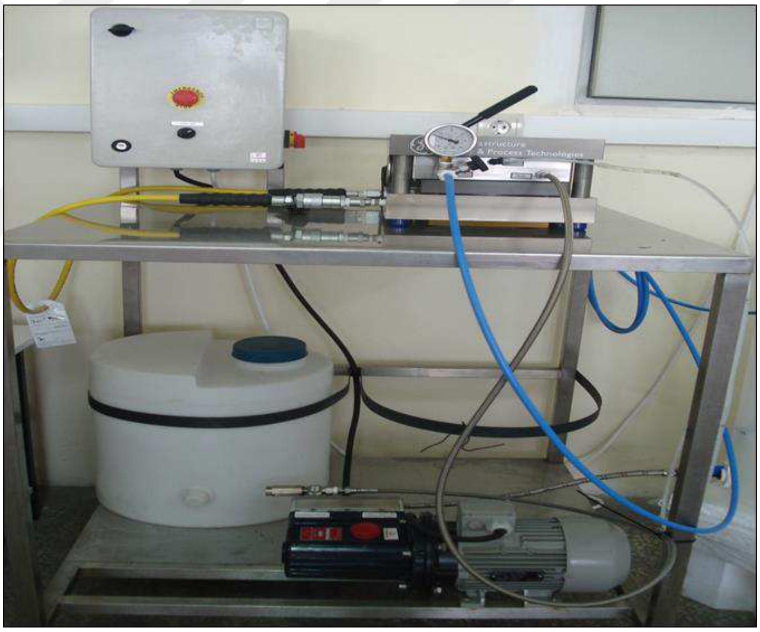

Figure 2.1 SEPA CF II GE-Osmonics Membrane test system

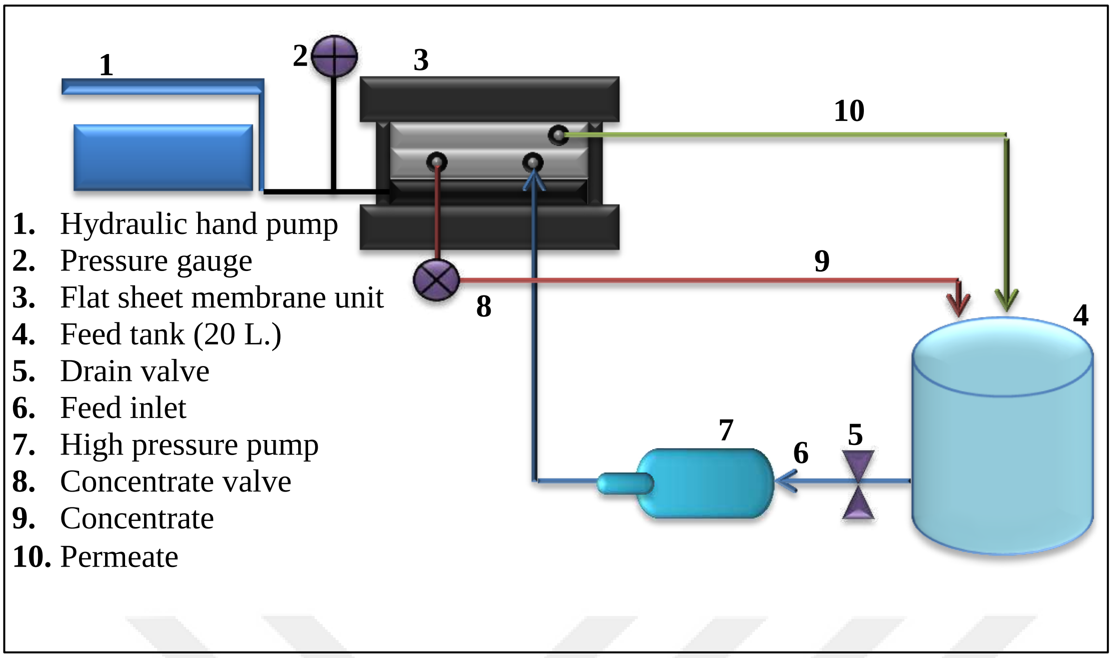

Figure 2.2 Flow diagram of lab-scale cross-flow flat sheet membrane test system

2.2.2. ED tests

ED test experiments were achieved by using a lab-scale Tokuyama TS-1-10 model ED system (Figure 2.3). The feed solutions of ED were obtained from NF/RO concentrate produced by two different membrane filtration systems: an RO concentrate of RO process (a system with 6 steps) used in fruit juice plant and NF/RO concentrate of NF/RO membranes (obtained from Sepa CF II membrane filtration test system) described in section 1.2.1. The flow diagram of the ED system is given in Figure 2.4. The ED system consists of 10 pairs of highly mechanically strong Neosepta cation (CMX) and anion (AMX) exchange membranes with an effective membrane area of 1 dm^2 and plated Ti anode and 316 stainless steel cathode electrodes. Table 2.3 shows Neosepta AMX and Neosepta CMX ion exchange membranes characteristics which used in ED cell and EDI cell.

In ED studies; concentrate, diluate and electrode rinse reservoirs were filled with 1 L of feed solution. Samples (10 ml) for analysis were taken from the concentrate and diluate compartments at interval time of 4 min. The pH and conductivity values of the samples were measured each 4 min. ED process was stopped when the current through the system dropped to 0.01 A. During ED process, three different electrical potentials (7, 10 and 13 V) were tested applied.

Figure 2.3 Tokuyama TS-1-10 model ED system

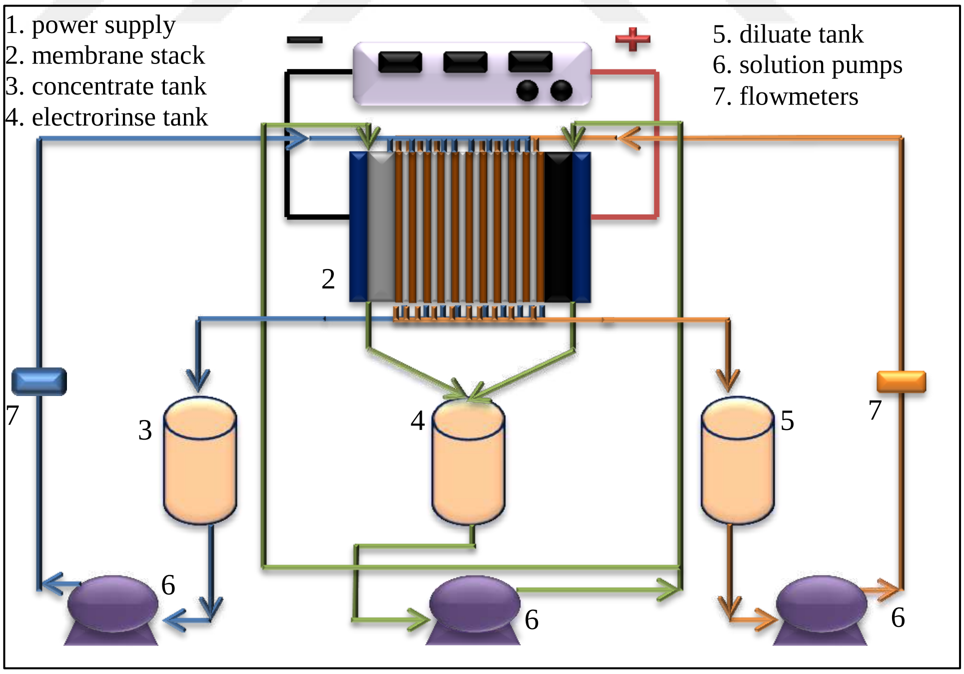

Figure 2.4 Flow diagram of Tokuyama TS-1-10 model ED system

2.2.3. EDI tests

Feed solutions used in EDI tests were obtained from three different water treatment systems: The first EDI feed solution was the permeates produced with different membranes from ground water (NF-90, NF-270, AK-BWRO and BW30) used in SEPA CF II membrane test system mentioned in section 1.2.2. The second EDI feed solution was the diluate effluent obtained from ED process applied at 10 V for NF/RO concentrates of different membranes (NF-90, NF-270, AK-BWRO and BW30) obtained from filtration tests described in section 1.2.1. The third EDI feed solution was obtained from ED process at 10 V applied for concentrate of RO process in fruit juice plant. During the production of ED diluate for EDI feed solution, ED process was stopped after reaching diluate conductivity of around 50 μS/cm. EDI experiments were carried out by using a micro-flow cell (MFC) electrodeionization system shown in Figure 2.5. It consists of three different compartments: anode, cathode and center. The active membrane area is 10.2 cm^2 (3.4 cm x 3 cm) and the electrodes include an anode (titanium with Ir/Ru coating) and a stainless steel cathode. Two different types of ion exchange membrane (Fujifilm AEM TYPE 2 - CEM TYPE 2 and Neosepta AMX-CMX) and ion exchange resins (Lewatit Ultrapure 1243 MD - 1213 MD and Purolite A500-CT175) were used. The characteristics of Neosepta membranes are given in Table 2.3. When ED diluates of NF-90, NF-270, AK-BWRO and BW30 membranes were fed to EDI, Fujifilm AEM TYPE 2 as the anion exchange membrane and Fujifilm CEM TYPE 2 as the cation exchange membrane were employed at 10 V of applied electrical potential. The ion exchange resins installed between Fujifilm AEM TYPE 2 and Fujifilm CEM TYPE 2 in mixed bed were Lewatit UltraPure 1243 MD and Lewatit UltraPure 1213 MD. The properties of these Lewatit UltraPure type resins are given in Table 2.4.

Neosepta AMX as an anion exchange membrane and Neosepta CMX were used as a cation exchange membrane were used in EDI experiments with ED diluate obtained from RO concentrate of the fruit juice plant. The resins placed between AMX and CMX membranes in mixed bed from were Purolite A500 (anion exchange resins) and Purolite CT175 (cation resins). The properties of Purolite type resins are given in Table 2.5. In all EDI experiments, a Na_2SO_4 solution with an electrical conductivity of around 500 μS/cm was used as electrode solution. All ED diluates fed to EDI were produced at 10 V of applied electrical potential. The volume of solutions used in EDI compartments (anode, cathode and center) was 1 L. In EDI, three different electrical potentials (7, 10 and

13) were tested. Electrical conductivity and pH values in all compartments were measured at each 15 min while the samples for analysis were taken each 30 min during 3 h of the experiment. Flow diagram of the EDI system is shown in Figure 2.6.

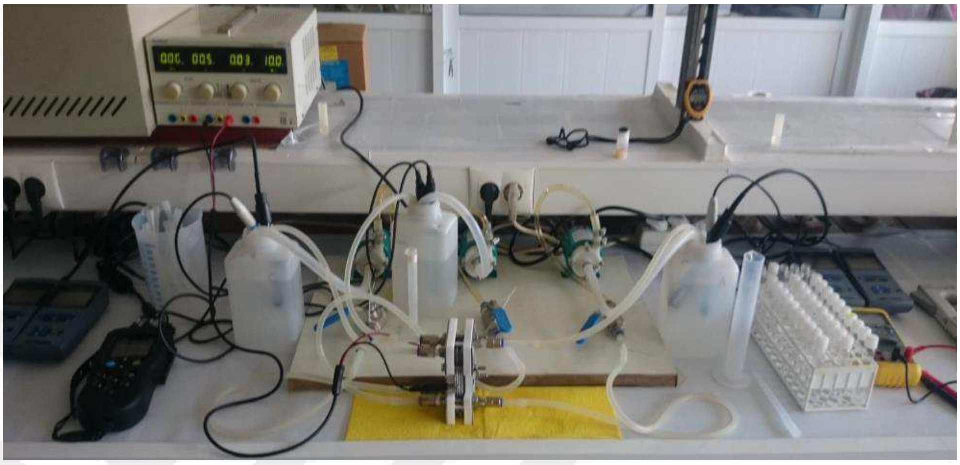

Figure 2.5 Single cell electrodeionization (EDI) system

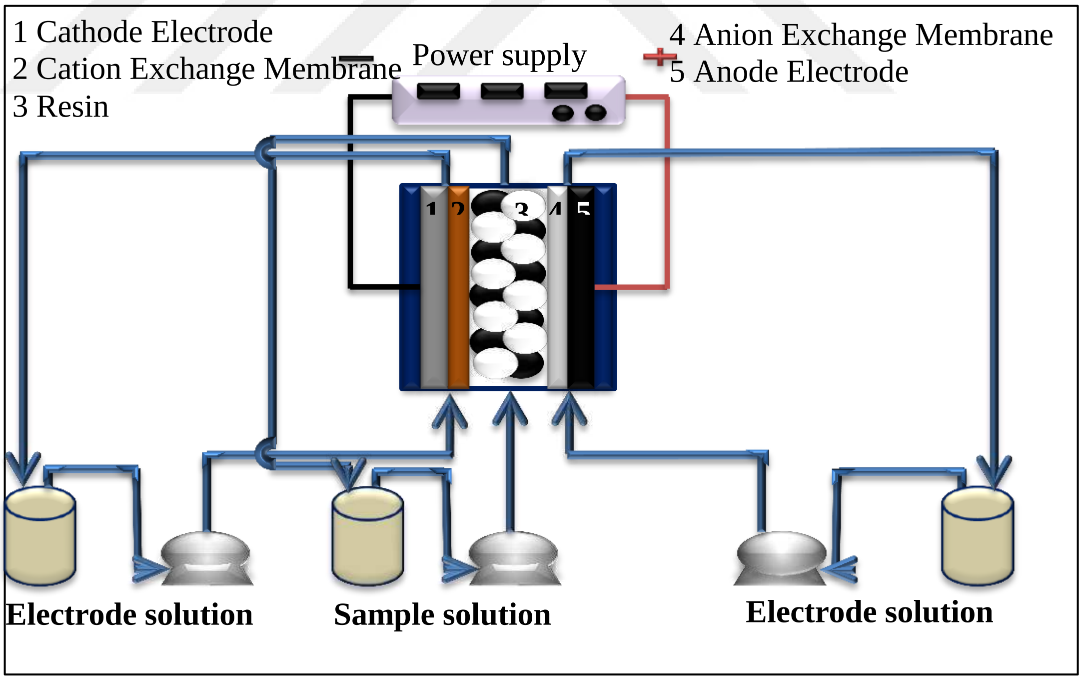

Figure 2.6 Flow Scheme of single cell electrodeionization (EDI) system

2.2.4. Measuring Equipments

2.2.4.1.pHmeter

pH values were measured by digital pH meters Hach Lange HQ40d multi meter and WTW pH 3110 (Figure 2.7 a).

2.2.4.2.Conductometer

Conductivity were measured by digital conductometer Hach Lange HQ40d multi meter and WTW Cond 3110 (Figure 2.7 b).

Salinity and TDS values were measured by Hach Lange HQ40d multi meter (Figure 2.7 c).

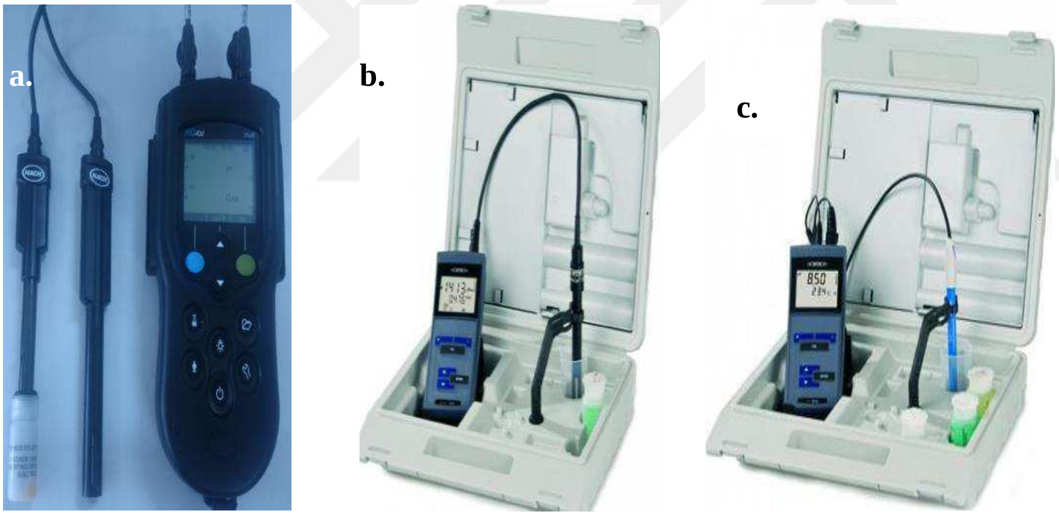

Figure 2.7 a. Hach Lange HQ40d multi meter, b.WTW Cond 3110 and c.WTW pH 3110

3. RESULTS AND DISCUSSIONS

3.1. NF/RO Tests

Ground water samples from the fruit juice plant were treated with NF and RO membranes processes using Sepa CF-II Osmonics cross flow flat sheet membrane test system. Two NF membranes such as NF-90 and NF-270, two RO membranes such as AK-BWRO and BW30 membranes were used. Applied pressure for NF membranes was 10 bar while for RO membranes applied pressure was 20 bar. The membrane permeates and concentrates steam water quality were shown in Table 3.1. Obtained result show that better permeate quality was obtained with RO membranes than with NF membranes. BWRO and AK-BWRO membranes as dense membranes were expected to give good water quality than NF membranes. NF-270 gave permeate with lower quality because of its larger molecular weight cut-off (MWCO) and big pore size. NF-270 membrane was considered to be loose membrane. The permeate streams of NF and RO membranes was feed to EDI polish them and to obtain ultrapure water while their concentrate streams were fed to ED system for water recovery.

Table 3.1 Characteristics of permeate and concentrate samples obtained after filtration of ground water with NF and RO membranes

Parameters	Unit	NF270		NF90		AK-BWRO		BW30	
		Concentrate	Permeate	Concentrate	Permeate	Concentrate	Permeate	Concentrate	Permeate
pH	-	7.89	8.08	8.48	8.64	8.18	8.93	7.90	8.48
Conductivity	µS/cm	888	382	791	93.5	744	22.5	609	37.9
TDS	mg/L	436.0	183.8	387.0	44.1	363.0	10.48	396.0	17.72
Salinity	‰	0.44	0.18	0.39	0.04	0.36	0.01	0.40	0.02
TSS	mg/L	0.00	0.00	1.00	0.00	2.00	1.00	0.00	7.00
Color	mg/L (Pt)	10.00	7.00	2.00	0.00	7.00	2.00	8.00	0.00
Turbidity	NTU	0.30	0.17	0.44	0.09	0.92	0.35	0.35	0.61
Ammonium	mg/L	0.086	0.027	0.04	0.05	0.09	0.02	4.39	<0.015
Chlorine	mg/L	44.1	25.0	37.80	3.59	40.5	1.47	48.5	0.00
Nitrite	mg/L	<0.015	<0.015	0.07	<0.015	0.017	<0.015	<0.015	<0.015
Nitrate	mg/L	17.4	13.0	12.9	2.35	13.0	1.08	17.1	1.52
COD	mg/L	<5	9.49	<5	5.39	7.72	6.44	9.04	<5
Sulfate	mg/L	61.70	1.00	36.10	0.00	39.50	0.00	51.40	0.00
Silica (SiO_2)	mg/L	30.80	22.90	16.80	2.90	17.70	2.05	21.60	4.72
Phosphate	mg/L	<0.05	<0.05	0.31	<0.05	0.25	0.39	1.03	<0.05
Bicarbonate	mg/L	0.68	0.21	0.55	0.42	0.51	0.25	0.59	0.08
TOC	mg/L	3.01	3.72	8.56	2.86	5.34	3.74	4.28	3.07

3.2. ED Tests

In ED experiments; 7, 10 and 13 V were applied as electrical potential to each feed solution. The ED feed solutions were the NF/RO concentrates of different membrane used in our laboratory with membrane filtration system and RO concentrate taken from fruit juice plant where ground water is treated with RO process with 6 RO membrane modules. During the operation, the concentrate of the first RO is fed to the second RO, the second to the third and up to the 6th RO membrane module in the same fashion. The feed of ED used in this work was the RO concentrate of the 6th RO membrane module.

3.2.1. ED studies with concentrates produced in laboratory with membrane test system Sepa CF II from ground water

3.2.1.1.ED studies with NF90 membrane concentrate

In this experiment feed solution was concentration of NF90 membrane which was produced from ground water under 10 bar pressure in laboratory with Sepa CF II membrane test system. The parameters such as pH, conductivity, TDS and salinity were measured in the diluate and concentrate compartments. The results of initial and final values of these parameters at different applied voltages were shown in Table 3.2. The conductivity and pH change vs. time plots as a function of applied electrical potential were shown in Figures 3.1-3.3. Conductivity removal reached 93% in 28 min at 10 and 13 V while similar conductivity removal was achieved in 60 min at 7 V of applied electrical potential (Figure 3.1). Conductivity change of concentrate and diluate compartments were shown in Figure 3.2. The change in conductivity showed a parallel change at all three applied electrical potentials. While the pH remains unchanged in concentrate during the experiments, the pH decreased with time in the diluate compartment (Figure 3.3). As there are not enough ions in the diluate solution to keep the electric current in the solution, the electrical potential used starts to dissociate the water into ions. It is thought that the drop in pH is mainly due to the H^+ ions generated by dissociation of water into its ions. An increase in applied electrical potential resulted in higher removal in short time as a result of driving force increase. Since 10 and 13 V achieved similar removal performance at the same time of the experiment, 10 V was chosen to be the optimum applied potential for the recovery of water from NF-90 concentrate by using ED process.

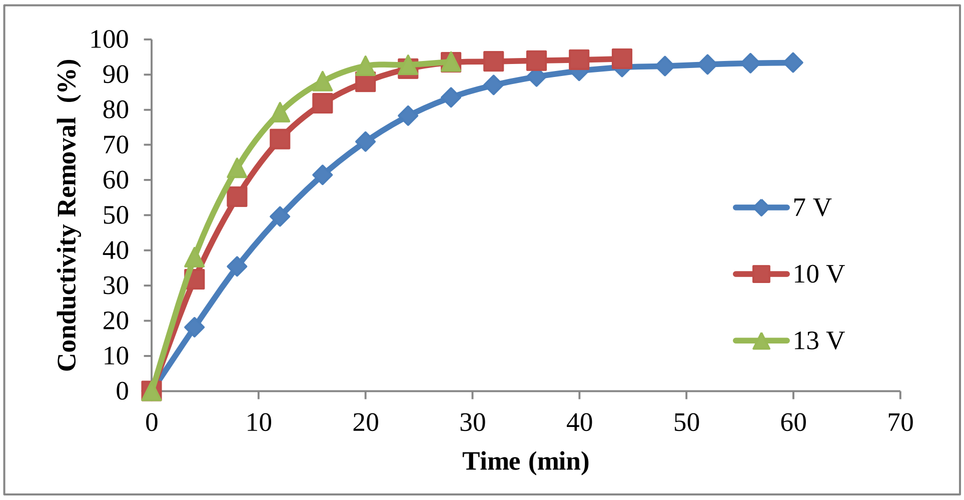

Figure 3.1 Conductivity removal from NF90 concentrate by ED as a function of applied voltage

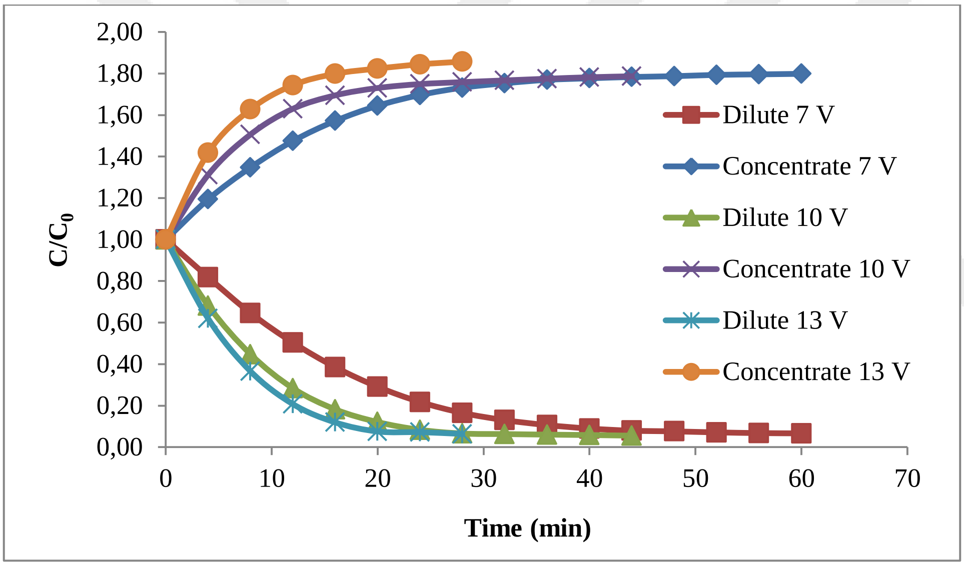

Figure 3.2 Conductivity change for NF90 concentrate as a function of applied voltage

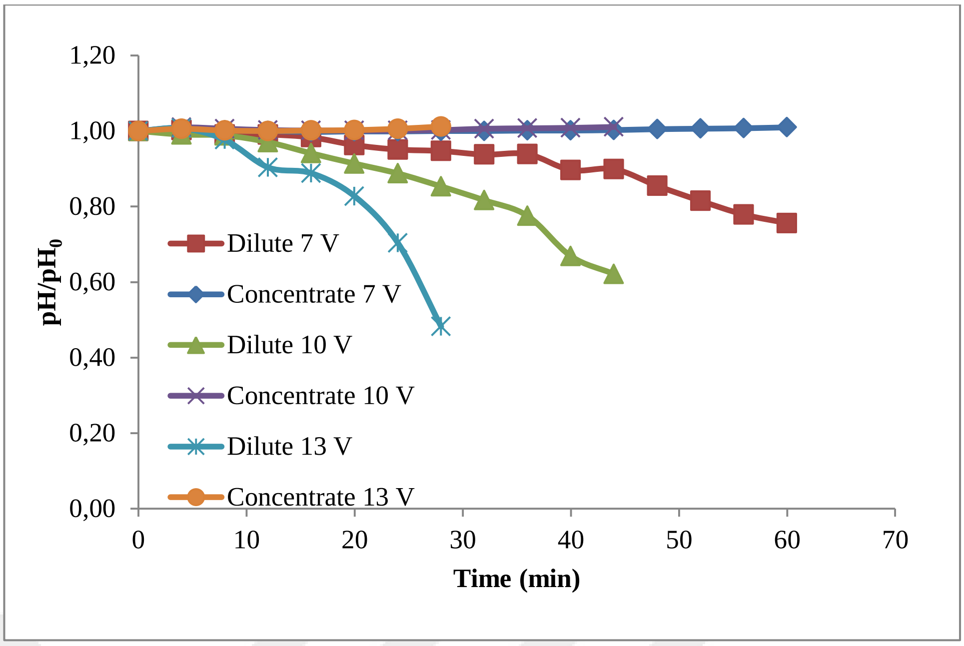

Figure 3.3 pH in diluate and concentrate streams change for NF90 concentrate as a function of applied voltage

Table 3.2 Properties of diluate and concentrate streams of NF90 concentrate during ED study

	Unit	NF90 Concentrate (ED feed)	7 V		10 V		13 V	
			Diluate	**Concentrate**	**Diluate**	**Concentrate**	**Diluate**	**Concentrate**
pH		8.48	8.29	4.38	7.88	4.16	7.60	3.92
Conductivity	μS/cm	791	978	56.7	1222	36.50	1023	43.40
TDS	mg/L	387	50.8	50.8	606	17.6	504	20.3
Salinity	‰	0.39	0.05	0.05	0.61	0.02	0.50	0.02

Table 3.3 Properties of diluate and concentrate streams of NF270 concentrate during ED study

	Unit	NF270 Concentrate (ED feed)	7 V		10 V		13 V	
			Diluate	**Concentrate**	**Diluate**	**Concentrate**	**Diluate**	**Concentrate**
pH		7.89	7.86	8.55	7.44	8.09	7.35	8.05
Conductivity	μS/cm	888.0	1400	99.50	1315	71.7	1339	50.90
TDS	mg/L	436	697	46.9	653	33.7	665	23.9
Salinity	‰	0.44	0.70	0.05	0.66	0.03	0.67	0.02

3.2.1.2.ED studies with NF270 membrane concentrate

Feed solution for this study was concentration of NF270 membrane which was produced from ground water under 10 bar pressure in laboratory with Sepa CF II membrane test system. ED was applied a different applied potential (7, 10 and 13) for NF-270 concentrate produced through Sepa CF II membrane test system from ground water. Conductivity removal, conductivity change and pH change plots were given in Figures 3.4-3.6 The results of initial and final values of these parameters at different applied voltages were shown in Table 3.3. Maximum conductivity removal were 86% in 44 min, 89% in 32 min and 91% in 24 min at 7, 10 and 13 V of applied electrical potential, respectively (Figure 3.4). The conductivity change in the concentrate and diluate compartments were shown in Figure 3.5. During the experiments, a decrease in pH by time was observed in diluate compartment while in concentrate compartment pH was almost constant (Figure 3.6). The higher conductivity removal observed at higher voltage in a short time was due to the increase of driving force. In addition to ion removal such bicarbonate from diluate solution, water dissociation into H^+ and OH^- ions was thought to cause the pH decrease in diluate compartment. For NF-270 concentrate treatment with ED, an electrical potential of 10 V was found to be enough to achieve a maximum conductivity removal as 89% in 32 min.

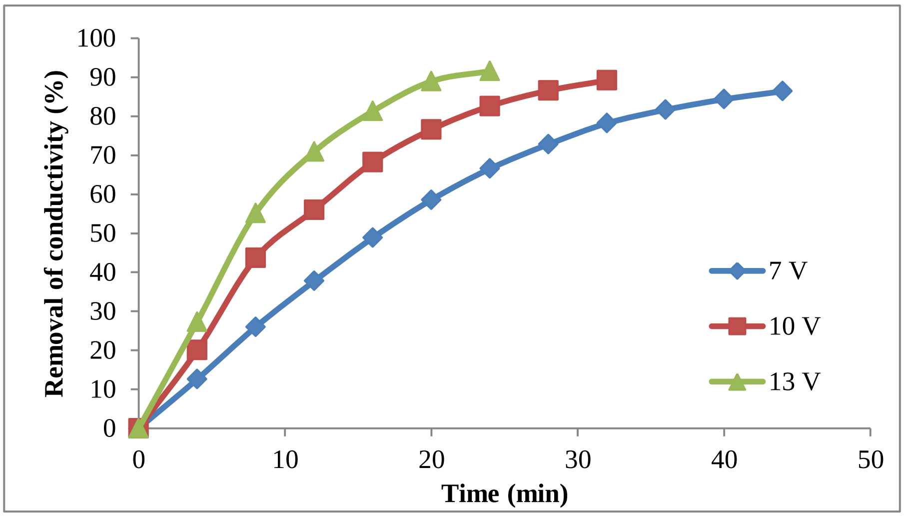

Figure 3.4 Conductivity removal from NF270 concentrate by ED as a function of applied potential

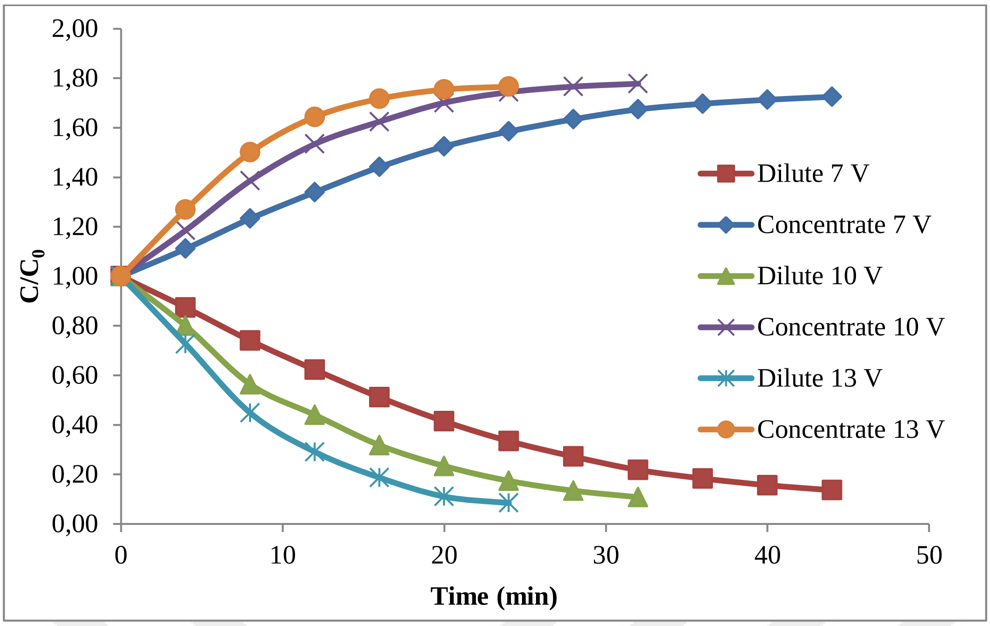

Figure 3.5 Conductivity change for NF270 concentrate by ED as a function of applied voltage

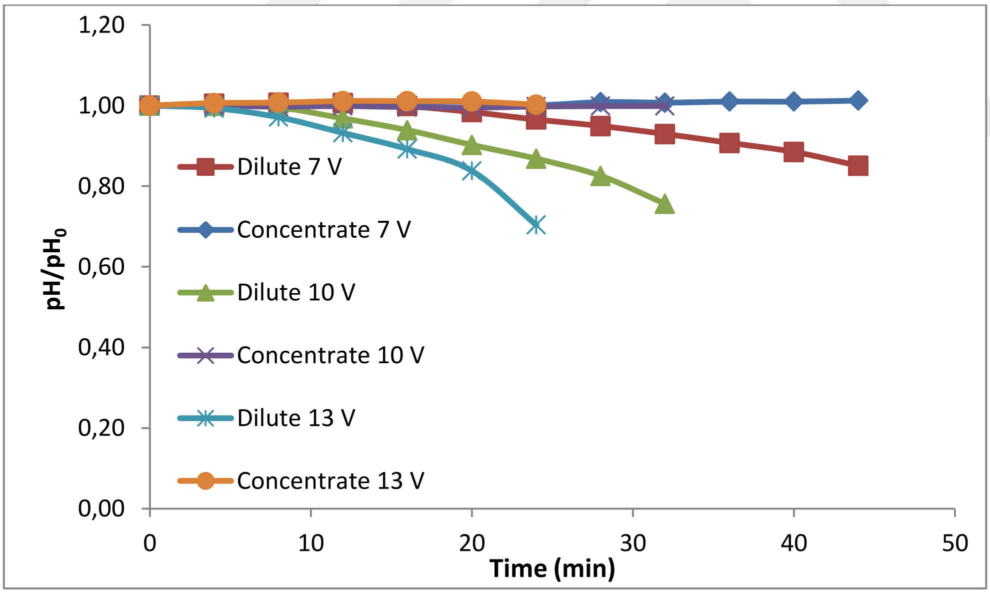

Figure 3.6 pH in diluate and concentrate streams change for NF270 concentrate by ED as a function of applied voltage

3.2.1.3.ED studies with AK-BWRO membrane concentrate

ED was applied for AK-BWRO membrane concentrate produced with Sepa CF II membrane test system by using ground water of fruit juice plant under 20 bar pressure. Effect of applied electrical potential ED performance was investigated at 7, 10 and 13 V. Conductivity removal, conductivity change and pH change plots were given in Figures 3.7-3.9. The results of initial and final values of these parameters at different applied voltages were shown in Table 3.4. The conduction removal was 90% at 7 and 10 V in 52 min and 24 min, respectively. At 13 V of applied electrical potential, a conductivity removal of 93% was achieved in 20 min (Figure 3.7). The higher removal efficiency in short time with an increase of applied electrical potential was due to the increase of driving force as explained previously. The reasons for pH decrease in diluate compartment for ED applied on NF-90 and NF-270 concentrates stand for AK-BWRO concentrate as well.

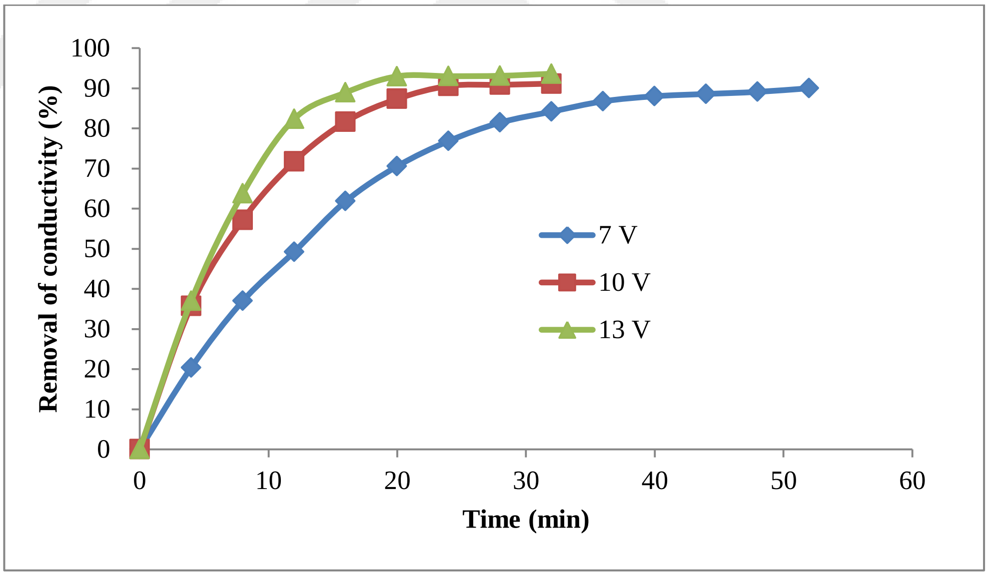

Figure 3.7 Conductivity removal from AK-BWRO concentrate by ED as a function of applied potential

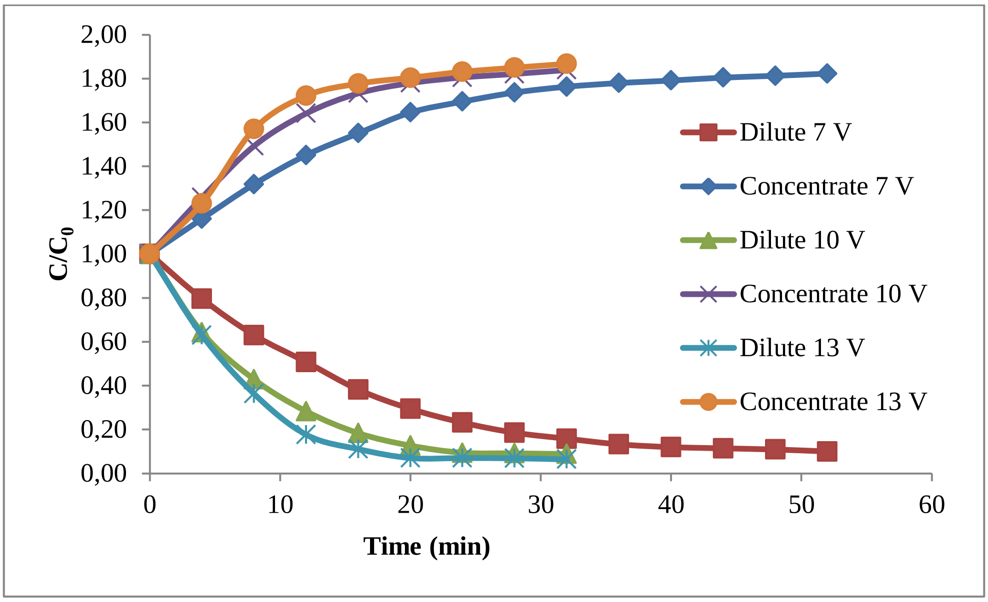

Figure 3.8 Conductivity change for AK-BWRO concentrate during ED process as a function of potential applied

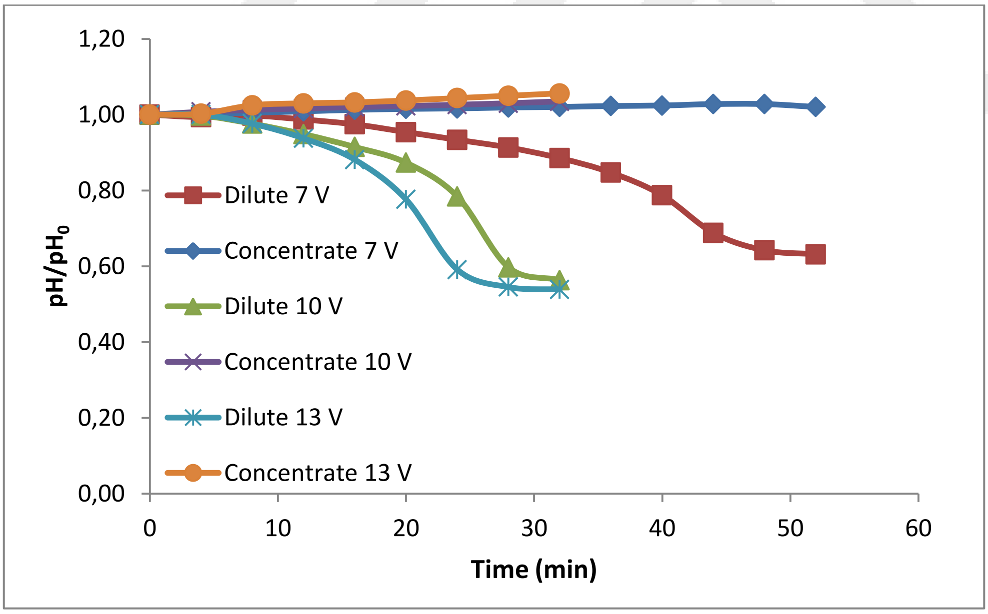

Figure 3.9 pH change for AK-BWRO concentrate during ED process as a function of potential applied

Table 3.4 Properties of diluate and concentrate streams of AK-BWRO concentrate during ED study

	Unit	AK-BWRO Concentrate (ED feed)	7 V		10 V		13 V	
			Diluate	**Concentrate**	**Diluate**	**Concentrate**	**Diluate**	**Concentrate**
pH		8.18	7.72	8.17	8.49	6.88	8.54	4.53
Conductivity	µS/cm	744.0	1007	58.70	1074	59.40	1101	47.40
TDS	mg/L	363	496	27.50	530	27.9	544.	21.7
Salinity	‰	0.36	0.50	0.03	0.53	0.03	0.25	0.02

Table 3.5 Properties of diluate and concentrate streams of BW30 concentrate during ED study

	Unit	BW30 Concentrate (ED feed)	7 V		10 V		13 V	
			Diluate	**Concentrate**	**Diluate**	**Concentrate**	**Diluate**	**Concentrate**
pH		7.90	8.11	8.85	8.04	8.40	8.19	8.56
Conductivity	µS/cm	609.0	1251	102.5	1295	79.70	1320	42.20
TDS	mg/L	396	621	48.3	643	37.5	656	19.8
Salinity	‰	0.40	0.62	0.05	0.65	0.04	0.66	0.02

3.2.1.4.ED studies with BW30 membrane concentrate

ED was applied for BW30 membrane concentrate produced by Sepa CF II membrane test system by using ground water of fruit juice plant under 20 bar pressure. Effect of applied electrical potential ED performance was investigated at 7, 10 and 13 V. Conductivity removal, conductivity change and pH change plots were given in Figures 3.10-3.12. The results of initial and final values of these parameters at different applied voltages were shown in Table 3.4. The electrical conductivity removal was 90% at 7 and 10 V in 52 min and 24 min, respectively. At 13 V of applied electrical potential, a conductivity removal of 93% was achieved in 20 min (Figure 3.7). The higher removal efficiency in short time with an increase of applied electrical potential was due to the increase of driving force as explained previously. pH decrease in diluate compartment was observed for BW30 concentrate as well.

When the electrical potentials applied to the BW30-RO concentrate streams during component ED were 7, 10, and 13 V, the resulting conductivity removal, conductivity change, and pH change graphs were shown in Figures 3.10-3.12, respectively. The conductivity removal was 84% after 40 minutes for 7 V, 89% after 28 minutes for 10 V and 92% after 28 minutes for 13 V (Figure 3.10). As observed in the other ED studies with concentrated samples of other membranes, the conductivity variation for the concentrate and diluate streams also showed similarity in this study as well. In the same way, the pH change is constant in the concentrate part as in other studies, while it decreases with time in the diluated part (Figure 3.12).

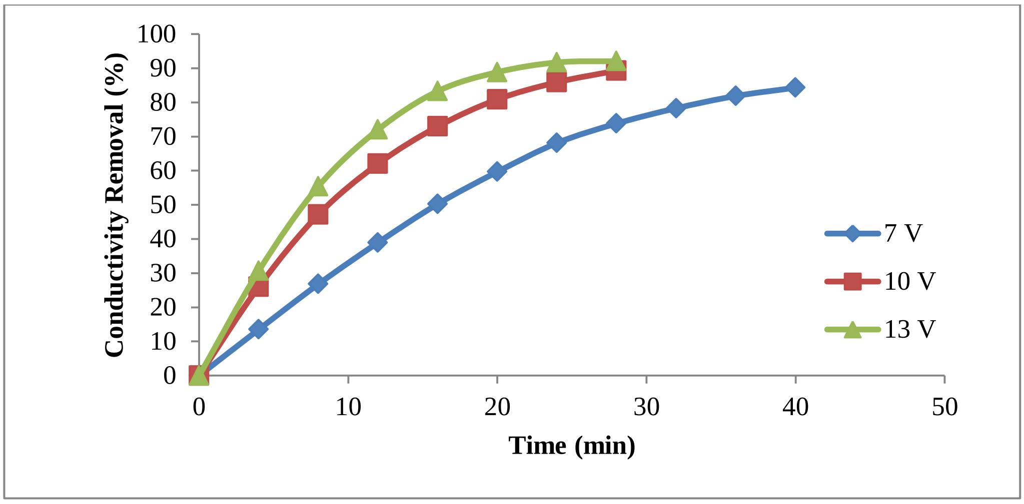

Figure 3.10 Conductivity removal from BW30 concentrate by ED process as a function of electrical potential applied

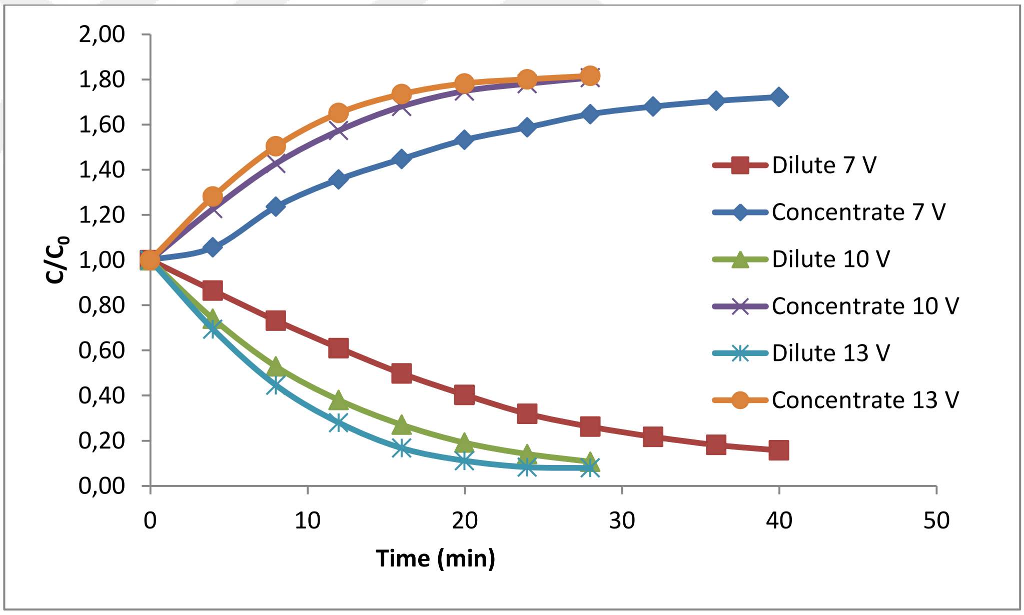

Figure 3.11 Conductivity in diluate and concentrate streams change for BW30 concentrate for all voltage during ED process

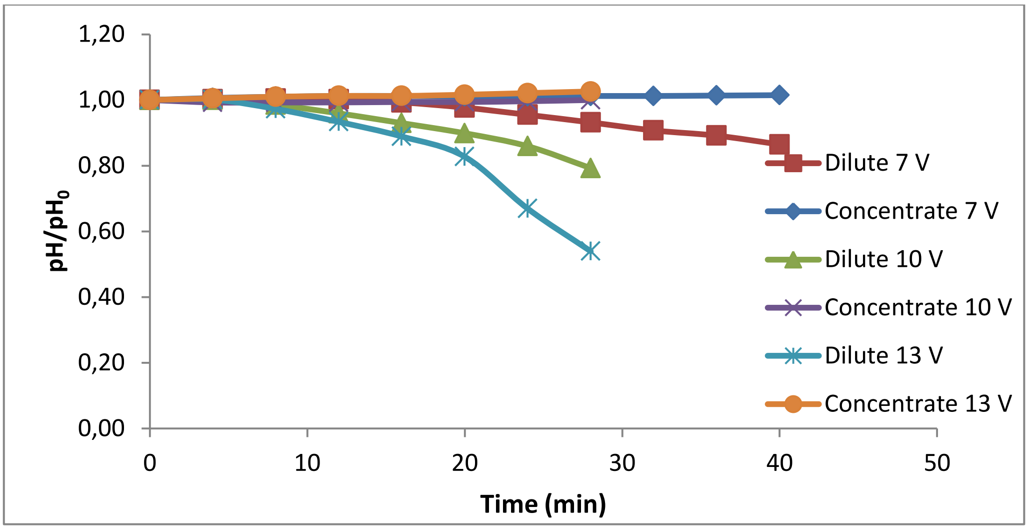

Figure 3.12 pH change for BW30 concentrate during ED process as a function of electrical potential applied

3.2.2. ED studies with the concentrate streams of RO plant in fruit juice plant

ED experiments were done using the concentrate of RO treatment system of the fruit juice plant. The pH, conductivity, TDS and salinity values of the diluate and concentrate compartments were given in Table 3.7. Conductivity removal, conductivity change and pH change plots obtained by ED tests at various electrical potentials (7, 10 and 13 V) were given in Figures 3.13-3.15. The conductivity removal was 94% in 84 minutes at 7 V. A 98% of conductivity removal was obtained at 10 V and 13 V after at 80 and 52 minutes, respectively (Figure 3.13). The conductivity variation for concentrated and diluated streams were shown in Figure 3.14. pH change in diluate and concentrate streams are given in Figure 3.15.

Table 3.6 Properties of diluate and concentrate streams during ED tests in which RO concentrate of treatment plant is used as feed

	Unit	**RO concentrate (ED feed)**	**7 V**		**10 V**		**13 V**	
			Diluate	**Concentrate**	**Diluate**	**Concentrate**	**Diluate**	**Concentrate**
pH		7.35	7.41	8.12	5.62	6.92	7.34	7.58
Conductivity	µS/cm	4843	279	8920	100.2	7420	82.9	7860
TDS	mg/L	2.55	4.32	133.9	3.98	51.50	4.23	39
Salinity	‰	2.61	0.13	4.46	0.01	4.10	0.04	4.37

Table 3.7 Feed and product water characteristics in EDI studies made with ED diluated products of concentrates produced in laboratory from ground water

	ED Diluate (10 V) of NF90 Concentrate		**ED Diluate (10 V) of NF270 Concentrate**		**ED Diluate (10 V) of AK-BWRO Concentrate**		**ED Diluate (10 V) of BW30 Concentrate**	
	EDI Feed	**Product water**	**EDI Feed**	**Product water**	**EDI Feed**	**Product water**	**EDI Feed**	**Product water**
pH	8.37	6.78	8.31	5.54	7.54	7.3	8.99	6.41
Conductivity (µS/cm)	36.0	1.65	42.0	8.87	32.0	9.61	49.8	7.73
TDS (mg/L)	16.9	0.78	19.7	4.17	15.0	4.52	23.4	3.63
Salinity (‰)	0.02	0	0.02	0	0.02	0	0.02	0

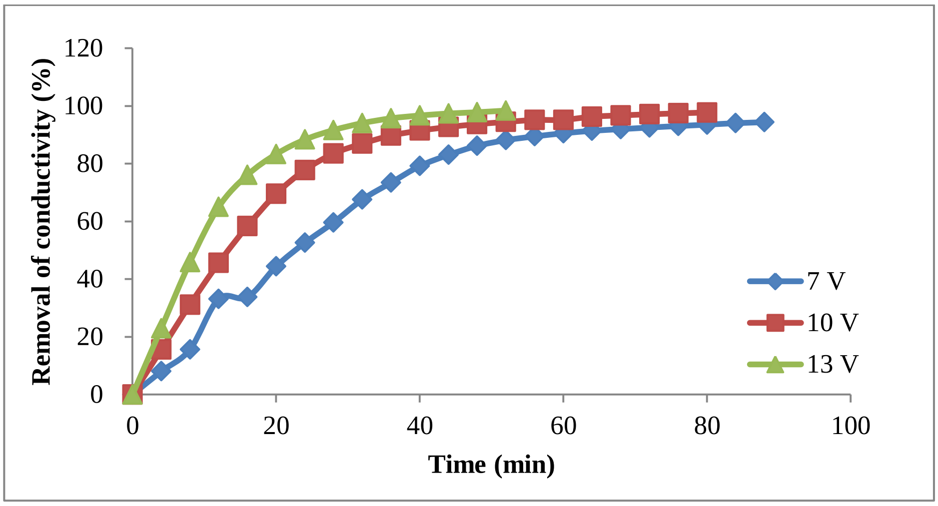

Figure 3.13 Conductivity removal for RO treatment system concentrate ED

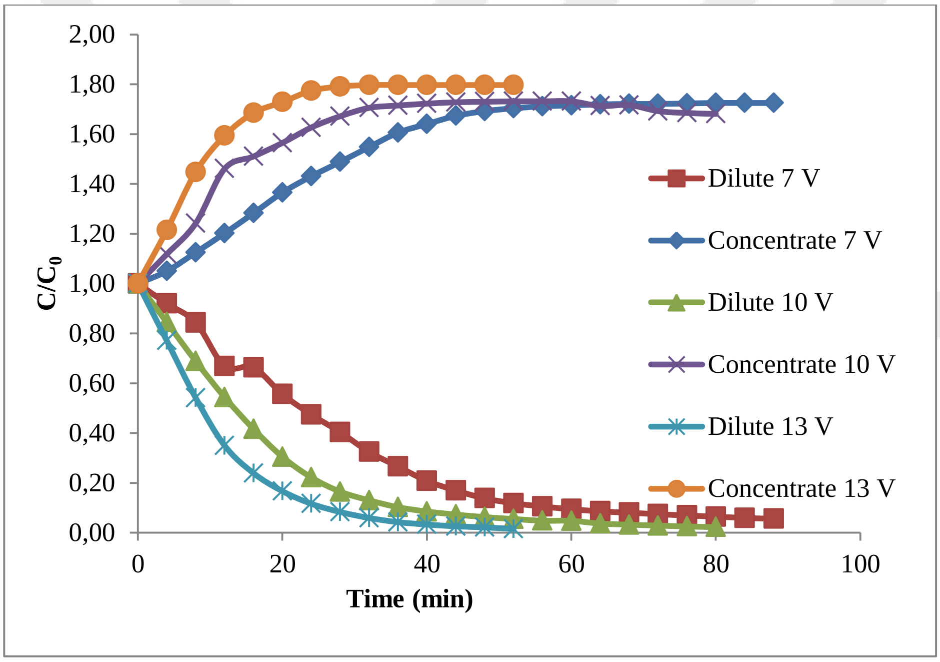

Figure 3.14 Conductivity change for RO treatment system concentrate during ED as a function of electrical potential applied

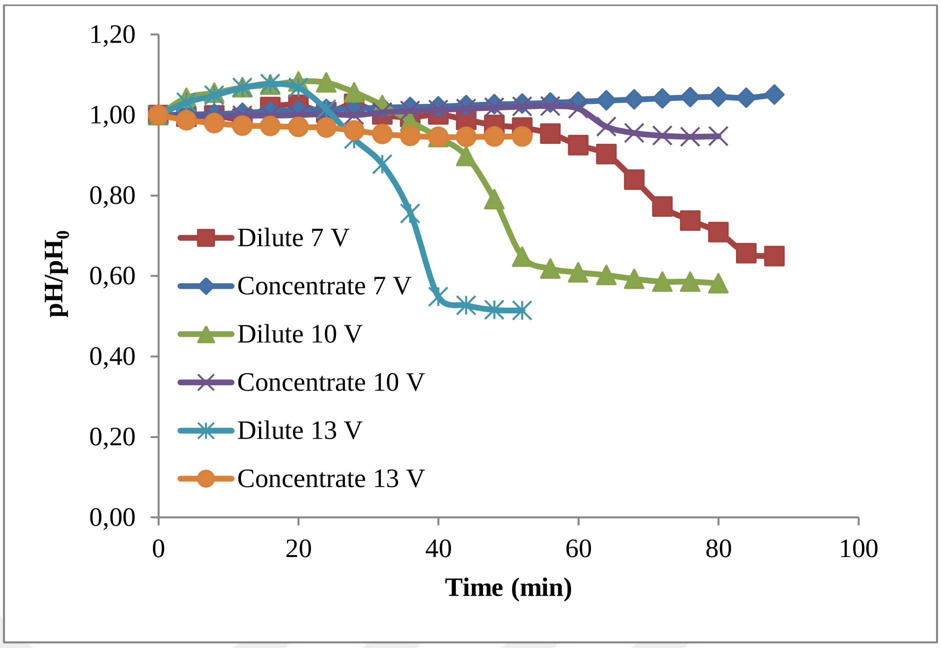

Figure 3.15 pH change in diluate and concentrate streams during ED as a function of electrical potential applied

3.3. EDI Tests

3.3.1. EDI studies with ED diluates of concentrate streams of various membranes which are obtained in the laboratory

Diluate part produced by ED system (10 V) from concentrates of NF90, NF270, AK-BWRO and BW30 membranes from ground water were used in these studies. Feed and product water characteristics are given in Table 3.7. Fujifilm AEM TYPE 2 and Fujifilm CEM TYPE 2 membranes along with Lewatit UltraPure 1243 MD and Lewatit UltraPure 1213 MD resins were used in these studies. During EDI process, 10 V of electrical potential was applied.

3.3.1.1.EDI studies with ED diluate of NF90 concentrate stream

The concentrate stream was collected by passing ground water of DİMES through a NF90 membrane using a laboratory scale Sepa CF II membrane test system at 10 bar pressure. After ED process carried out using this concentrate sample at 10 V, diluate was used as feed water for EDI work.

Removal of conductivity, conductivity change, pH change and electrical potential and current change were given in Figures 3.16-3.19.Feed and product water characteristics were given in Table 3.8. As shown in Figure 3.16, the conductivity removal reached 95% after 75 min and then remained constant.

Table 3.8 Characteristics of feed and product water of EDI

	Unit	**ED Diluate of NF90 concentrate (obtained at 10 V)**	
		EDI Feed	**EDI Product**
pH		8.37	6.78
Conductivity	µS/cm	36.00	1.65
TDS	mg/L	16.92	0.78
Salinity	‰	0.02	0

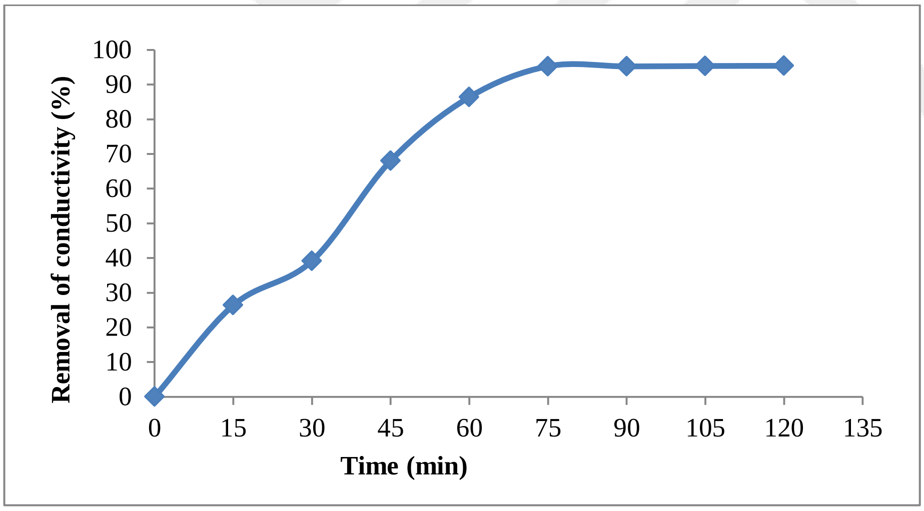

Figure 3.16 Removal of conductivity by EDI from ED diluate of NF90 concentrate

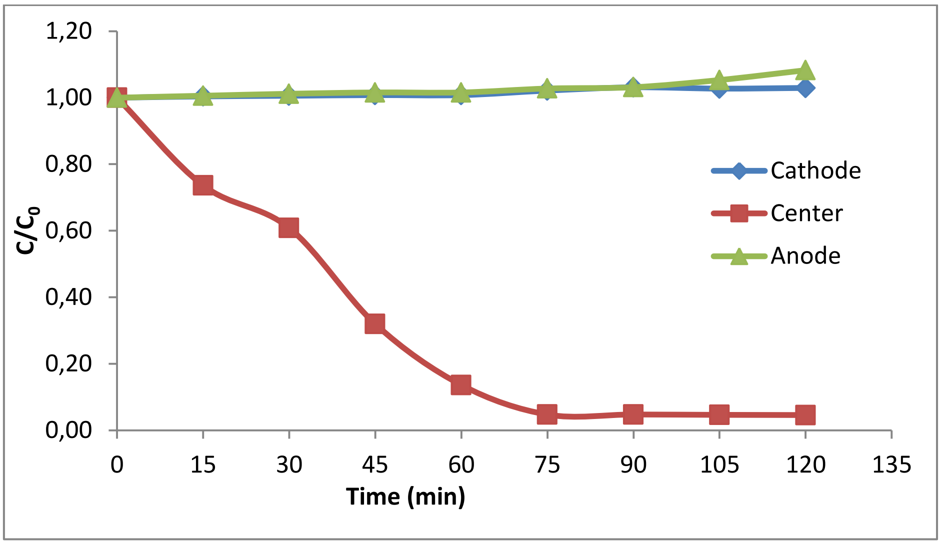

Figure 3.17 Conductivity changes during EDI for ED diluate of NF90 concentrate

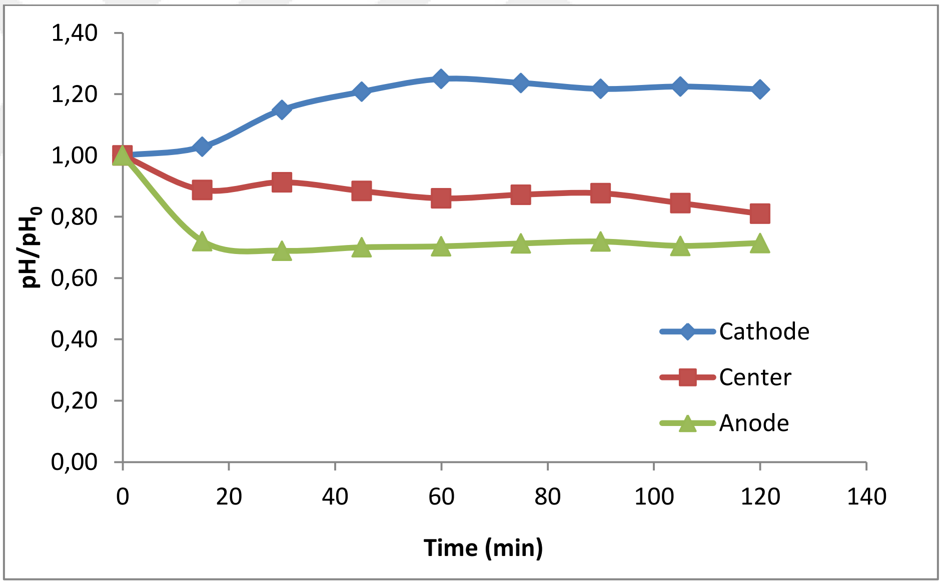

Figure 3.18 pH changes for EDI in cathode, center and anode compartments for ED diluate of NF90 concentrate

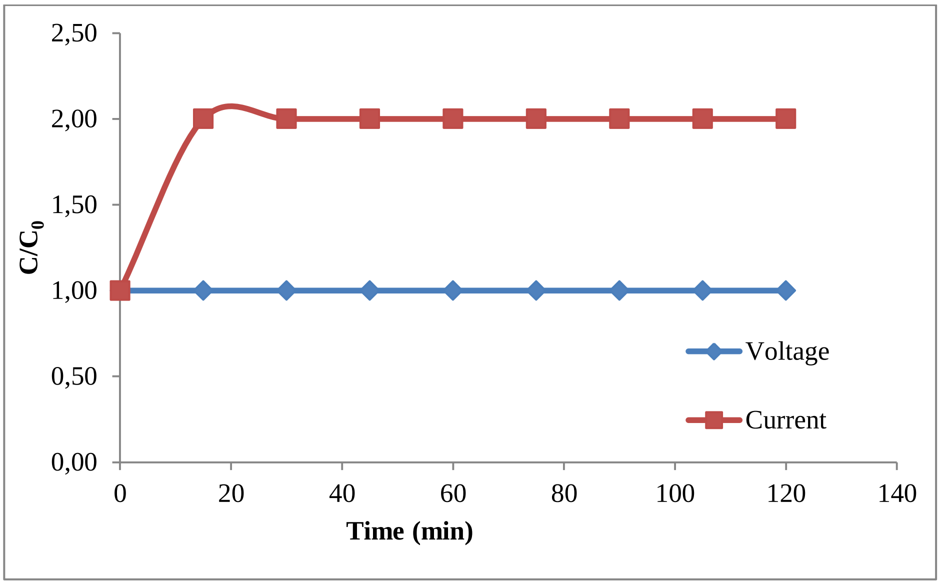

Figure 3.19 Voltage and current changes during EDI for ED diluate of NF90 concentrate

3.3.1.2.EDI studies with ED diluate of NF270 concentrate

A NF was performed by passing ground water of DİMES through a laboratory scale Sepa CF II membrane test system using NF270 membrane at 10 bar of pressure. Concentrate stream of NF270 membrane was then fed to ED system. The diluate solution was used as feed for EDI work. Removal of conductivity, conductivity change, pH change and electrical potential and current change were given in Figures 3.20 -3.23. Feed and product water characteristics were given in Table 3.9, respectively. As shown in Figure 3.20, conductivity removal is shown in Figure 15, which reached 78% after 105 minutes.

Table 3.9 Characteristics of feed and product water of EDI

	Unit	**ED Diluate of NF270 concentrate (obtained at 10 V)**	
		EDI Feed	**EDI Feed**
pH		8.31	5.54
Conductivity	µS/cm	42.0	8.87
TDS	mg/L	19.7	4.17
Salinity	‰	0.02	0

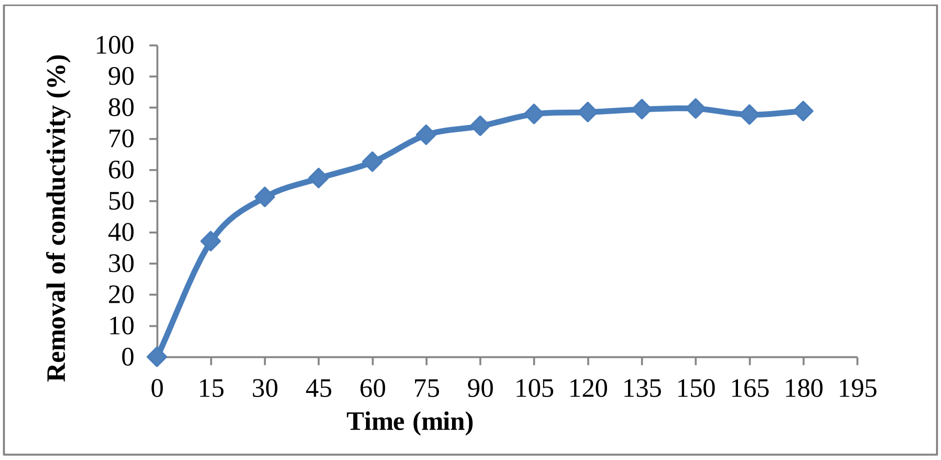

Figure 3.20 Removal of conductivity during EDI study for ED diluate of NF270 concentrate

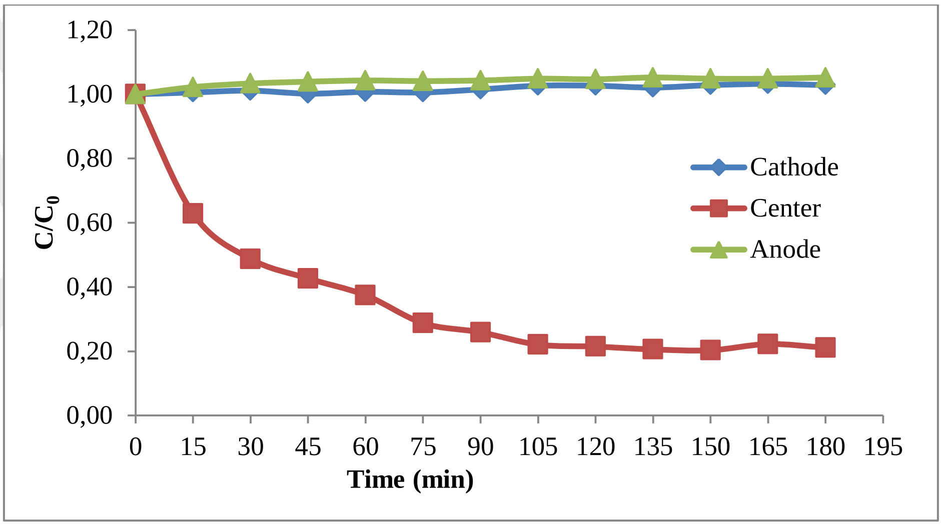

Figure 3.21 Conductivity changes for EDI study of 10 V ED diluated NF270 concentrate

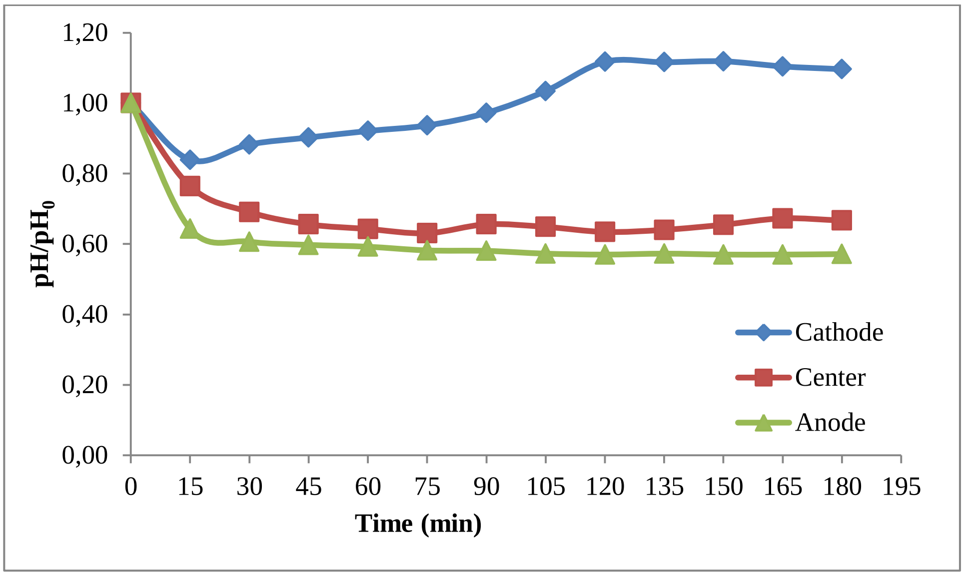

Figure 3.22 pH changes in cathode, center and anode compartments during EDI study for ED diluate of NF270 concentrate

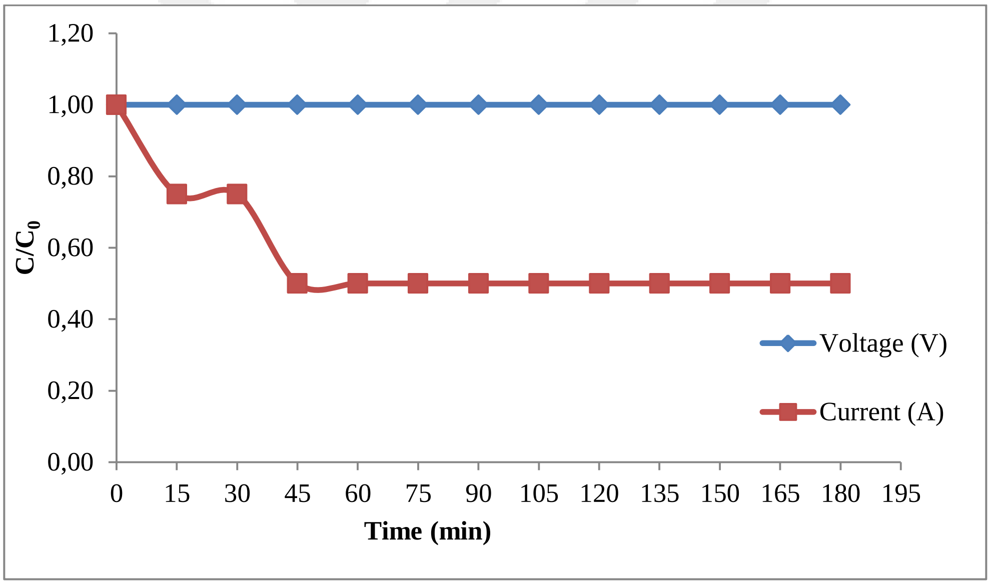

Figure 3.23 Voltage and current changes during EDI study for ED diluate of NF270 concentrate

3.3.1.3.EDI studies with ED diluate of AK-BWRO concentrate

Produced concentrate by using AK-BWRO membrane at 20 of bar pressure was collected and used as feed of ED system. After applying 10 V of electrical potential in the ED system the diluate part solution conductivity reached a value less than 50 µS/cm. This diluate solution was used as feed water for EDI work. During EDI process, 10 V of electrical potential was applied to the system. Removal of conductivity, conductivity change, pH change and electrical potential and current change were given in Figures 3.25 - 3.28. Feed and product water characteristics were given in Table 3.10, respectively. As shown in Figure 3.25, the conductivity removal reached 69% after 75 min and remained constant then after

Table 3.10 Characteristics of feed and product water of EDI of 10 V ED diluated AK-BWRO concentrate

	Unit	**ED Diluate of AK-BWRO concentrate (obtained at 10 V)**	
		EDI Feed	**EDI Product**
pH		7.54	7.3
Conductivity	µS/cm	32.00	9.61
TDS	mg/L	15.04	4.52
Salinity	‰	0.02	0

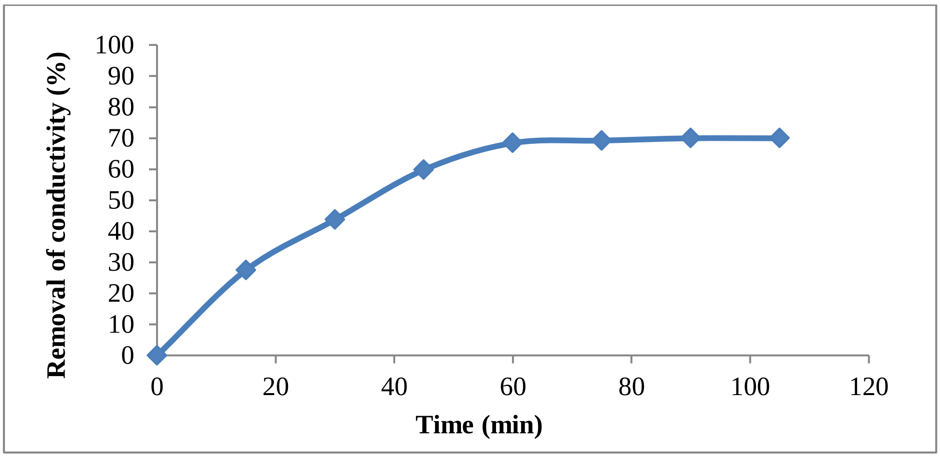

Figure 3.24 Removal of conductivity during EDI study for ED diluate of AK-BWRO concentrate

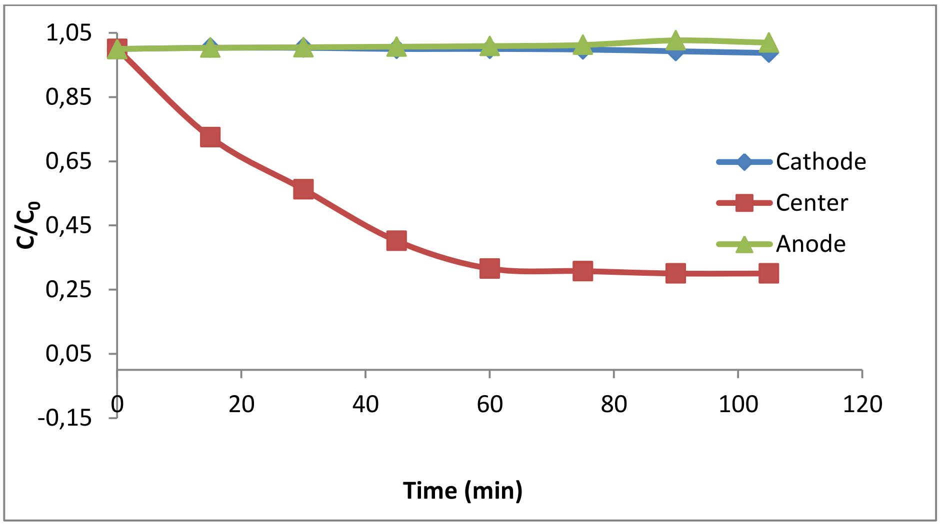

Figure 3.25 Conductivity changes during EDI study for ED diluate of AK-BWRO concentrate

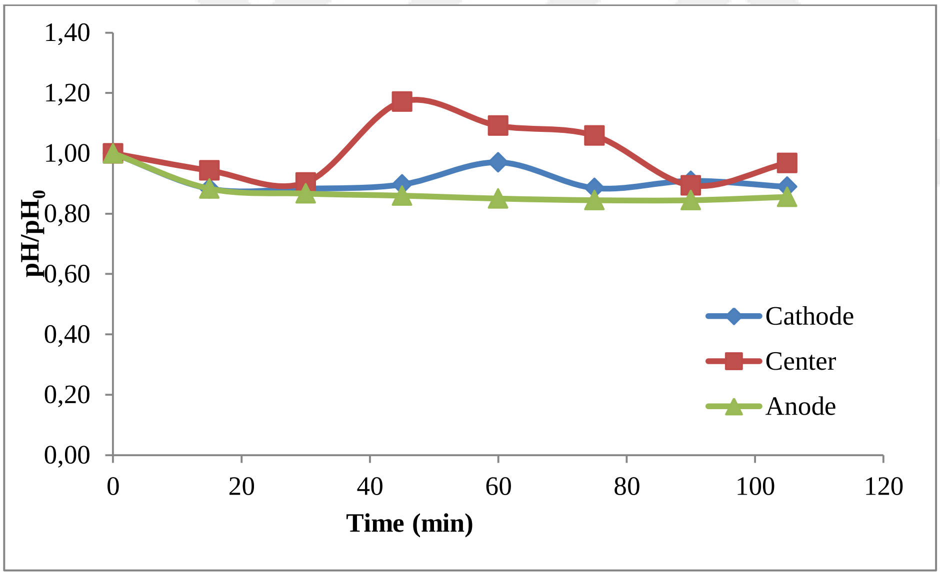

Figure 3.26 pH changes for cathode, center and anode compartments during EDI study for ED diluate of AK-BWRO concentrate

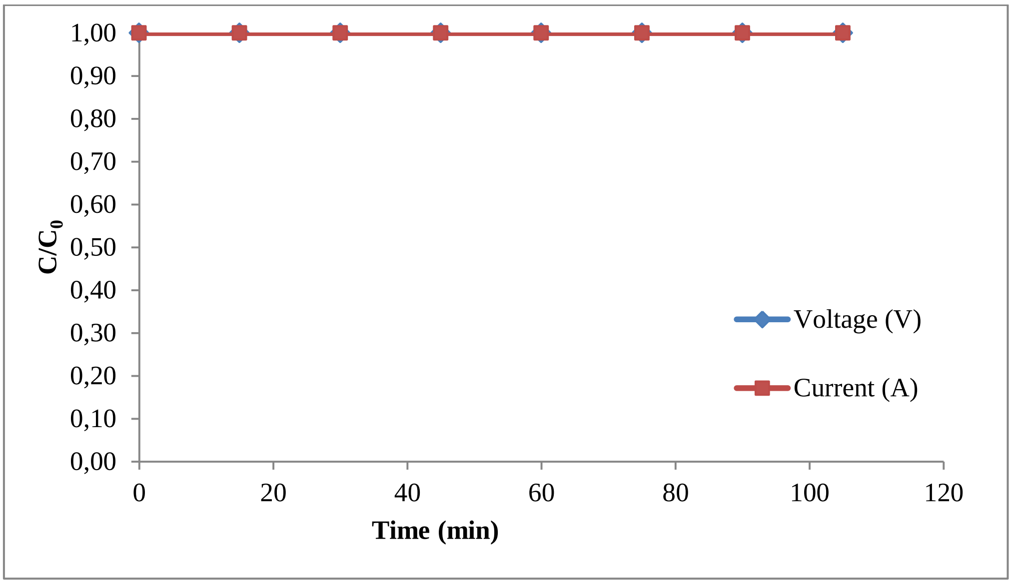

Figure 3.27 Voltage and current changes during EDI study for ED diluate of AK-BWRO concentrate

3.3.1.4.EDI studies with ED diluate of BW30 concentrate

The ground water passed through the BW30 membrane under a 20 bar of pressure and the concentrate stream was collected. By applying 10 V of electrical potential to this concentrate solution by the ED system, the diluate part conductivity in ED system as reduced less than 50 µS/cm. Then this diluate solution was used as feed EDI test. Figures 3.29- 3.32 showed removal of conductivity, conductivity change, pH change and electrical potential and current change respectively. Feed and product water characteristics for EDI were given in Table 3.11. The conductivity removal was reached 85% at the end of 180 min (Figure 3.29).

Table 3.11 Characteristics of feed and product water during EDI study for ED diluate of BW30 concentrate

	Unit	**ED Diluate of BW30 concentrate (obtained at 10 V)**	
		EDI Feed	**EDI Product**
pH		8.99	6.41
Conductivity	µS/cm	49.80	7.73
TDS	mg/L	23.41	3.63
Salinity	‰	0.02	0

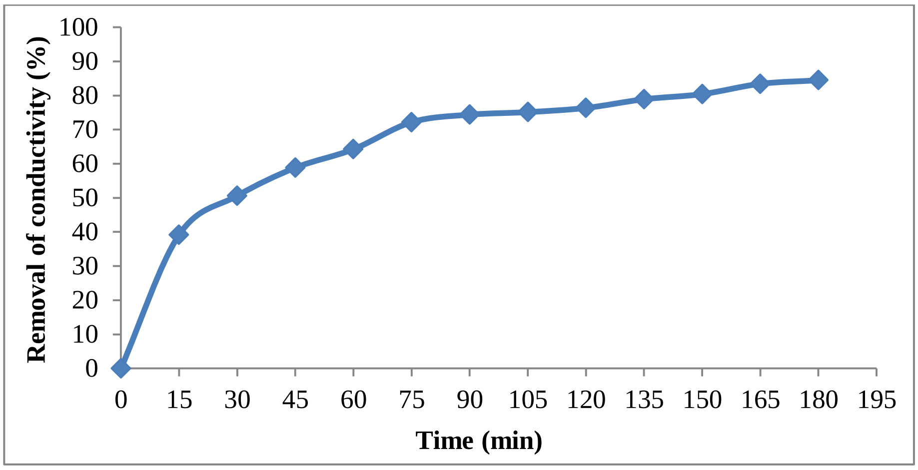

Figure 3.28 Removal of conductivity during EDI study for ED diluate of BW30 concentrate

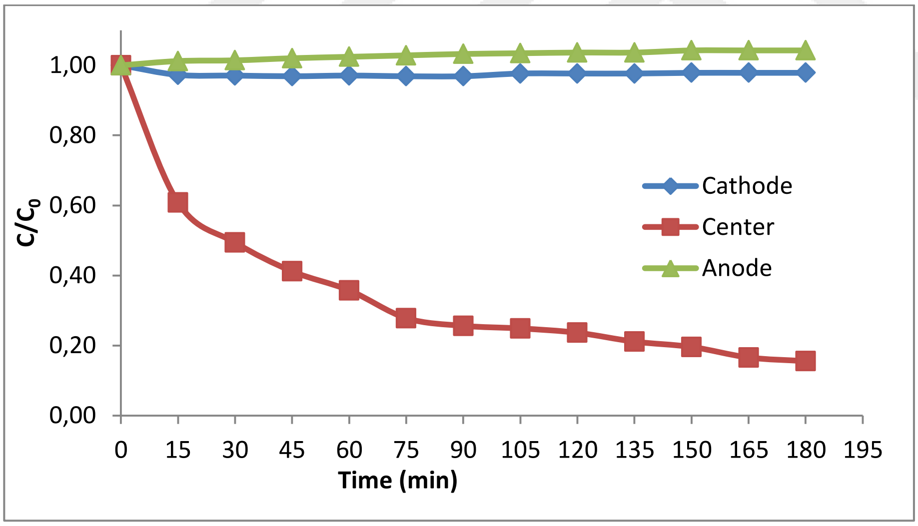

Figure 3.29 Conductivity changes for EDI study of 10 V ED diluated BW30 concentrate

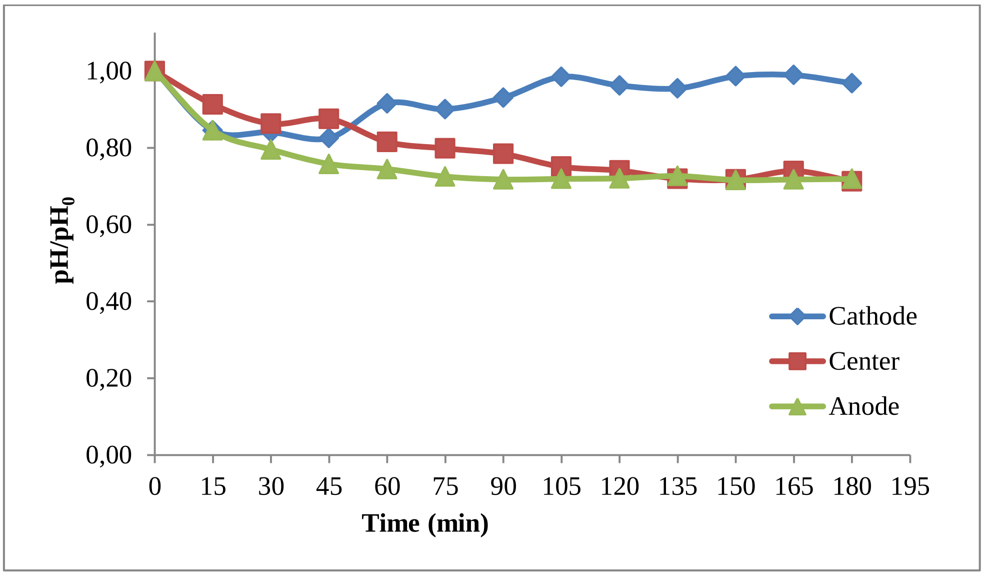

Figure 3.30 pH changes for EDI study of 10 V ED diluated BW30 concentrate

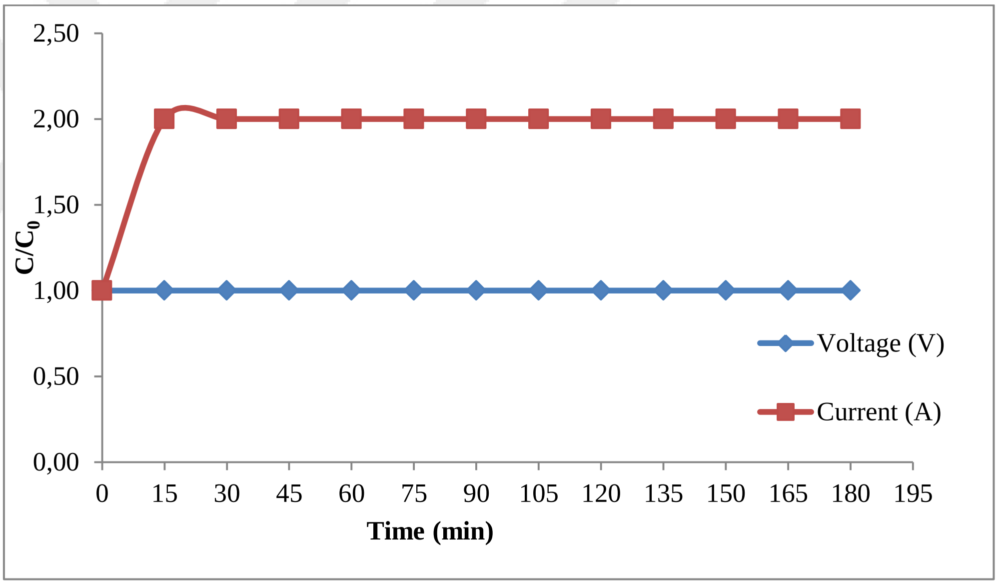

Figure 3.31 Voltage and current changes during EDI study for ED diluate of BW30 concentrate

3.3.2. EDI studies with permeates of various membranes obtained from ground water

In these studies, the permeates of NF90, NF270, AK-BWRO and BW30 membranes obtained using ground water were used as the EDI system. Feed and product water characteristics were given in Table 3.12.

Neosepta AMX and Neosepta CMX membranes along with Purolite A500 and Purolite CT175 resins were used in these studies. A 7 V of electrical potential was applied in all studies.

Table 3.12 Characteristics of feed and product water in EDI studies

		pH	Conductivity	TDS	Salinity
Unit			**µS/cm**	**mg/L**	**‰**
NF-90 permeate	EDI Feed	8.44	118	54.4	0.02
	EDI Product	4.52	19.22	9.06	0
NF-270 permeate	EDI Feed	5.89	343	180.3	0.02
	EDI Product	4.72	51.1	23.5	0
AK-BWRO permeate	EDI Feed	8.66	67.4	31.1	0.02
	EDI Product	4.63	4.11	1.85	0
BW30 permeate	EDI Feed	4.08	42.3	19.46	0.02
	EDI Product	5.11	1.85	0.84	0.01

3.3.2.1.EDI studies with NF90 permeate

The ground water from the fruit juice factory was filtered with a NF90 membrane through SEPA CF II membrane test system. During this filtration a pressure of 10 bar was applied. The permeate generated by filtration was collected and used as feed solution in the EDI study. Electrical potential of 7 V was applied in the EDI system. Removal of conductivity, conductivity change, pH change and electrical potential and current change plots were given in Figures 3.33- 3.36 respectively. Feed and product water characteristics were given in Table 3.13. Conductivity removal from NF90 permeate of ground water reduction reached 83% at the end of 180 min (Figure 3.33).

Table 3.13 Characteristics of feed and product water of NF90 permeate during EDI studies

Parameters	Unit	EDI feed (NF90 permeate)	Product water (EDI diluate)
pH	-	8.44	4.52
Conductivity	µS/cm	118	19.22
TDS	mg/L	54.4	9.06
Salinity	‰	0.06	0.01

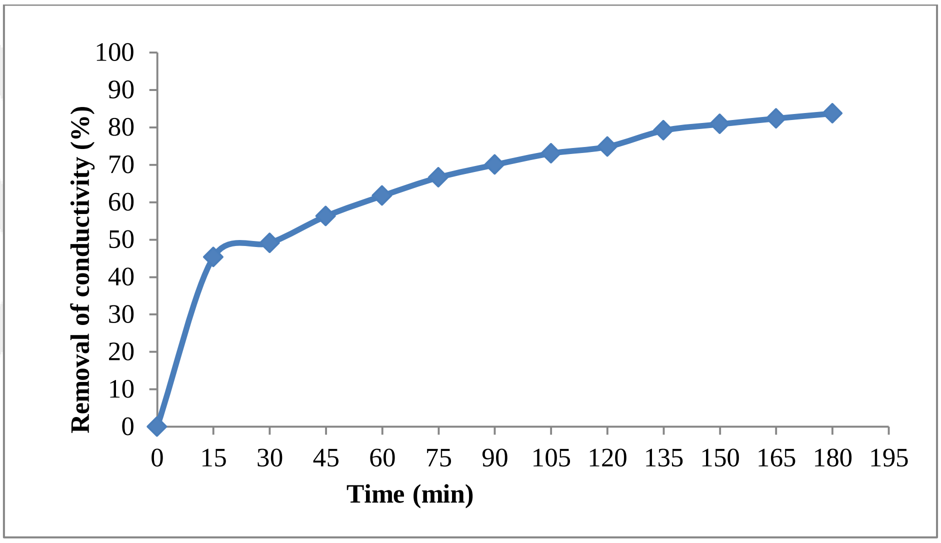

Figure 3.32 Removal of conductivity from NF90 permeate during EDI study

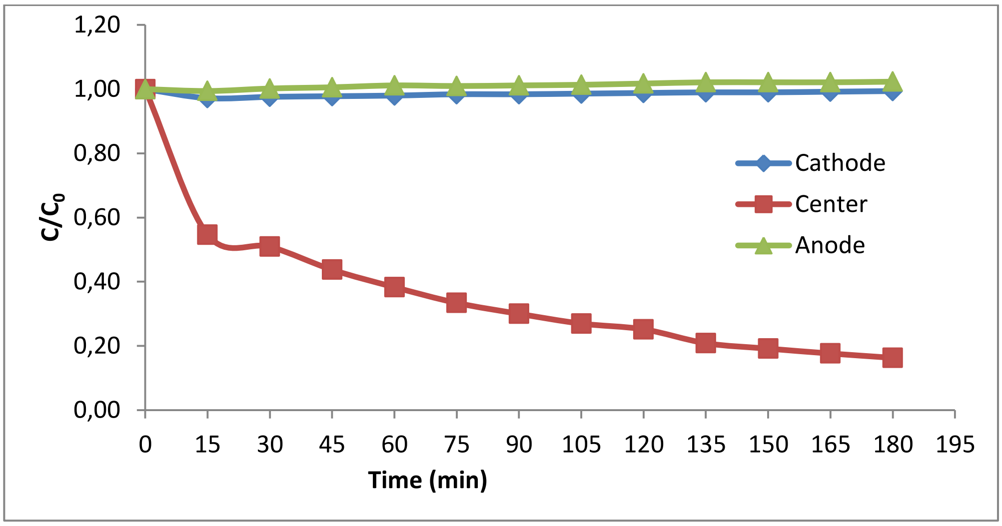

Figure 3.33 Conductivity changes cathode, center and anode compartments during EDI study with NF90 permeate

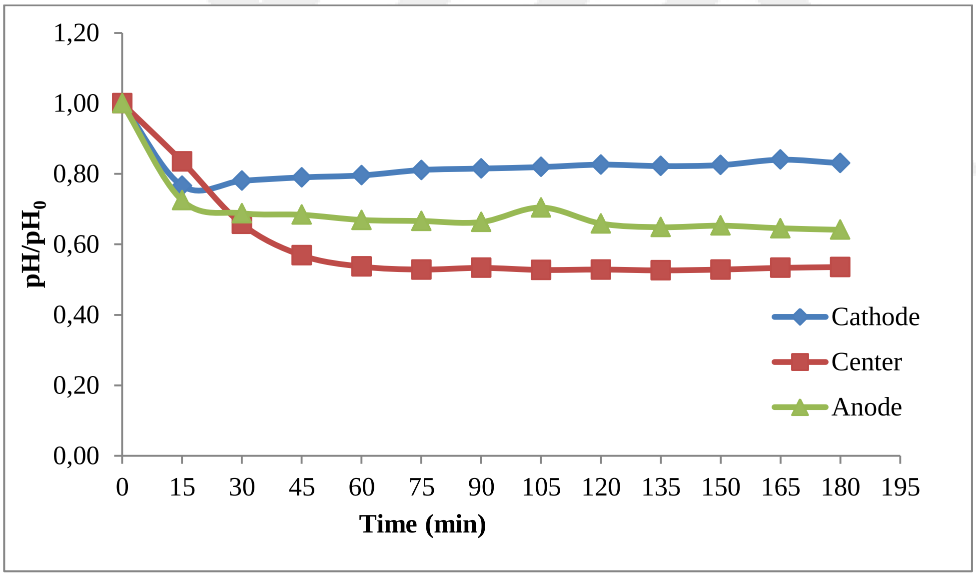

Figure 3.34 pH changes cathode, center and anode compartments during EDI study with NF90 permeate

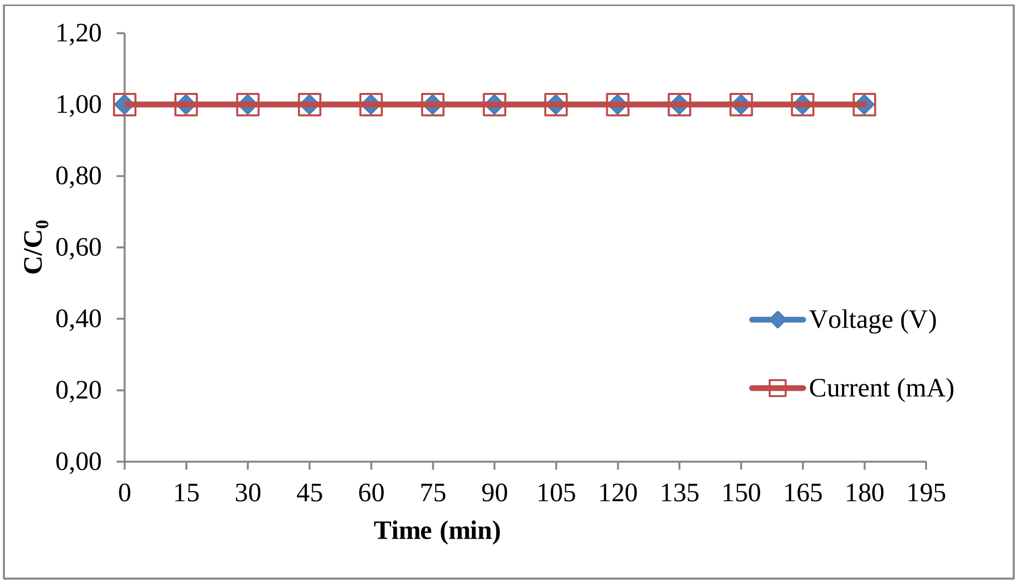

Figure 3.35 Voltage and current changes during EDI study with NF90 permeate

3.3.2.2.EDI studies with NF270 permeate

The permeate of NF270 membrane generated by Sepa CF II membrane test system under 10 bar of pressure was used as the feed solution for the EDI study. A 7 V of electrical potential was applied during the EDI study.

Removal of conductivity, conductivity change, pH change and electrical potential and current change graphs were given in Figures 3.36 3.39 respectively. Feed and product water characteristics were given in Table 3.14. Conductivity reduction reached 85% at the end of 180 minutes (Figure 3.37).

Table 3.14 Characteristics of feed and product water in EDI studies with NF270 permeate

Parameters	Unit	EDI feed	Product water
pH	-	5.89	4.72
Conductivity	μS/cm	343	51.1
TDS	mg/L	180.3	23.5
Salinity	‰	0.18	0.02

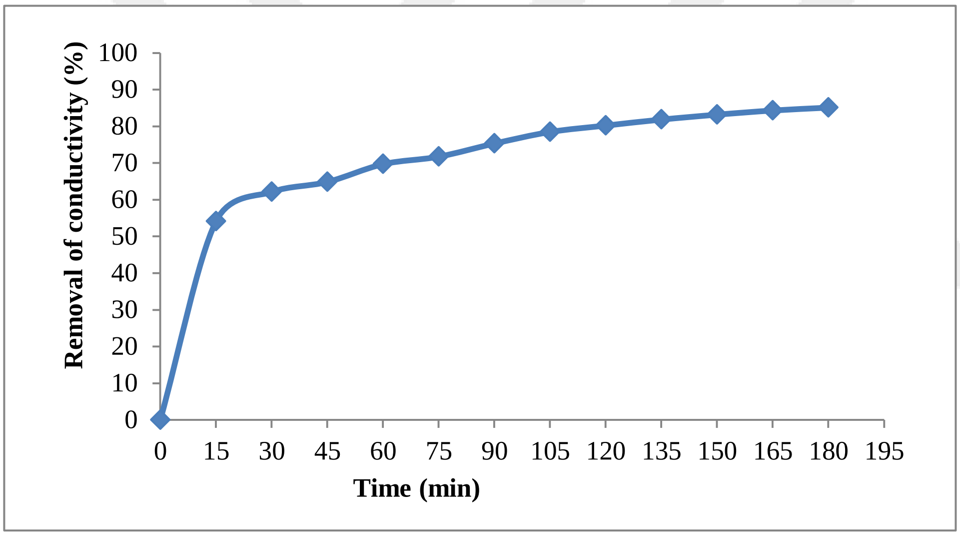

Figure 3.36 Removal of conductivity by EDI for NF270 permeate

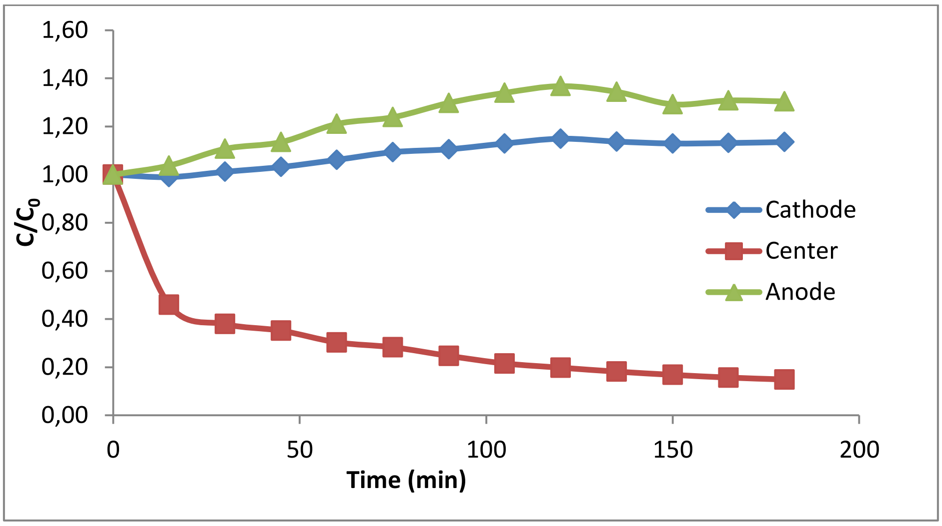

Figure 3.37 Conductivity changes in cathode, center and anode compartments during EDI by NF270 permeate

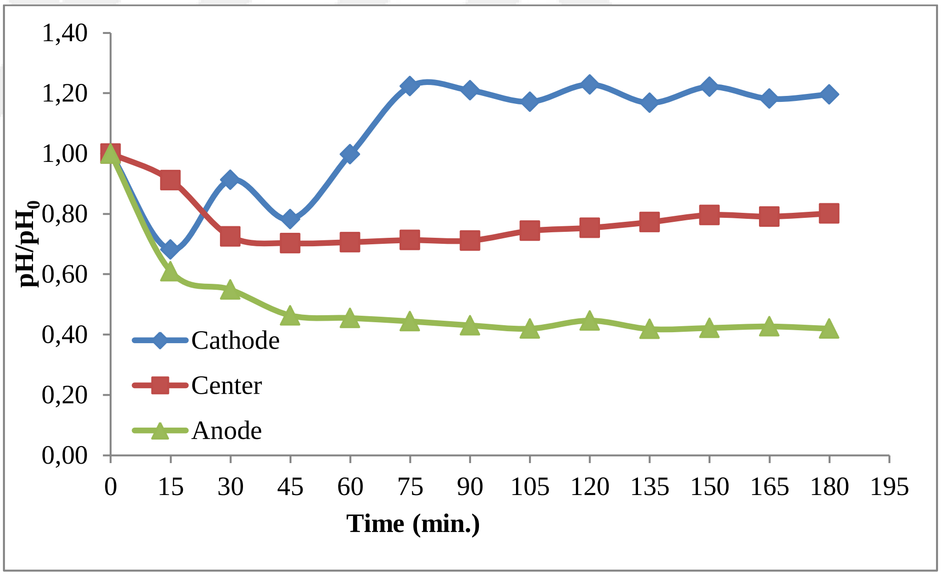

Figure 3.38 pH changes in cathode, center and anode compartments during EDI study byNF270 permeate

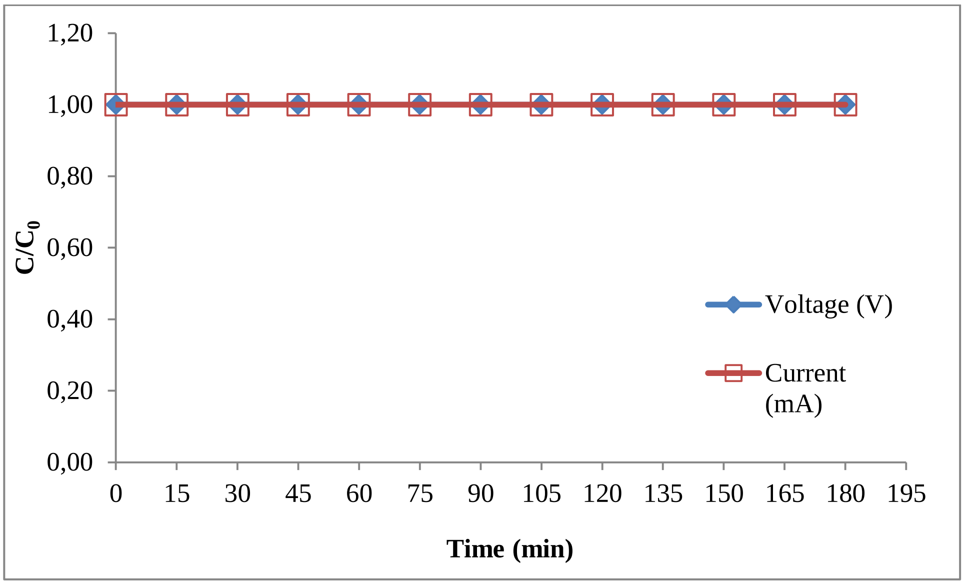

Figure 3.39 Voltage and current changes during EDI study by NF270 permeate

3.3.2.3.EDI studies with AK-BWRO permeate

The filtrate of AK-BWRO membrane produced from ground water of DİMES by using Sepa CF II membrane test system under 20 bar of pressure was used as a feed solution in the EDI study. A 7 V of electrical potential was applied during the EDI study.

Feed and product water characteristics were given in Table 3.15. Removal of conductivity, conductivity change, pH change and electrical potential and current change graphs are given in Figures 3.40 - 3.43 respectively. As shown in Figure 3.40 conductivity removal reached to 90% in 90 min.

Table 3.15 Characteristics of feed and product water in EDI studies with AK-BWRO permeate

Parameters	Unit	EDI feed	EDI product
pH	-	8.66	4.63
Conductivity	µS/cm	67.4	4.11
TDS	mg/L	31.1	1.85
Salinity	‰	0.04	0

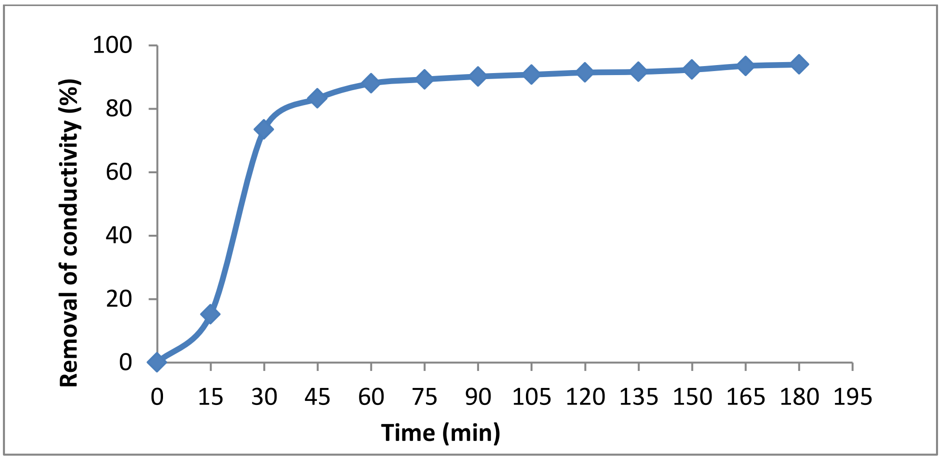

Figure 3.40 Removal of conductivity during EDI study by AK-BWRO permeate

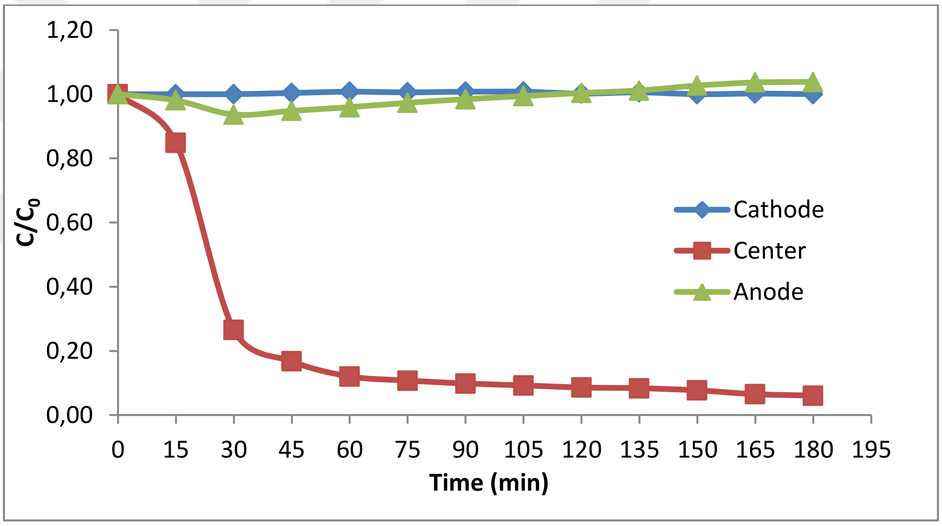

Figure 3.41 Conductivity changes for EDI study of AK-BWRO permeate

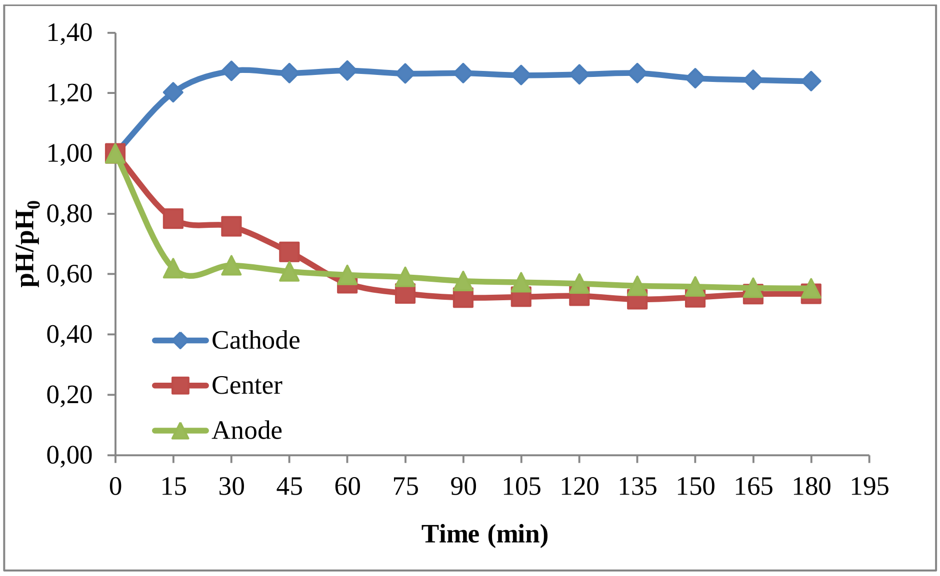

Figure 3.42 pH changes in cathode, center and anode compartments during EDI by AK-BWRO permeate

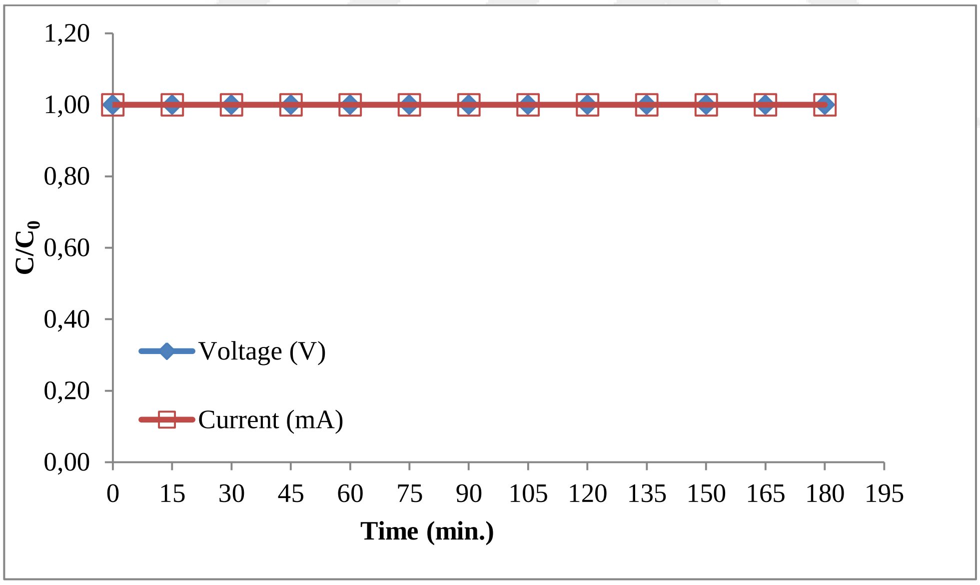

Figure 3.43 Voltage and current changes during EDI study by AK-BWRO permeate

3.3.2.4.EDI studies with BW30 RO permeate

The ground water obtained from the fruit juice factory was filtered with BW30 RO membrane through Sepa CF II membrane test system. For this filtration a pressure of 20 bar was applied and the filtrate was collected and used as feed solution in the EDI test. Electrical potential of 7 V was applied in the EDI system. Feed and product water characteristics were given in Table 3.16. Removal of conductivity change, pH change and electrical potential and current change plots were given in Figures 3.44-3.47 respectively. Conductivity reduction reached 82% at the end of 75th minute (Figure 3.44).

Table 3.16 Characteristics of feed and product water in EDI studies with BW30 permeate

Parameters	Unit	EDI feed	EDI product
pH	-	4.08	5.11
Conductivity	µS/cm	42.3	1.85
TDS	mg/L	19.46	0.84
Salinity	‰	0.02	0

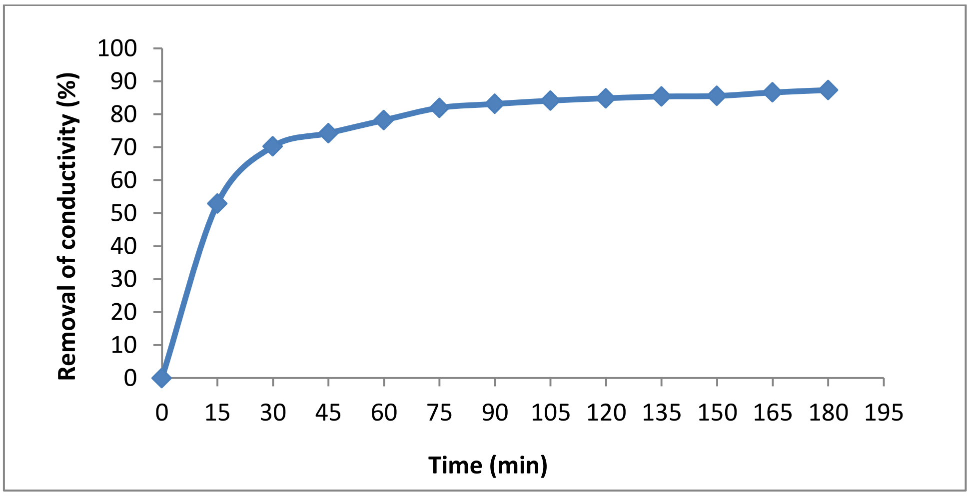

Figure 3.44 Removal of conductivity during EDI study of BW30 permeate

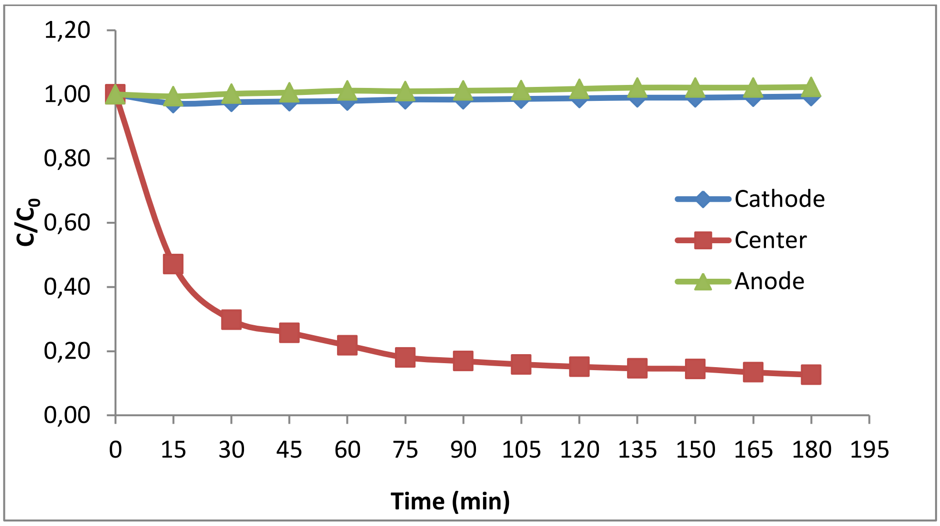

Figure 3.45 Conductivity changes in cathode, center and anode compartments during EDI by BW30 permeate

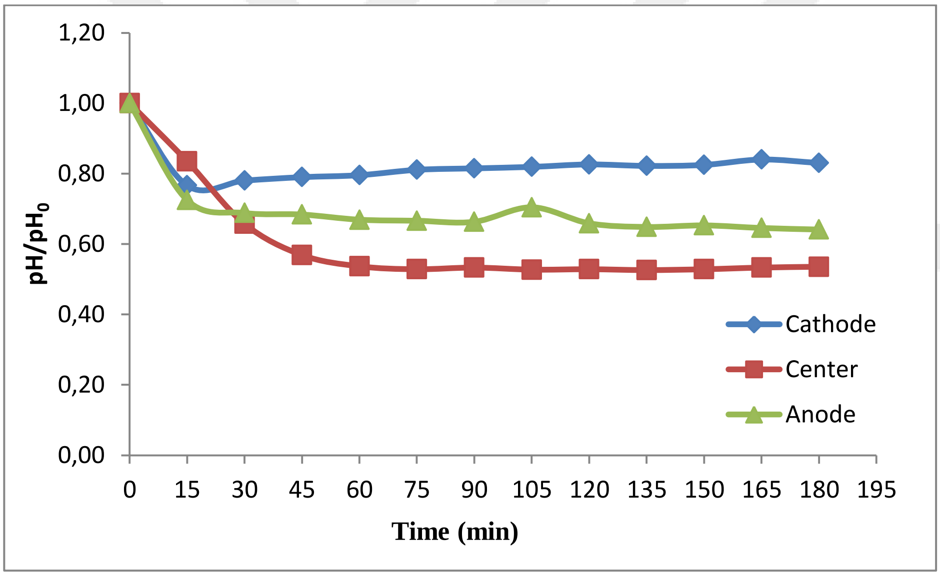

Figure 3.46 pH changes in cathode, center and anode compartments during EDI by BW30 permeate

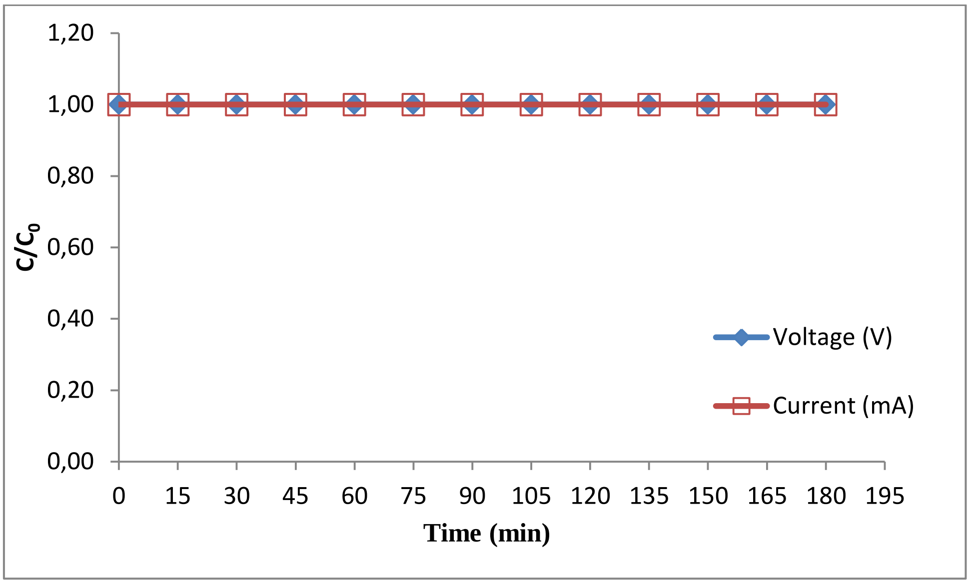

Figure 3.47 Voltage and current changes during EDI study by BW30 permeate

3.3.3. EDI studies with ED diluate of RO plant concentrate in fruit juice plant

A 10 V of electrical potential was applied to the diluate of ED obtained from the concentrate effluent of the RO treatment system of fruit juice plant for ground water purification. According to litrerature (Wood et al., 2010) the conductivity of the feed solution for the EDI system should be less than 50 µS/cm. However, the conductivity of the ED diluate was higher than 50 µS/cm due to concentration polarization perhaps. To overcome this the concentrate compartment was diluated with distilled water to a total conductivity lower than 1000 µS/cm, when the conductivity drop was stopped. So, conductivity decrease was started again and the desired 50 µS/cm in diluate was obtained.

The diluate solution was then used as feed water for EDI work. Neosepta AMX and Neosepta CMX membranes along with Purolite A500 and Purolite CT175 resins were used in these studies. The studies were carried out at 5, 7, 10 and 13 V. Feed and product water characteristics were given in Table 3.17.

Table 3.17 Characteristics of feed and product water during EDI studies

Electrical potential		pH	Conductivity (µS/cm)	TDS (mg/L)	Salinity (‰)
5 V	EDI feed	4.3	51.3	24.11	0.02
	EDI product	4.23	10.48	4.93	0
7 V	EDI feed	4.2	52	23.9	0.02
	EDI product	4.72	3.42	1.57	0
10 V	EDI feed	7.55	50.2	23.59	0.02
	EDI product	4.41	7.42	3.49	0
13 V	EDI feed	5.77	58.3	26.79	0.02
	EDI product	3.9	25.9	11.9	0.01

3.3.3.1.EDI tests at 5 V

The ED diluate of RO concentrate obtained from DİMES RO plant was further treated with EDI at 5V.

Feed and product water characteristics were given in Table 3.18. The plots of conductivity removal, conductivity change, pH change, electrical potential and current change plots were given in Figures 3.48- 3.51 respectively. Conductivity removal reached 80% at the end of 180 min (Figure 3.48).

Table 3.18 Characteristics of feed and product water during EDI studies at 5 V for ED diluate of RO concentrate from DİMES plant

Parameters	Unit	EDI feed	EDI product
pH		4.30	4.23
Conductivity	µS/cm	51.30	10.48
TDS	mg/L	24.11	4.93
Salinity	‰	0.02	0

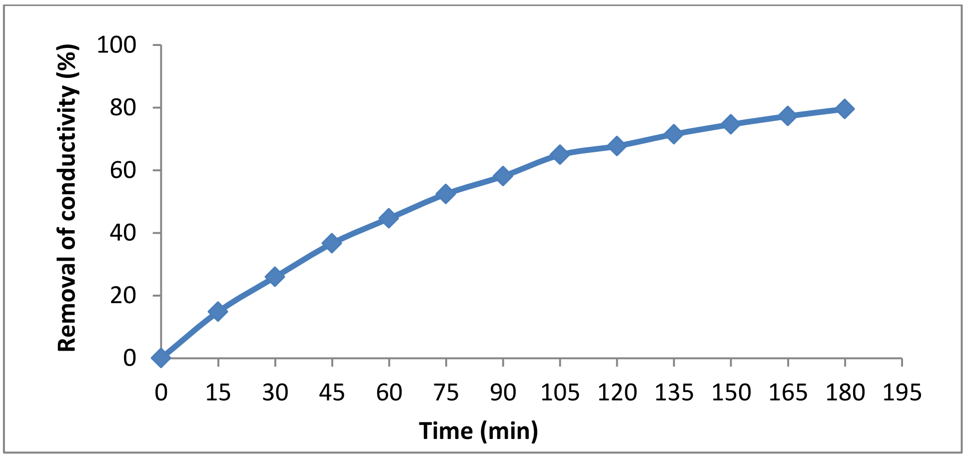

Figure 3.48 Removal of conductivity from ED diluate of RO brine of Dimes RO plant by EDI at 5 V

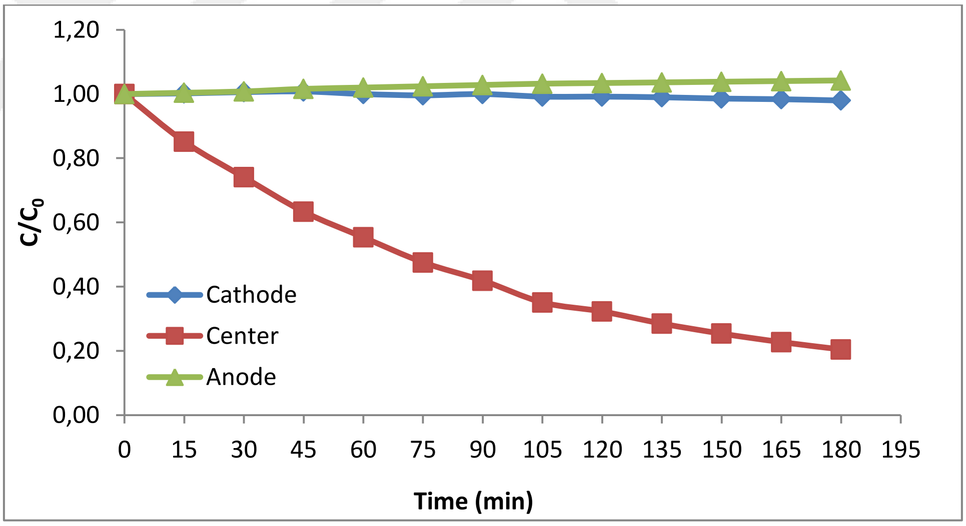

Figure 3.49 Conductivity changes during EDI study at 5 V for ED diluate of RO concentrate from Dimes RO plant

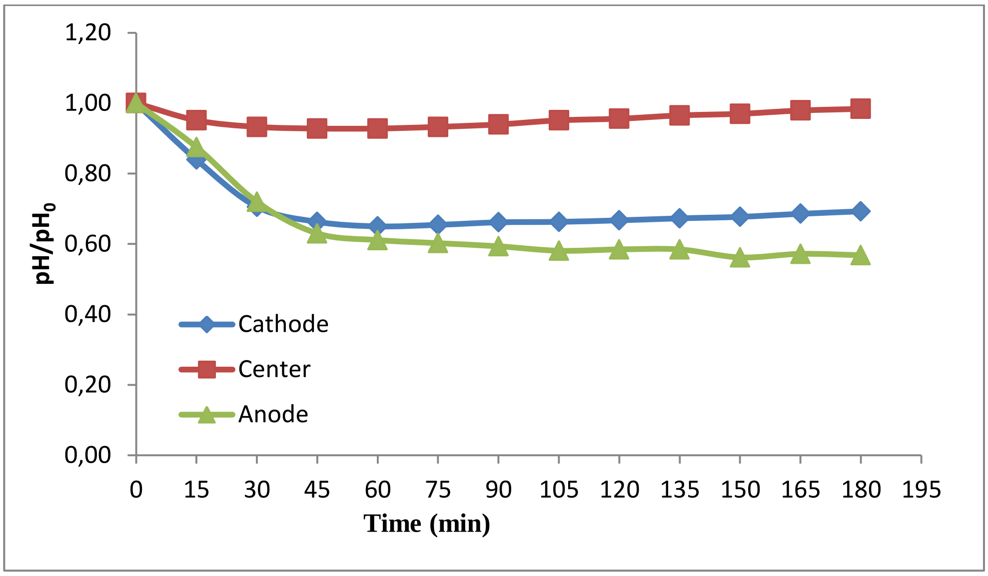

Figure 3.50 pH changes in cathode, center and anode compartments during EDI study at 5 V for ED diluate of RO concentrate from Dimes RO plant

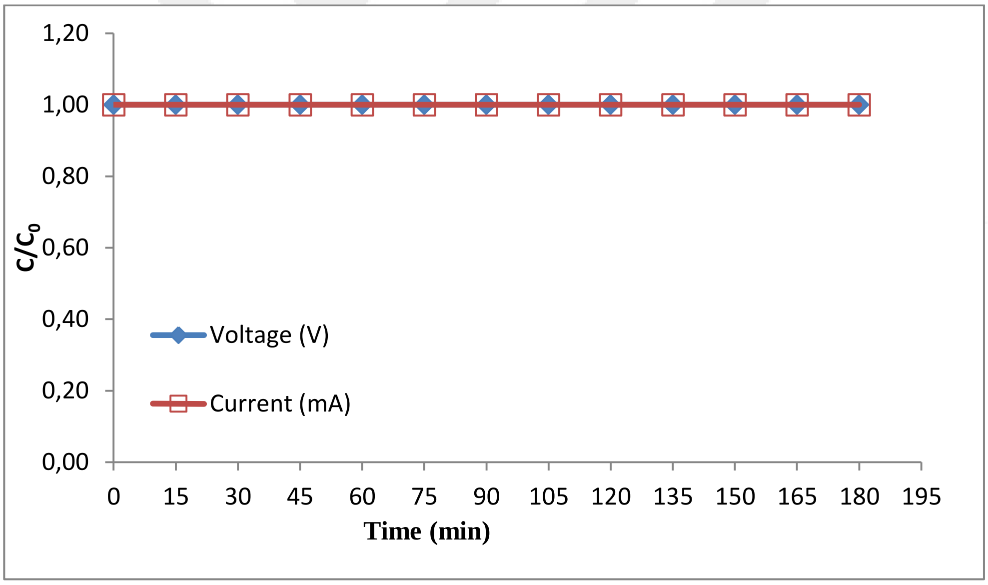

Figure 3.51 Voltage and current changes during EDI study at 5 V for ED diluate of RO concentrate from Dimes RO plant

3.3.3.2.EDI tests at 7 V

The ED diluate of RO concentrate obtained from DİMES RO plant was further treated with EDI at 7V.

Feed and product water characteristics were given in Table 3.20. The plots of conductivity removal, conductivity change, pH change, electrical potential and current change plots were given in Figures 3.56- 3.59 respectively. Conductivity removal was 98% at the end of 180 min (Figure 3.56).

Table 3.19 Characteristics of feed and product water during EDI studies at 7 V for ED diluate of RO concentrate from DİMES RO plant

Parameters	**Unit**	**EDI feed**	**EDI product**
pH		4.2	4.72
Conductivity	µS/cm	52	3.42
TDS	mg/L	23.90	1.57
Salinity	‰	0.02	0

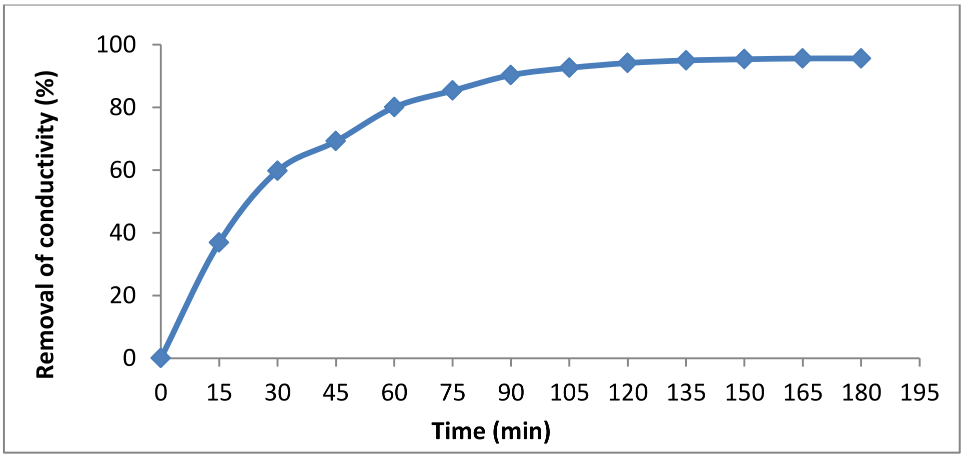

Figure 3.52 Removal of conductivity during EDI studies at 7 V for ED diluate of RO concentrate from DİMES RO plant

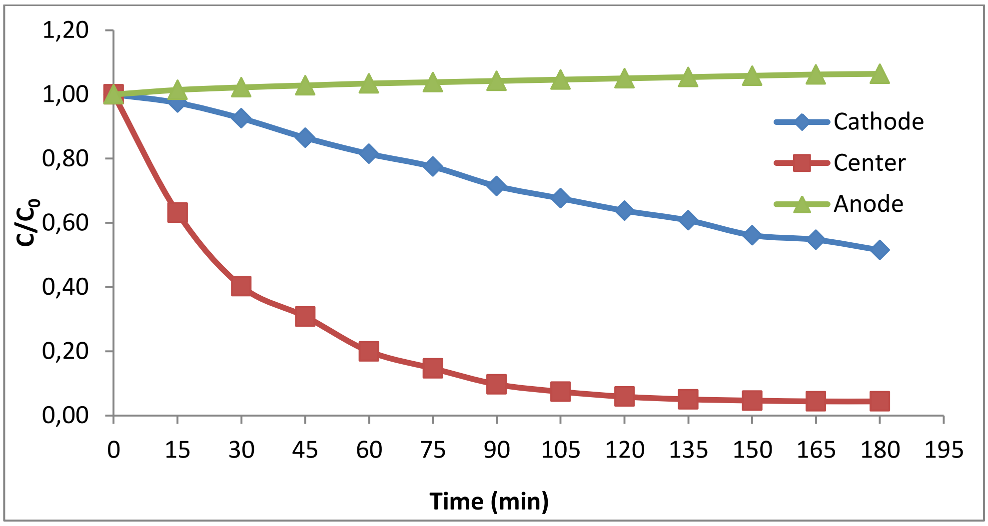

Figure 3.53 Conductivity changes during EDI studies at 7 V for ED diluate of RO concentrate from DİMES RO plant

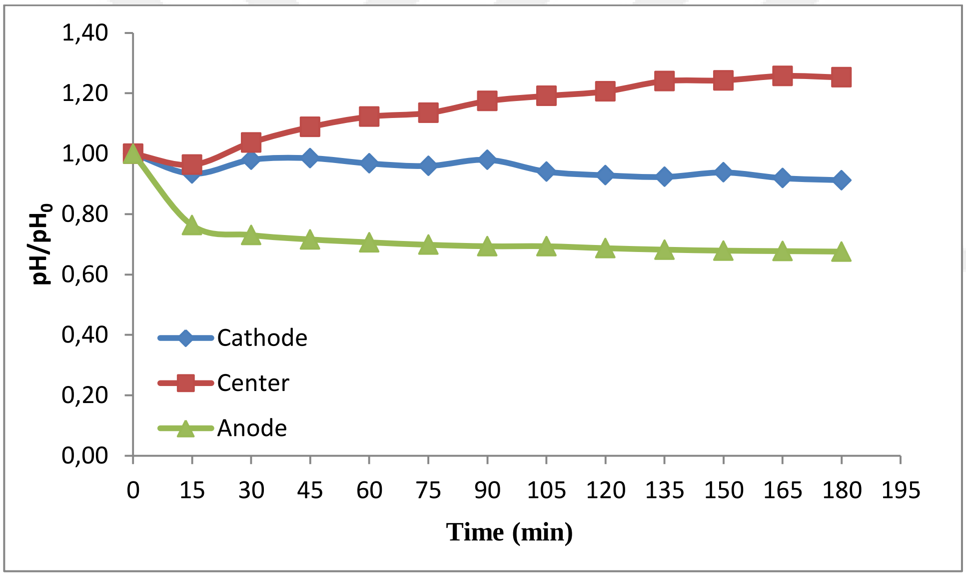

Figure 3.54 pH changes during EDI studies at 7 V for ED diluate of RO concentrate from DİMES RO plant

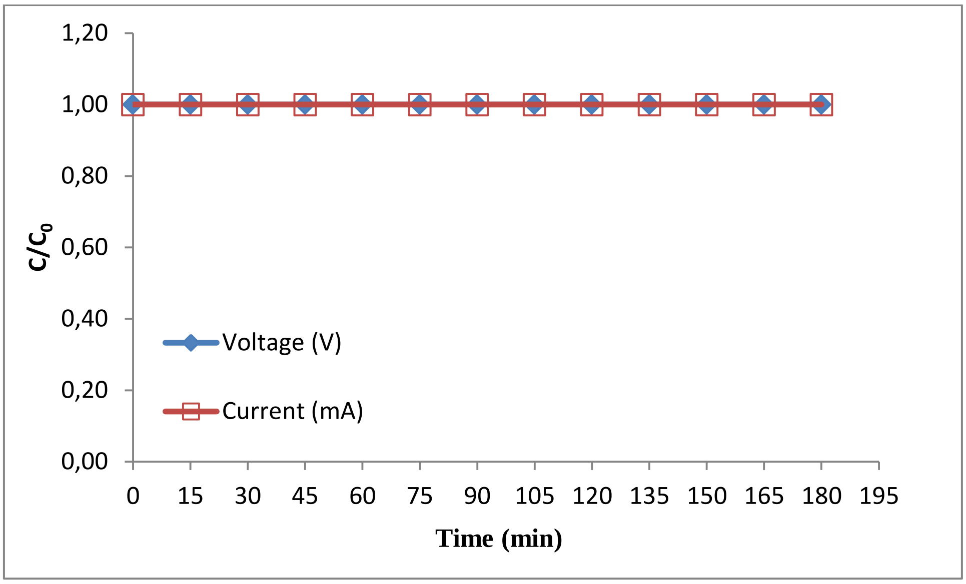

Figure 3.55 Voltage and current changes during EDI studies at 7 V for ED diluate of RO concentrate from DİMES RO plant

3.3.3.3.EDI tests at 10 V

The ED diluate of RO concentrate obtained from DİMES RO plant was further treated with EDI at 10 V.

Feed and product water characteristics were given in Table 3.20. The plots of conductivity removal, conductivity change, pH change, electrical potential and current change plots were given in Figures 3.56- 3.59 respectively. Conductivity removal was 85% at the end of 180 min (Figure 3.56).

Table 3.20 Characteristics of feed and product water during EDI studies at 10V for ED diluate of RO concentrate from DİMES RO plant

Parameters	Unit	EDI feed	EDI product
pH		7.55	4.29
Conductivity	µS/cm	50.2	7.42
TDS	mg/L	23.59	3.49
Salinity	‰	0.02	0

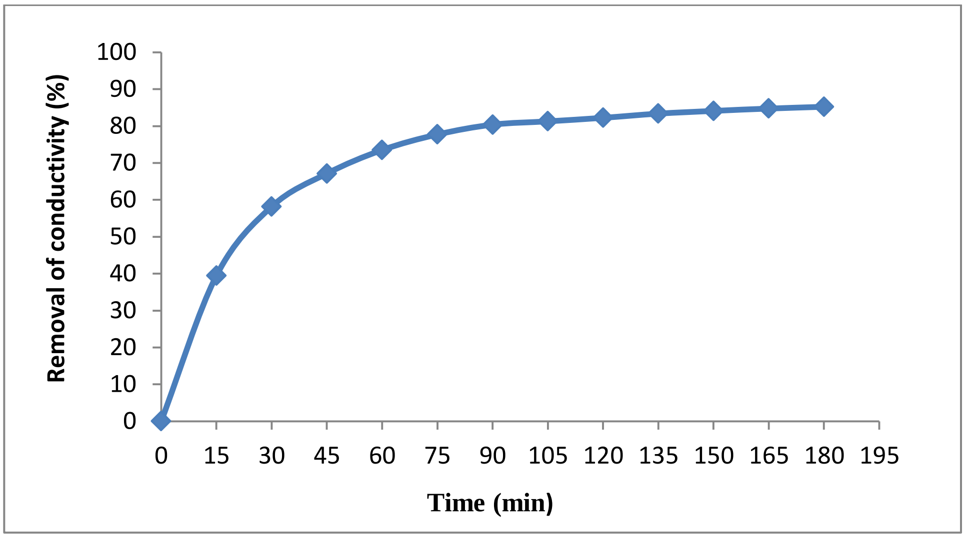

Figure 3.56 Removal of conductivity from ED diluate of RO concentrate of Dimes RO plant by EDI at 10 V

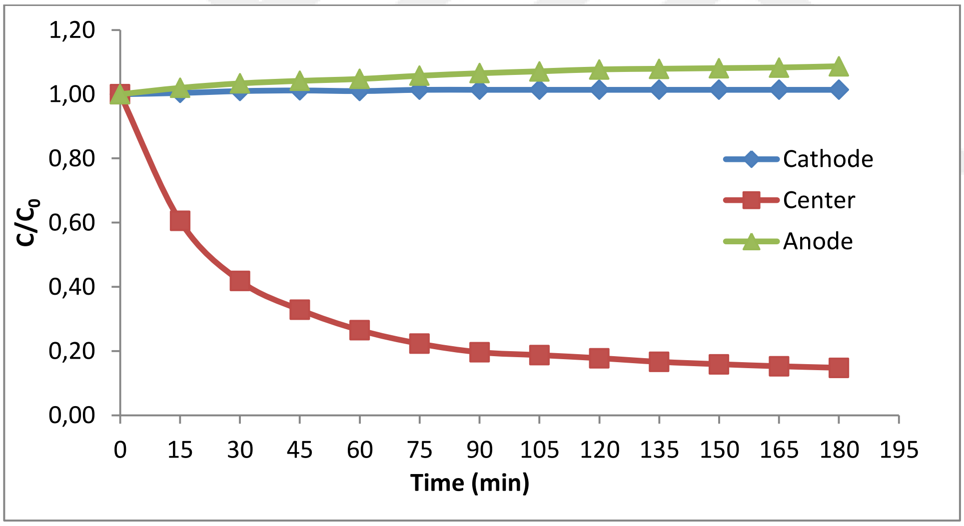

Figure 3.57 Conductivity changes in cathode, center and anode compartments during EDI study at 10 V for ED diluate of RO concentrate from DİMES RO plant

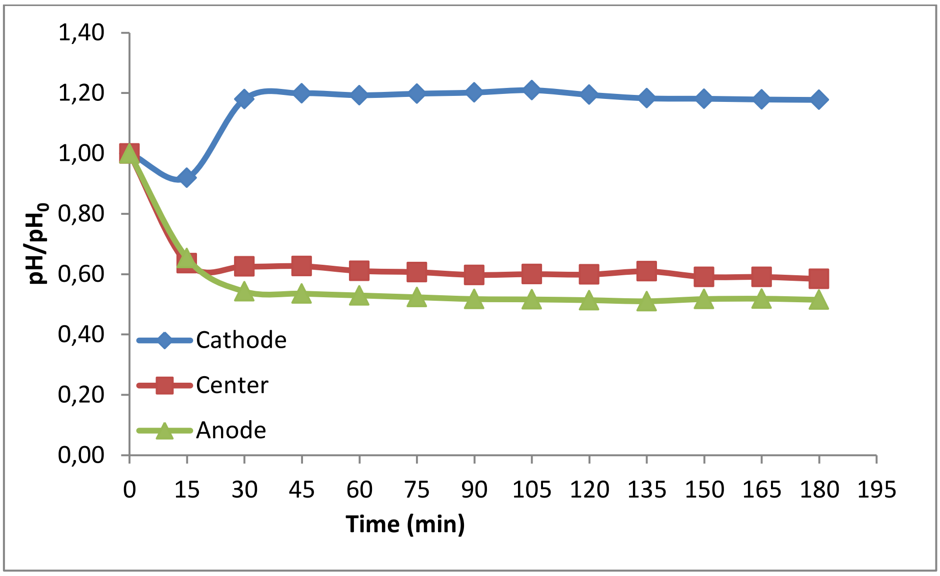

Figure 3.58 pH changes in cathode, center and anode compartments during EDI study at 10 V for ED diluate of RO concentrate from DİMES RO plant

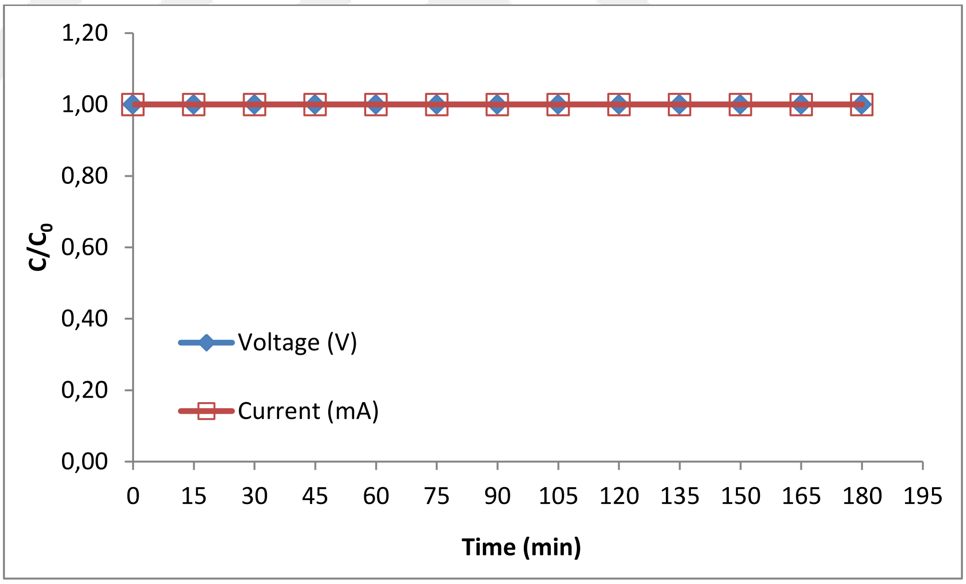

Figure 3.59 Voltage and current changes during EDI study at 10 V for ED diluate of RO concentrate from DİMES RO plant

3.3.3.4.EDI tests at 13 V

The ED diluate of RO concentrate obtained from DİMES RO plant was further treated with EDI at 13V.

Feed and product water characteristics were given in Table 3.21. The plots of conductivity removal, conductivity change, pH change, electrical potential and current change plots were given in Figures 3.60- 3.63 respectively. Conductivity removal was 55% at the end of 180 min (Figure 3.60).

Table 3.21 Characteristics of feed and product water during EDI studies at 13 V for ED diluate of RO concentrate from DİMES RO plant

Parameters	**Unit**	**EDI feed**	**EDI product**
pH	-	5.77	3.90
Conductivity	µS/cm	58.3	25.9
TDS	mg/L	26.79	11.90
Salinity	‰	0.02	0.01

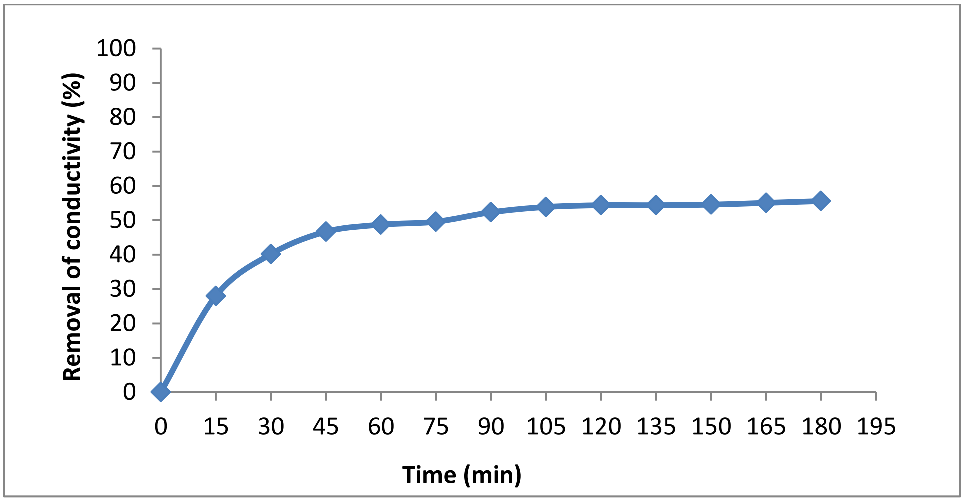

Figure 3.60 Removal of conductivity from ED diluate of RO concentrate of Dimes RO plant by EDI at 13 V

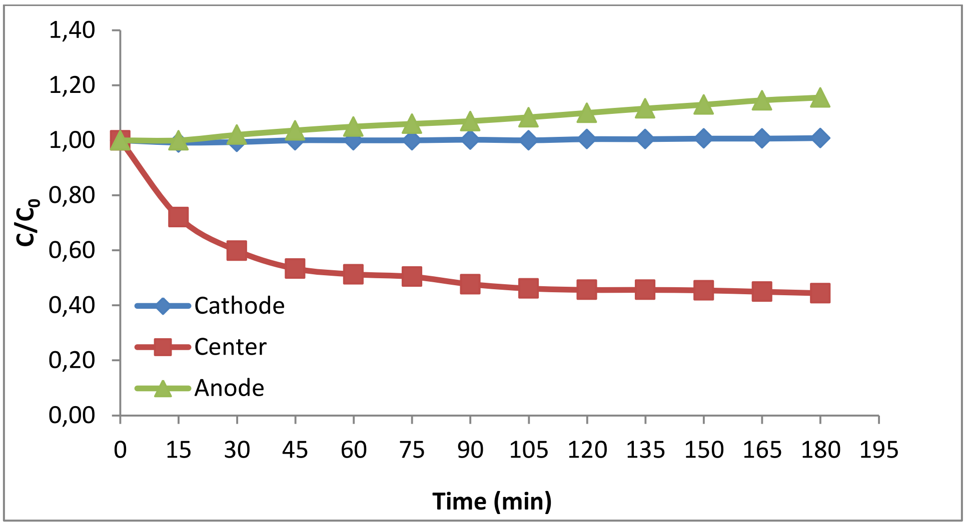

Figure 3.61 Conductivity changes in cathode, center and anode compartments during EDI study at 13 V

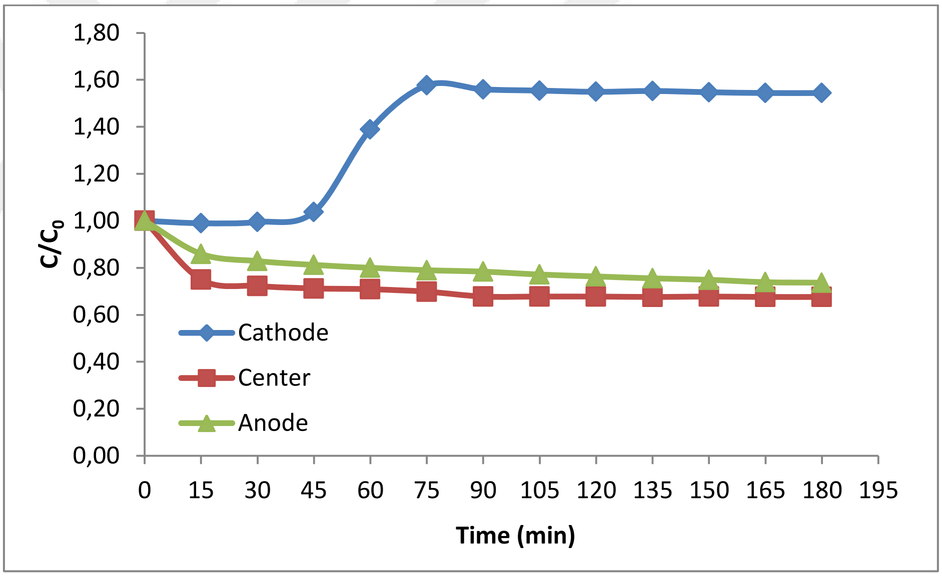

Figure 3.62 pH changes in cathode, center and anode compartments during EDI study at 13V

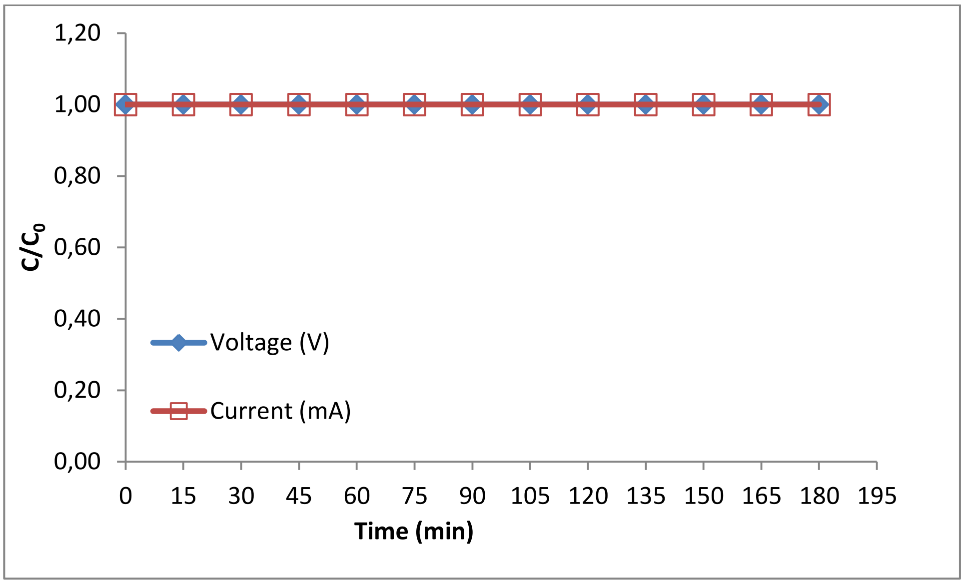

Figure 3.63 Voltage and current changes during EDI study at 13 V

4. CONCLUSIONS

Electrodialysis (ED) and electrodeionization (EDI) methods in the recovery of brine of reverse osmosis treatment of raw well water in a fruit juice company has been investigated. During the ED and EDI parametric study, the influence of applied electrical potential and initial sample solution concentrations was monitored. The samples solution with different quality for both ED and EDI processes were produced by using lab scale filtration system and were directly obtained from the RO treatment system of fruit juices company. The following conclusions were obtained from the analysis of results:

- An increase of applied potential influenced ED and EDI performances in terms of conductivity removals. An increase of applied potential up to the optimum value resulted in an increase of ED and EDI performances. Applying an electrical potential over the optimum one reduced the ED and EDI performances in terms of conductivity removals.
- With higher applied electrical potential, the maximum ED and EDI performances were reached in shorter time than at lower electrical potential.
- Feed water solutions with higher conductivity resulted in longer time of ED and EDI process.
- ED and RO product water was suitable for EDI process.
- NF product quality with higher conductivity resulted in lower EDI performances.
- The resins placed in EDI cell became exhausted in short time and the process continued as ED not as EDI.
- NF product water quality needs an addition post-treatment to meet EDI feed requirements.
- NF/RO brines from ground water were successfully recovered by integrating ED and EDI methods.

A combination of membrane filtration (NF and RO) and electro-membrane process (ED and EDI) is a good strategy to increase water recovery by post treatment of NF/RO concentrates with integrated ED and EDI methods. A closed loop of NF/RO, ED and EDI systems should be optimized to increase water recovery with higher quality from NF/RO brines and to reduce the negative effects of NF/RO brines on the environment.

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TÜBİTAK-NASU 114M551 Gıda Endüstrisinde Kullanılan Suyun Geri Kazanılmasında ve Peynir Altı Suyunun Tuzsuzlaştırılmasında Membran Teknolojilerin Uygulanması

TÜBİTAK-JSPS 214M360 Adsorpsiyon-Elektrodiyaliz Hibrit Prosesiyle Jeotermal Sulardan Bor ve Lityum Kazanılması.

ULUSAL VE ULUSLARARASI KONGRELERDE SUNUMLAR

Poster

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