İSTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL OF SCIENCE ENGINEERING AND TECHNOLOGY

DEMINERALIZATION OF WHEY BY ELECTRODIALYSIS

M.Sc. THESIS

Cansu GÜLSEVEN

Department of Food Engineering

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İSTANBUL TEKNİK ÜNİVERSİTESİ ★ FEN BİLİMLERİ ENSTİTÜSÜ

ELEKTRODİYALİZ İLE PEYNİR ALTI SUYUNUN DEMİNERALİZASYONU

YÜKSEK LİSANS TEZİ

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Üzerimde sonsuz emeği olan canım aileme,

FOREWORD

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September 2014

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ABBREVIATIONS

BPM : Bipolar Membranes
COD : Chemical Oxygen Demand
DC : Direct Current
DF : Diafiltration
DM : Dry Matter
ED : Electrodialysis
IEM : Ion Exchange Membranes
ISO : International Organization for Standardization
MC : Membrane Contactor
MF : Microfiltration
NF : Nanofiltration
PV : Pervaporation
RO : Reverse Osmosis
TCA : Trichloroacetic Acid
UF : Ultrafiltration

SYMBOLS

α	: Transport of water
Δm_{Ash}	: Ash content change in diluate
Ash_D	: Ash content of diluate
Ash_F	: Ash content of feed
b	: Volume of the hydrochloric acid solution used in the blank test
c	: Concentration of standard solution of hydrochloric acid
C_{D_DM}	: Capacity of electrodialysis according to produced dry matter
C_F	: Capacity of electrodialysis according to processed feed
$c\_s,_{FD},_0$	: Concentration of salt into the diluate at the beginning of experiment
$c\_s,_{FD},_{final}$	: Concentration of salt into the diluate at the end of the experiment
DM_D	: Dry matter of diluate
DM_F	: Dry matter of feed
i	: Volume
I_{ef}	: Electrodialysis current
J	: Flux of salt
k	: Electrical charge
L	: Phenomenological coefficient relating the fluxes to the driving forces
m	: Mass of the test portion
m_0	: Mass of the dish, lid and sea sand
m_1	: Mass of the dish, lid and sample portion
m_2	: Mass of the dish, lid and dried sample portion
$m_{D,0}$	: Mass of diluate before electrodialysis
$m_{D,final}$	: Mass of diluate after electrodialysis
M_{FD}	: Mass of feed
$m\_{FD},_0$	: Weight of salt into the diluate at the beginning of experiment
$m\_{FD},_{final}$	: Weight of salt into the diluate at the end of the experiment
M_{fp}	: Weight of filter paper
mk	: Weight of crucible
mp	: Weight of sample after ashing
Mr	: Exact molarity of hydrochloric acid standard volumetric solution
mv	: Weight of sample before ashing
N	: Number of membranes in electrodialysis
Nfp	: Average nitrogen content in filter paper
t_{final}	: Time of electrodialysis in total
Vb	: Volume of the hydrochloric acid standard volumetric solution used in the blank test
Vs	: Volume of the hydrochloric acid standard volumetric solution used in the determination
W	: Power of electrodialysis, which represents how many volts are used to processed for 1 kg of feed
W_{ef}	: Electrodialysis power

$\mathbf{W_N}$	**:** Nitrogen content of sample
Wp	**:** Ash content of sample
X	**:** Generalized driving force

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DEMINERALIZATION OF WHEY BY ELECTRODIALYSIS

SUMMARY

Whey is a green-yellow colored liquid obtained from cheese production. Whey can separated into sweet and acid whey, depending on the processing technique consequential in casein subtraction from fluid milk.

Sweet (rennet) whey is obtained after chymosin treatment from the casein fraction of milk, which is the major fraction of milk proteins. After chymosin enzymatic reaction, colloidal solubility is lost. This leads to separation of coagulum from casein into the cheese curd and whey. Whey is made up of 93-94% of water and milk serum as lactose, soluble proteins, minerals, lactic acid and fats. In addition, whey can contains B group vitamins, citric acid and some non-protein nitrogen compounds (urea and uric acid), β-lactoglobulin, α-lactoglobulin, serum albumin, immunoglobulins and lactoferrin. The second type is acid whey, obtained from fermentation processes. For both of the sweet and acid wheys, lactose is the leading part, whey proteins and minerals follow subsequently. Acidity, whey protein composition and mineral content are the major differences between the two whey types. The chief difference between them is lactic acid content, acid whey can have more lactic acid than sweet whey, which can affect the processing of whey along with the nutritional properties.

About whey production and processing techniques, there has been a significant development over the past three decays. Particularly separation and fractioning of whey constituents, has an increased commercial interest. Whey proteins have a high nutritional value and great functional properties. As a result of these properties, they are extensively used both in animal and human nutrition. Whey proteins have a great functionality and they can be used as emulsifiers, stabilizers, foaming and texturizing agents and also they can be part in infant formula, dairy and bakery products, meats and beverages as food additive. If whey proteins are purified and isolated from other constituents in whey, their functionality and nutritional value are increased.

On the other hand, because of high salt content, application of whey constituents and whey are restricted. It is directly affect the functional and nutritional properties of whey, as well as its flavor and quality. Therefore, desalination process is precursor technique and should be applied effectively. For demineralization process; electrodialysis, ion exchange, nanofiltration, reverse osmosis and microfiltration are the main membrane techniques to be encounter, which differ, in their driving force and operation princibles.

Electrodialysis with ion-exchange membranes is one of the most significant membrane separation processes where it separates ions from an aqueous solution with the help of electrically charged membranes influence of an electrical potential difference as a driving force. It can be used for many water treatments additionally; it is used in wine industry as stabilizer and in sugar industry for demineralization.

There are ion exchange membranes in electrodialysis stack, which have two electrodes name as anode and cathode and consist of a chain of anion and cation exchange membranes settled in an alternating array. Under the influence of a direct current, an ionic solution is pumped through these membranes. Due to this driven force, positively charged cations pass easily through the negatively charged cation exchange membrane and move toward cathode and they retained by the anion exchange membrane. For anions situation take place contrarily. As a result of this process, one solution is concentrated conferring to ion content, named as brine or concentrate, whereas other solution is become depleted, name as diluate.

For this purpose electrodialysis with anion-cation membranes is applied to rennet whey powder from Moravia Lacto a.s. company (Jihlava, Czech Republic) as four different percentage as 3.5, 7, 10 and 14 at negative and positive polarity. Before each experiment, electrodialysis stack was washed with distilled water. For the diluate part, whey solution was used with different concentration, for the concentrate part HNO_3 solution was used with a pH 2, and the electrical solution was used a solution of $NaNO_3$ at a concentration of 10 $g.l^{-1}$. Before and after electrodialysis, in order to check the membranes working properly or not, a saline test for a model solution Na_2SO_4 was prepared and conducted. At the end of the test, Javg values are calculated by using macro excel document. During desalination process, changes in conductivity, pH, and temperature were recorded 5 minutes intervals. The experiment was conducted at a constant voltage of 16 V and terminated after reaching 95% desalination of the diluate. At the end of the each experiment 45 ml of diluate was collected in order to determine ash content and dry matter.

In addition to electrodialysis, ultrafiltration is applied to 7% of whey solution unit from ARNO 700 with tubular ceramic TAMI-Industries membranes type Clover. Ultrafiltration was conducted at a constant pressure of 2 Bar with a 50 kDa membrane and it was terminated after obtaining 300 grams permeate. Concentration factor during experiment was 2, because of obtaining 3 liters of permeate and 3 liters of retentate from the 6 liters of feed.At the end of the each experiment 45 ml of feed, permeate and retentate were collected in order to determine ash content, dry matter and protein analysis. Correspondingly permeate and retentate samples collected ± 1000 ml for ED experiment.

At the end of each experiment, datas are settled in to a macro excel document in order to assess the factors as J_{avg}, C_F, C_{D_DM}, Δm_{ASH}, α and W, which can influence efficiency and process of electrodialysis of whey. These factors are a novel parameters of electrodialysis process and can be used as a good source to optimize the desalination process especially for whey and whey products. It can assist to optimization of energy, quality, functional and nutritional properties of whey.

It can be concluded, sweet rennet whey is treated by electrodialysis with ion exchange membranes and factors which influence efficiency and process of electrodialysis of whey are determined and calculated with a help of a macro excel document especial to this project. In addition, electrodialysis process of retentate and permeate solutions, which are obtained after ultrafiltration process are calculated. It is shown that retentate and permeate solution, successfully applied in electrodialysis. Without meeting any problem, retentate and permeate solutions can be desalinated. According to their W values, it can be said that ultrafiltration before electrodialysis can be effectively adapted to food industry and they are open to new research and development.

Whey desalination is an essential and crucial process in food technology. On the other hand, it has been examined only for last few decays. Consequently, further research and examination is needed to apply. There are many features that have not been yet completely determined and some problems needed to be solved. Especially in dairy industry electrodialysis has an excessive potential because it is a safe, economic, fast and very effective method, that can be applied to remove salts even from highly salted wheys.

ELEKTRODİYALİZ YÖNTEMİYLE PEYNİR ALTI SUYUNUN DEMİNERALİZASYONU

ÖZET

Peynir altı suyu, peynir üretiminden elde edilen yeşil-sarı renkli bir sıvıdır. Peynir altı suyu, sütün kazein enzimi ile işlenmesine bağlı olarak tatlı ve asidik peynir altı suyu olarak sınıflandırılır. Tatlı (rennet) peynir altı suyu, kimozin enziminin süt proteinlerinin önemli bir kısmını oluşturan sütün kazein fraksiyonu ile tepkimesi sonrası elde edilir. Kimozin enzimatik reaksiyonu sonrasında, sütün kolloidal çözünürlüğü kaybolur. Kolloidal çözünürliğün kaybolması sonucunda kazeinden gelen bir pıhtılaşma görülür ve oluşan bu yapı peynir altı suyu ve lor olarak ayrışır.

Peynir altı suyu, %93-94 oranında su ve laktoz, çözülebilir proteinler, mineraller, laktik asit ve yağ gibi süt serumundan oluşmaktadır. Buna ek olarak, peynir altı suyu B grubu vitaminleri, sitrik asit ve bazı protein olmayan azot bileşikleri ile (üre ve ürik asit), β-laktoglobülin, α-laktoglobülin, serum albümin, immünoglobulin ve laktoferrin içermektedir.

İkinci tip peynir altı suyu ise fermentasyon işlemi ile elde edilen, asidik peynir altı suyudur. Hem tatlı ve hem asidik peynir altı suyu içinde laktoz en fazla orana sahip bileşendir, bunu peynir altı suyu proteinleri ve mineraller izler. Asidite, peynir altı suyu proteini bileşimi ve mineral içeriği iki peynir altı suyu türü arasındaki en önemli farkları oluşturmaktadır. Aralarındaki en belirgin fark ise, laktik asit içeriğinde görülmektedir. Asidik peynir altı suyu tatlı peynir altı suyuna göre daha fazla laktik asit içermekle beraber, laktik asit oranı peynir altı suyunun besin ve işleme ait değerlerini etkiler.

Peynir altı suyu üretimi ve işleme teknikleri hakkında, özellikle son otuz yılda önemli gelişmeler kaydedilmiştir. Özellikle peynir altı suyu bileşenlerinin ayrılması ve sınıflandırılması üzerine artan bir ticari ilgi vardır. Peynir altı suyu proteinleri, yüksek besin değeri ve fonksiyonel özelliklere sahiptir. Bu özelliklerin bir sonucu olarak, büyük ölçüde hem hayvan hem de insan beslenmesinde kullanılmaktadır. Peynir altı suyu proteinleri sahip oldukları fonksiyonel özellikler nedeniyle emülsiyon yapıcı, stabilize edici, köpük ve yapı geliştirici maddeler olarak gıda sanayisinde kullanılabilirler. Aynı zamanda bebek maması, süt ve fırıncılık ürünlerinde, et ve içecek sanayilerinde katkı maddesi olarak da kullanılmaktadırlar.

Süt proteinlerinin fonksiyonel özellikleri incelendiğinde, sahip oldukları yüksek besin değeri dışında yapısal, görünüm, viskozite ve tekstür gibi peynir altı suyuna özel özelliklerle de tanımlanır. Ancak proteinlerin fonksiyonel özelliklerini etkin kılan parametreler, taşıdıkları bazı fiziksel ve kimyasal özelliklerinden kaynaklanmaktadır. Bu proteinlerin fonksiyonel, fiziksel ve kimyasal özelliklerinin gıda işleme sırasında uygulanan farklı tekniklerle değiştirebilineceği tespit edilmiştir. Buna en belirgin örnek olarak denatürasyon ile, proteinlerin yapısının ve hidrofobik etkileşimlerinin artması verilebilinir. Bu nedenle gıda sanayinde önemli bir yere

sahip olan süt proteinlerinin fonksiyonel özellikleri bozulmadan işlenmesi büyük önem arz etmektedir.

Sahip oldukları bu özellikler, peynir altı suyu proteinleri saflaştırılarak ve diğer bileşenlerden izole edilerek işlevsellik ve besin değeri bakımından arttırılmaktadır. Öte yandan, sahip oldukları yüksek tuz içeriği, peynir altı suyu bileşenlerinin ve peynir altı suyunun uygulama alanını sınırlandırır. Tuz içeriği doğrudan sahip oldukları fonksiyonel özellikleri ve besin değerini etkilemenin yanı sıra, aroma ve kaliteyi de etkiler. Bu nedenle demineralizasyon tekniği diğer teknikler öncesinde uygulanmalıdır.

Demineralizasyon işlemi için; elektrodiyaliz, iyon değişimi, nanofiltrasyon, ters ozmoz ve mikrofiltrasyon temel membrane teknikleridir. Bu uygulamalar sahip oldukları itici güç ve operasyon çalışma prensipleri ile farklılaşmaktadır.

İyon değiştirme membranları ile elektrodiyaliz, itici güç olarak elektriksel potansiyel farkın kullanıldığı, elektriksel olarak yüklü membranların etkisiyle sulu çözeltiden iyonları ayıran en önemli membran ayırma işlemlerinden biridir. Elektrodiyaliz birçok su işleme teknikleri için kullanılmakla beraber, şeker endüstrisinde demineralizasyon için ve şarap endüstrisinde stabilazatör olarak kullanılmaktadır.

Elektrodiyaliz düzeneği içerisinde bulunan iyon değişim membranları, anot ve katot olarak iki elektrottan oluşurlar. Ayrıca elektrodiyaliz düzeneğinde alternatif dizide yerleşmiş anyon ve katyon değişim membranları bulunmaktadır. Doğru akım etkisi ile, bir iyonik çözelti, bu membranlara pompalanır. İtme kuvveti nedeniyle, pozitif katyonlar negatif yüklü katyon değişim zarından kolayca geçer ve katoda doğru hareket eder ve anyon değişim membranı tarafından ise tutulurlar. Anyonlar için ise durum için tam tersi olarak gerçekleşir. Bu işlemin bir sonucu olarak, elektrodiyaliz düzeneği içindeki çözelti, bir tarafta iyon içeriği olarak konsantreedilirken, diğer taraftaki çözeltinin iyon içeriği azaltılarak seyreltilir.

Elektrodiyaliz uygulaması ile demineralizasyon işlemi peynir altında bulunan tuzlar bakımından incelendiğinde, sodyum, klor ve potasyum gibi tek değerlikli iyonların kolaylıkla peynir altı suyundan ayrıldığı, kalsiyum gibi çift değerlikli iyonların uzaklaşmasının ise daha zor olduğu görülür. Bunun başlıca sebebi iyonların hareket etme yetenekleridir. Bu yetenek, iyonların sahip oldukları hız, iyonların spesifik elektrik geçirgenliği ile doğru, ortamdaki elektriksel kuvvet, molekül konsantrasyonuyla ters orantılıdır.

Demineralizasyon işlemi için, anyon-katyon membranlı elektrodiyaliz, Moravia Lacto A.Ş. (Jihlava, Çek Cumhuriyeti)şirketinden temin edilen, tatlı (rennet) peynir altı suyu tozu ile negatif ve pozitif polaritede 3.5, 7, 10 ve 14 olarak dört farklı yüzdede uygulanır. Her deney öncesi, elektrodiyaliz cihazı damıtılmış su ile yıkanmıştır. Uygulama öncesi elektrodiyaliz düzeninde kullanılmak üzere çözeltiler hazırlanır. Seyreltim yapılacak çözelti kısmı için (diluate) dört farklı yüzdedeki peynir altı suyu çözeltileri hazırlanır. Konsantrasyon yapılacak kısım için HNO_3 çözeltisi pH'ı 2 olacak şekilde hazırlanır. Elektriksel çözelti için is 10 $g.l^{-1}$ konsantrasyonunda $NaNO_3$ çözeltisi hazırlanır. Her elektrodiyaliz deneyi öncesi ve sonrasında, elektrodiyaliz yapısında bulunan iyon değişim zarlarının düzgün çalışıp çalışmadığını kontrol etme amacıyla bir tuz model solüsyonu Na_2SO_4 kullanılarak hazırlanır ve uygulanır. Testin sonunda, Javg değerleri makro excel belgesi kullanılarak hesaplanmıştır.

Demineralizasyon işlemi sırasında, iletkenlik, pH ve sıcaklık değişiklikleri 5 dakika aralıklarla kaydedildi. Deney 16V sabit voltajda gerçekleştirilir ve seyreltilen çözeltinin (diluate) %95 demineralizasyonu sonucunda sonlandırılır. Kullanılan her farklı çözelti için bu zaman aralıkları kaydedilir ve her deney sonunda 45 ml örnek, kül içeriği ve kuru madde belirlenmesi için toplanılır.

Elektrodiyaliz uygulamasına ek olarak, ultrafiltrasyon ARNO 700 Clover tip boru şekilli seramik TAMI - Industries membranlarla, %7 oranında hazırlanan peynir altı suyu çözeltisi hazırlanarak uygulanır. Ultrafiltrasyon 50 kDa membran kullanılarak, 2 bar'lık bir sabit basınçta uygulanır ve 300 gram çözelti eldesi sonucunda sonlandırılır. Deney sırasında konsantrasyon faktörü 2 olarak alındı. Böylece 6 litre retentattan 3 litre permeate 3 litre de retentat edilir. Her deney öncesi hazırlanan solüsyondan ve her deney sonrası elde edilen permeate ve retentate örneklerinden 45 ml örnek kül içeriği, kuru madde ve protein analizi belirlenmesi için toplandı. Ayrıca, elektrodiyalizde kullanılan permeate ve retentate numuneleri de 1000 ml ± toplandı.

Her bir deney sonunda, elde edilen veriler, peynir altı suyu elektrodiyaliz uygulamasının verimliliğini ve işlevselliğini değerlendirmek amacıyla makro excel belgesine girildi. Bu tezde belirlenen başlıca faktörler J_{avg}, C_F, C_{D_DM}, Δm_{ASH}, α ve W olarak belirlendi. Hesaplanan bu faktörler, elektrodiyaliz işlemi için yeni bir yaklaşım olarak, özellikle peynir altı suyu ve peynir altı suyu ürünlerinin demineralizasyon işlemi optimizasyonu için iyi bir kaynak olarak kullanılabilir. Bu parametreler ile elektrodiyaliz ile peynir altı suyu demineralizasyon işlemini enerji, kalite, işlevsellik ve besin değerlerinin optimizasyonu ve iyileştirilmesi yönlerine yardımcı olabilir.

Sonuç olarak özetlemek gerekirse, bu çalışmada tatlı (rennet) peynir altı suyu, iyon değişim membranlı elektrodiyaliz yöntemi kullanılarak demineralizasyon yapılmış ve bu süreçte yeni bir yaklaşım olarak elektrodiyaliz yöntemini etkileyen parametreler belirlenerek bu projeye özel bir makro excel belgesinin yardımı ile hesaplanmıştır. Böylece peynir altı suyu elektrodiyaliz sürecini etkileyen faktörlerin belirlenmesi ve bu faktörlerin ilerki uygulamalarda optimizasyonu amaçlanmıştır. Buna ek olarak, ultrafiltrasyon işlemi sonrasında elde edilen permeate ve retentate çözeltileri elektrodiyaliz edilebilmişlerdir.
Herhangi bir sorunla karşılaşmadan, permeate ve retentate çözeltileri başarıyla demineralize edilmiştir. Bu çözeltilerin W değerlerine bakılarak, elektrodiyaliz öncesi uygulanan ultrafiltrasyon yöntemi, etkin bir şekilde gıda sanayinde adapte edilebilir. Elde edilen sonuçlara göre bu yöntemin yeni araştırma ve geliştirme çalışmalarına açık olduğu söylenebilinir.

Peynir altı suyu demineralizasyon işlemi, gıda teknolojisinde önemli ve kritik bir süreçtir. Özellikle peynir altı suyu proteinlerinin çeşitli membran teknikleri kullanılarak izolasyonu ve konsantre edilmesi alanında son birkaç yıldır yapılan çalışmalar hız kazanmıştır. Gıda sanayisi için değerli olan bu proteinler, kullanıldığı ürünün yapısal ve duyusal özelliklerinin iyileştirilmesine imkân vermektedir. Sahip oldukları yüksek protein içeriği nedeniyle peynir altı suyundan elde edilen protein izolat ve konsantratları, hem sporcular ve vücut geliştiriciler tarafından hem de vejeteryanlar tarafından gıda takviyesi olarak kullanılır.

Sonuç olarak, peynir altı suyunun elektrodiyaliz yöntemiyle demineralizasyon işlemi daha fazla araştırma ve incelemeye açıktır. Elektrodiyaliz yönteminin henüz tam olarak tespit edilmemiştir ve çözülmesi gereken pek çok problem bulunmaktadır.

Elektrodiyaliz yöntemi, çevreye güvenli, ekonomik, hızlı ve çok etkili bir yöntem olması nedeniyle, özellikle süt endüstrisi için büyük bir potansiyele sahiptir. Bu yöntemle tuz oranı yüksek peynirlerin de demineralizasyonu uygulanabilir.

1. INTRODUCTION

Membrane is a semipermeable structure that separates two homogeneous phases according to their attributes. Abbe Nollet is the first person study about membranes in 1748. Historical review and milestones of membrane technology are shown in Table 1.1 (Singh, 2015).

Table 1.1 : Historical development of membrane technology (Singh, 2015).

Event	Scientist	Year
Osmosis	Abbe Nollet	1748
Laws of diffusion	Fick	1855
Dialysis, gas permeation	Graham	1861, 1866
Osmotic pressure	Traube, Pfeffer, Van't Hoff	1860-1887
Microporous membranes	Zsigmondy	1907-1918
Distribution law	Donnan	1911
Membrane potential	Teorell, Meyer, Sievers	1930s
Hemodialysis	Kolff	1944
Skinned membrane	Sourirajan and Loeb	1959
Membrane-transport models	Kedem, Katachalsky, Lonsdale, Merten, Pusch, Sourirajan	1960-1970
Spiral-wound membrane element	Westmoreland, Bray	1965-1970
Hollow-fibre Rsmosis membrane	Mahon, Hoehn and Milford	1965-1970
Thin-film composite membrane	Cadotte and Rozelle	1972

Dates back then, novel applications have been found and the membrane industry is rapidly developing (Singh, 2015).

Membrane processes are ideal for food industry since:

- They are non-thermal,
- They are segmented and can be easily expandable,
- Do not require chemical agents,
- Do not require high energy for operation,
- Do not include phase transformation and

They have a great prospect for raw material application, recovery and reuse (Drioli and Romano, 2001).

1.1 Purpose of Thesis

There are two main objectives of this thesis. The first aim is trying to treat sweet dry whey by electrodialysis (ED) with cation-anion exchange membranes to assess the factors, which can influence efficiency and process of ED of whey.

Second objective is to evaluate ED process of retentate and permeate solutions, which are obtained after ultrafiltration (UF) treatment. For each product, ED was needed to identify and track changes should be necessary for optimum process conditions.

1.2 Literature Review

In this part, literature search about whey, membrane processes and ED applications will be explained.

1.2.1 Whey

Whey is a green-yellow colored liquid, which is cause by its riboflavin (vitamin B2) content, is the leading by product attained after precipitation and removal of caseins in cheese production, which contains high amount of protein and lactose (De Wit, 2001; Siso, 1996; Yorgun and Balcioglu, 2008). Lactose is the leading fraction (90%) among organic compounds of whey and lactose content of whey is between 39-60 $kg.m^{-3}$ (Ghaly and Kamal, 2004; Kisaalita et al., 1990). Fat content is around

0,99 to 10,58 kg.m^{-3} and protein content is about 1,4 to 8,0 kg.m^{-3}. Inorganic compounds are presented in the form of NaCl, KCl and calcium salts (predominantly phosphates) (Backus et al., 1988; Dragone et al., 2009; Venetsaneas et al., 2009). In Figure 1.1 whey components are shown.

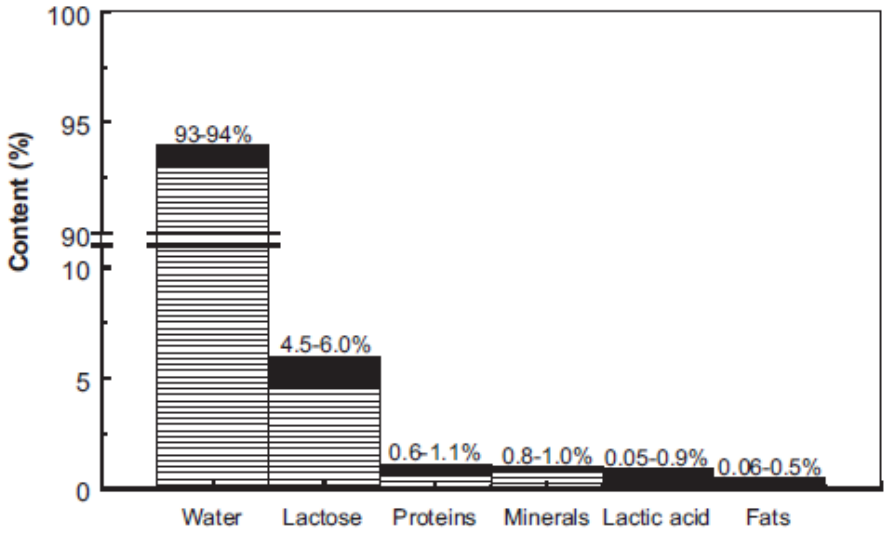

Figure 1.1 : Whey components (Prazerez et al., 2012).

pH of whey is determined by the type of whey and changes in the range of 3,8 to 6,5 (Ghaly, 2006). Whey contains 93-94% of water and contains almost everything was in milk such as lactose, soluble proteins, minerals, lactic acid and fats. As well as these molecules, whey can contains vitamins (B group), citric acid and some non-protein nitrogen compounds like urea and uric acid, β-lactoglobulin, α-lactoglobulin, serum albumin, immunoglobulins and lactoferrin (Bilbao, 1981; Casal et al., 2006; Kosikowski and Wierzbicki, 1973; Kosikowski, 1979; Panesar et al., 2007).

Whey and whey protein products' applications differ according to their composition and interaction with other products. About whey production and processing technologies, there has been a substantial development over the past three decays. Large amounts of whey is utilized to whey powder which has increased from 1,8 million to 2,6 million years between 1995-2006. Dry whey applications develop nearly at the same rate as milk bulks between 2003 and 2006.

Nowadays, application and processing methods of whey are evolving whereas producers are discovering more effective methods for production particularly in nutraceuticals, cheese production, and for dietary improvement. It is believed that novel whey products and processing techniques will be presented and current ones

will remain to progress their production and usages in the upcoming future (Foegeding et al., 2011).

1.2.1.1 Types of whey

Whey can be classified into several types varying on the processing order consequential in casein subtraction from fluid milk. Mainly the type of whey come upon initiates from the production of cheese or some other industrialized casein products which are based on coagulating casein by rennet, chymosin or other casein-coagulating enzymes.

The first basic whey type is sweet whey that it is obtained at a pH between 6,5-6,0 attributable to the rennet prompted coagulation of casein and the consequent whey drainage take place at the same pH intervals.

The second basic whey type is acid whey, obtained from fermentation processes. Additionally it can be obtained from manufacture of acid coagulated cheeses like cottage or quark, by the way of addition of organic or mineral acids in order to coagulate the casein (pH below 5.1). After water (93% as is basis), lactose (70–72% of the total solids) is the leading parts both of the sweet and acid wheys. Whey proteins (8–10%), and minerals (12–15%) follow subsequently.

Acidity, whey protein fraction's composition and mineral content are the leading differences among the acid and sweet whey types. The foremost difference between them is lactic acid content, acid whey can have 16 times more lactic acid than sweet whey which can affect the processing of whey along with the dietary properties (Chen et al., 2016; Jelen, 2011).

1.2.1.2 Whey components

Lactose, serum proteins and lactic acid are the three main whey components. They will be explained at the following titles.

Lactose

Lactose is a disaccharide is made up D-glucose and D-galactose linked together with β-glycosidic bond. Lactose a substrate is needed for the development of a comprehensive range of bacteria.

Under the influence of the heat, it reacts with free amino groups of proteins. In several products, as a result of its limited solubility it leads to crystallization (Hinkova et al., 2002).

Serum proteins

Whey proteins, which can be named as β-lactoglobulin, α-lactalbumin, immunoglobulins, lactoferrin, lactoperoxidase, serum albumin and glycomacropeptide are outstanding source of proteins, which have fundamental importance for young mammals and human consumption.

Owing to their disponibility, relatively low operation cost, high nutritional value and functional properties they are immensely used in the food industries (Cayot and Lorient, 1998; Hambraeus, 1982; Maubois, 1982; Pierre and Fauquant, 1986).

Lactic acid

Lactic acid has an extensive range of applications in the food industry such as in bakery, confectionery, dairy production, ready to eat meals, beverages.

Fermentation of carbohydrates is the furthermost applied technique between other manufacture processes because of its low financial cost (Wee et al., 2006).

1.2.1.3 Processing of whey

Whey products have exceptional functional properties and great dietary values. Consequently, there are several membrane technologies in order to isolate and concentrate of whey proteins (Caric et al., 2000).

Reverse osmosis (RO), microfiltration (MF), UF, and nanofiltration (NF) are the pressure driven membrane processes, which can be applied in order to obtain whey protein concentrates and whey protein isolates and ED.

Electro-deionization and ED are the electrically driven membrane techniques that are used for other whey products (Foegeding et al., 2011).

1.2.1.4 Application of whey

Whey ingredients or whey protein products are used for several functional and dietary purposes. Whey formerly is not used for human nutrition or further processing. They often have been considered as a waste or animal feed.

Today majority of whey is dehydrated and processed to powder form. In that way storage and transportation costs are reducing, and its shelf life is extending (Ferran, 2009).

Its application can be diverged as infant formulas, beverages, cheese products, special dietary products, bakery products, soups, sauces and many other products.

Whey products are utilized especially in sports drinks on account of their high protein content which is stable during the shelf life of the product.

On the other hand, a great majority of whey together with lactose, demineralized whey and modified whey protein products are important especially for infant formulas.

Industrial application of whey and whey products growing owing to their distinctive foaming, heat gelling and acid solubility properties. They can be used as a result of these properties in acid beverages, yogurts, foamed dairy desserts and in some other dairy and nondairy industries (Jelen, 2011; Foegetting et al., 2011).

1.2.2 Membrane processes

Membrane permits the transition of certain particles while holds some others under the effect of a driving force (Singh, 2015).

Membrane process can be categorized into four classes conferring to driving force prevail at the system:

1. Pressure gradient process: RO, UF, MF,
2. Concentration gradient process: Dialysis,
3. Partial pressure process: Pervaporation,
4. Electrical potential process: Electrolysis, ED (Strathmann, 2001).

Application of membrane processes not only conventional but also novel processes have many aspects to elucidate.

Together with conventional membrane processes (for instance UF, MF, RO and NF) and novel membrane processes (for instance membrane PV and MCs) have received demanding attention in agricultural - food and biotechnology industries.

Overview of applications can be seen in Table 1.2 (Lipnizki, 2010).

Table 1.2 : Applications of membrane technologies (Lipnizki, 2010).

Industry Area	Applications	Membrane Technologies
Dairy Industry	Milk products	Diafiltration (DF) MF RO UF
	Whey processing	DF ED MF NF UF
	Cheese making	DF UF
Alcohol Industry	Beer	DF MF RO
	Wine	DF ED MF NF RO
Fruit Juice Industry	Apple juice	DF RO UF
	Orange juice	UF
Food Additives Industry	Animal blood plasma	DF NF RO UF
	Gelatin	RO UF
	Carrageenan and other seaweed extracts	UF
	Pectin	DF RO UF
Sugar Industry	Beet sugar	DF ED MF RO UF
	Cane sugar	MF UF
Corn Starch Industry	Corn-based sweetener production	MF RO UF
	Other starch productions	RO UF
Water Industry	Potable water	RO UF
	Waste-water	MF NF
Bulk Biotechnology Industry	Antibiotics	DF RO UF MF
	Enzymes	DF RO UF NF
	Citric acid	DF RO UF
	Lactic acid	UF ED
	Lysine	RO UF
	Glutamic acid	RO UF
	Vitamin C	MF NF UF
	Xanthan	RO UF

1.2.3 Electrodialysis

One of the developing membrane processes in dairy industry is ED. As a unit operation ED is used for separation or concentration of charged particles with the help of its permselective membrane and under the effect of potential gradient in the system. ED is a filtration process by using membranes on the other hand it is different from other membrane processes like UF, RO and NF since ED does not separate particles according to their size, separates according to their electrical charges (Özdemir et al., 2008).

In ED, because of the semipermeable structure of membrane it either permits passage of cations or anions. Therefor the main purpose of using ED is concentration, separation or removing electrically charged particles from any solutions (Fidalio and Moresi, 2006). As well, it can be used in order to achieve good product quality especially for water treatment processes (Mega, 2006).

There are several advantages and disadvantages of ED comparing with other membrane technologies such as:

Advantages:

- ED operation requires low energy for separation of particles since it does not cause any phase change in the system,
- Desalinated brackish water with ED does not require any further pre-treatment only disinfection or chlorination is sufficient,
- In an attempt to concentrate of salt solutions to higher degree, pressure can be used in ED system due to osmotic pressure does not included.

Disadvantages:

- ED cannot removed colloids, SiO_2 and organic matters in the system,
- In order to keep ED at optimum condition, comprehensive controls are needed,
- To confirm the relevance of feed stream with ED system, materials of membranes and stack is vital,
- Concentration solution, which is attained after ED is one of the drawback since it is not recycled easily.

1.2.3.1 Basic concepts of electrodialysis

There are ion exchange membranes (IEM) in ED, which have two electrodes name as anode and cathode (Tado et al., 2015). Cathode is the negative end of the direct current (DC) source of ED while positive end is named as anode. When positive and negative ions are transferred in an aqueous solution, current flow is generated. The current flow is named as positive, while positively charged particles are transferred to the electrons in the outer circuit, and named as negative if negatively charged particles are transferred (Fidalio and Moresi, 2006).

ED device has different parts: DC generator, ED stack, piping, pumps, tanks and also conductivity, pH, flow rate, temperature and pressure measuring devices (Fidalio and Moresi, 2006). In Figure 1.2 ED from Mega is shown.

Figure 1.2 : Electrodialysis.

In ED stack two kinds of ion-exchange membranes present. First one contains negative ions which are set in the polymeric matrix, it is named as cationic membrane, and second one consists of positive ions the matrix is named as anionic membrane (Strathman, 2010). Electrodialyzer is the name of the equipment which ED happens and it contains these cationic-anionic membranes together with spacers (Mega, 2006).

1.2.3.2 Structure of an electrodialyzer

Parts of electrodialyzer are fastening frame, solution feeding frame, gasket, slot, spacer, electrode and electrode chamber, press are shown in Figure 1.3.

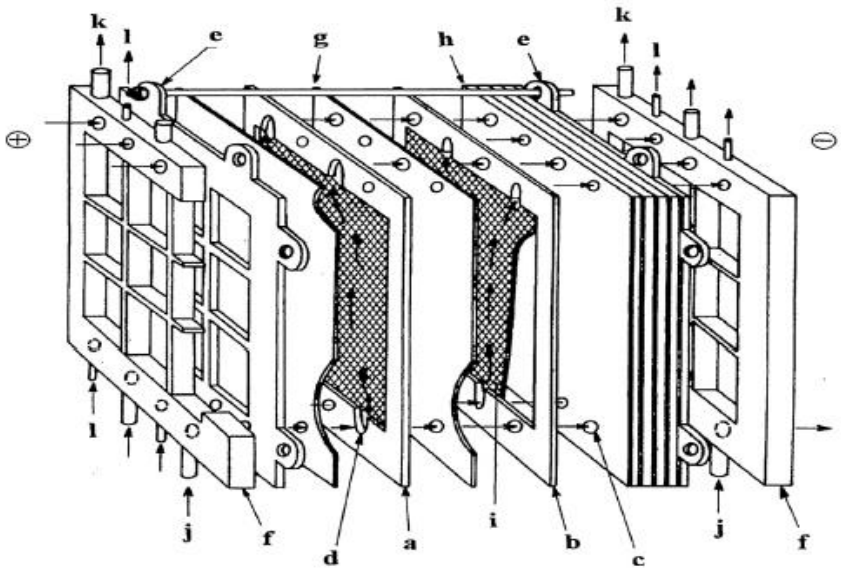

Figure 1.3 : Parts of an electrodialyzer: (a)Desalting cell. (b)Concentrating cell. (c)Duct. (d)Slot. (e)Fastening frame. (f)Feeding frame. (g)Cation exchange frame. (h)Anion exchange membrane. (i)Spacer. (j)Feeding solution. (k)Desalted solution. (l)Concentrated solution (Azechi,1980).

Electrode and electrode chamber

There are two electrodes in an electrodialyzer as anode and cathode. They categorized as flat, bar and net. Anode can be made up graphite, magnetite or platinum plated titanium and cathode can be made up iron or stainless steel.

Inside of the anode chamber, deterioration is one of the main problems as a result of oxidation of ionic membrane. For this reason, a buffer chamber should be placed between two partitions. Moreover, with the purpose of avoiding intercourse of two solutions together, a partition is placed between stack and an electrode chamber.

Inside of the cathode chamber, precipitation of magnesium hydroxide can be seen. For this reason inside of a cathode solution, an acidic solution is added and pH is control (Tanaka, 2015).

Gasket

Gasket is the part of the electrodialyzer is used for:

i. From feeding side to outer side; inhibition of solution leakage,

ii. Among desalting and a concentrating cell; inhibition of solution leakage,
iii. Among cationic and an anionic membrane; arranging distance.

From bottom side to upper side, feeding solution is inserted from the inlet duct, runs to the slot and discharged to the outlet slot at the duct.

It can be made from an ethylene vinyl acetate copolymer, rubber, polyethylene, polyvinyl chloride, etc (Tanaka, 2015). In Figure 1.3 gasket is shown.

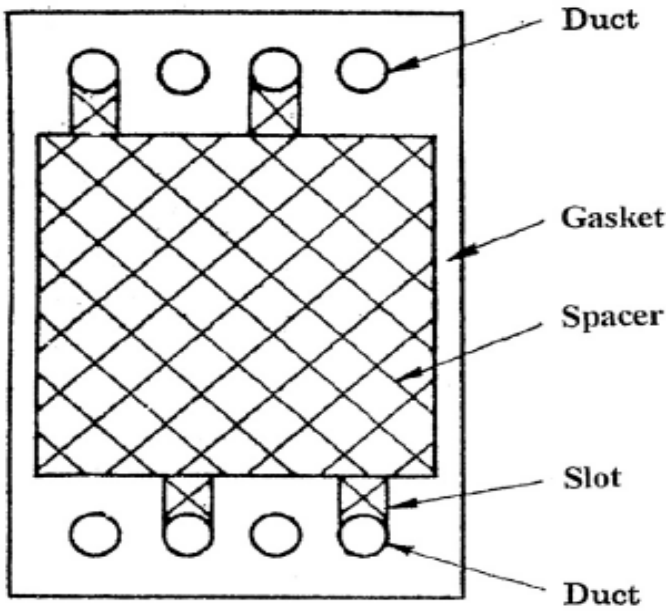

Figure 1.4 : Gasket of an electrodialyzer (Urabe and Doi, 1978).

Slot

Slot can be seen in the Figure 1.4, which is important for prevention of solution leakage (Tanaka, 2015).

Spacer

Spacer is the part of the electrodialyzer is important for arranging the distance between two membranes as well as it intensifies the limiting current density. An ideal spacer:

i. Friction head loss should be low,
ii. Influence of electric current screening should be low,
iii. Air release should be easy,
iv. Because of the precipitation of constituent parts within the feeding solution, it should have fewer blocking of flow pass (Tanaka, 2015).

Solution feeding frame

Solution feeding frame is used for feeding of solutions both dilute and concentrate from duck holes in the gasket (Tanaka, 2015).

Fastening frame

Fastening frame is the part of the electrodialyzer, which is used for integration of solutions both of, dilute and concentrate through the solution-feeding frame. It can be made from polypropylene, polyvinyl chloride, etc. (Tanaka, 2015).

Press

Press is the part of the electrodialyzer that it is used for pressure adjustment to 5-10 kg/cm^2 (Tanaka, 2015).

1.2.3.3 Working principles of electrodialysis

In order to transfer cations in the direction to the cathode and anions to the anode, an electrical potential difference should be exist between two electrodes. This driving force is the reason for permeation of anions to anion-exchange membrane conversely retention at cation- exchange membrane and also cations are free to pass through cation-exchange membrane while they are hold by anion-exchange membrane.

As a consequence of ion exchange in the ED circuit, one ions are concentrated which is named as concentrate while other solution is diluted as named is dilute.

A cell pair, which is a restating element in a stack, is composed of this cationic membrane with diluate, and anionic membrane with concentrate parts.

Among two electrodes a cell pair can be set several ways and membrane stack is the most common type and it can be equipped to 200 pairs. In the Figure 1.5 design of an ED stack is shown (Strathmann, 2010).

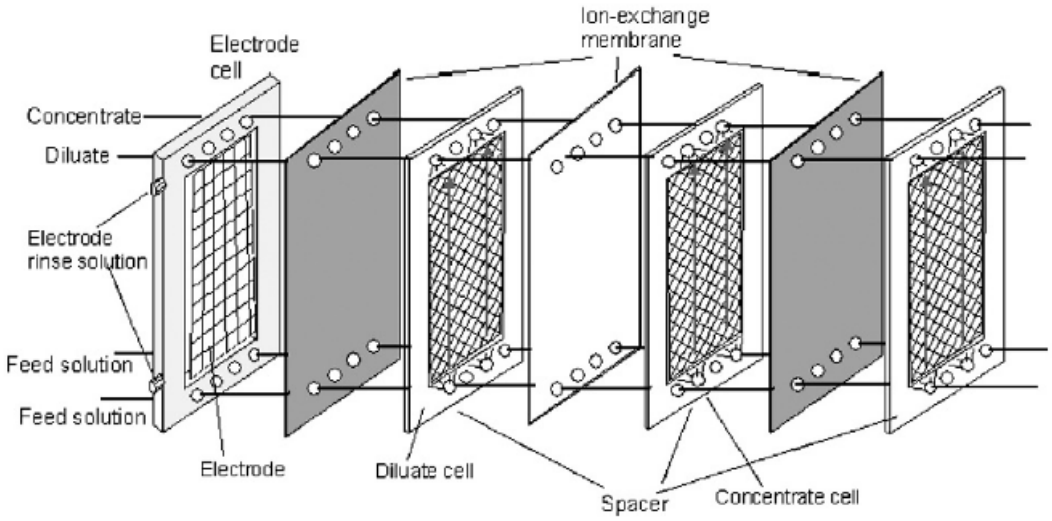

Figure 1.5 : Design of an electrodialysis stack (2010).

1.2.3.4 Ion exchange membranes

IEM can be classified into two groups as: anionic and cationic membranes which are ion exchange polymer matrix as a form of film layer. In the Figure 1.6 it is shown as cationic membranes consist of negative ions set to the matrix, and anionic membranes consist of positive ions set to the matrix (Strathmann, 2010).

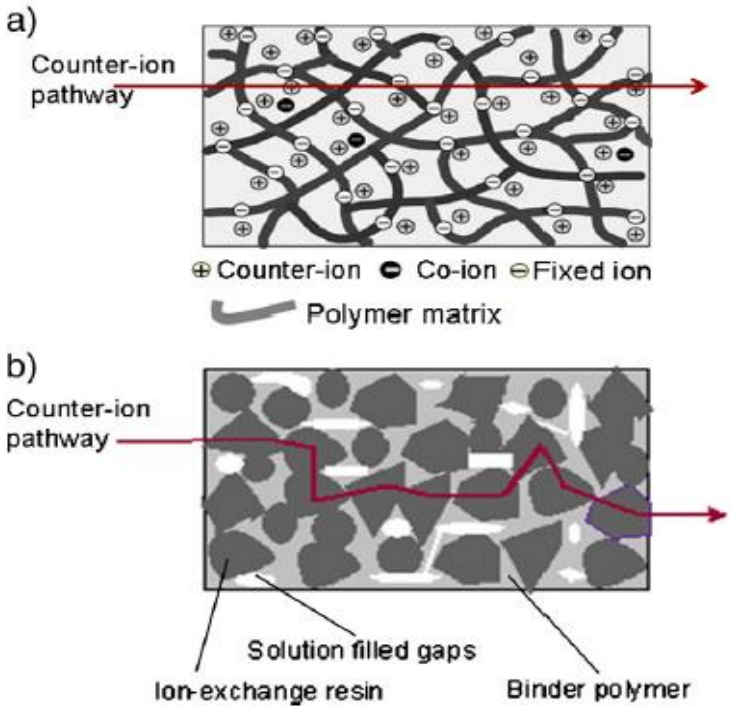

Figure 1.6 : (a)Homogeneous cationic membrane. (b)Heterogeneous anionic membrane made from a powder of ion-exchange resin and a folder polymer (Strathmann, 2010).

According to membranes, structure IEM classified into heterogeneous and homogeneous. Heterogeneous membranes made from a powder of ion-exchange resin and a folder polymer under high temperature as a consequence of ionic groups are group together and dispersed irregularly.

In contrast to heterogeneous membranes, in homogeneous membranes, ionic groups dispersed uniformly since homogeneous membranes are made straightly from ion exchange moiety.

Ion exchange membranes should be:

- Semi-permeability should be high,
- Electrical resistance should be low,
- IEM should has high mechanical, thermal and chemical stability,
- IEM should has low production costs (Strathmann, 2010).

1.2.3.5 Electrodialysis steps

ED process can be divided into 3 steps:

1) Pretreatment of raw materials:

pH adjustment and heat treatment adjustments include treatment of raw materials to prevent the growth or reduce the total number of bacteria in order to clean the raw material prior to use in ED. The extent and type of such operations depend on the type of raw material and its subsequent use.

2) Application of raw materials through the membrane:

Processed material is pumped through ED unit, which can contain up to 200 pairs of membranes. At this stage, separation of the raw material to concentrate is taken place. This process continues until a desired diluate conductivity, which indicates whether the material is sufficiently desalted or not.

3) Cleaning of equipment:

The cleaning process consists of five steps - the first rinse (3x 15 minutes) is taken place by using HNO_3 (0.5%, 45 minutes), second rinsing with demineralized water (3x 15 minutes), then used alkaline detergent (0.5% NaOH, 45 minutes), and finally washed membranes demineralized water (3 times, 15 minutes) (Greiter et al., 2002).

1.2.3.6 Bipolar membranes

Bipolar membranes are produced at the end of the 1980s as a novel type of electro-membrane, which consisted of chemically or physically composed anionic and cationic membrane and a hydrophilic layer at their intersection (Fidaleo and Moresi, 2006). Under the influence of an electric field, bipolar membranes (BPM) discharge water molecules into H^+ and OH^- ions (Mani, 1991). In Figure 1.7 bipolar membrane structure is shown.

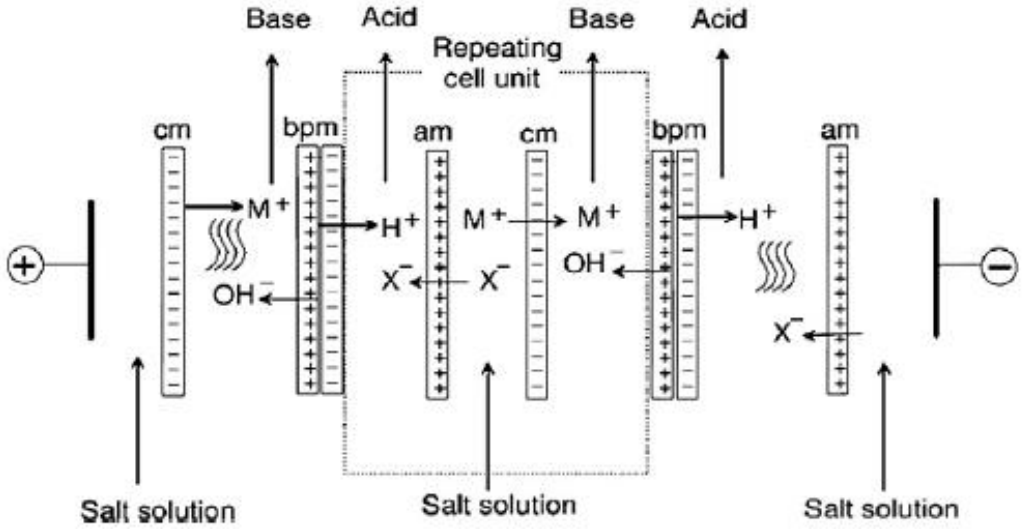

Figure 1.7 : Structure of a bipolar membrane (Strathmann, 2010).

Optimum bipolar membranes:

- At high current density, they should have low electrical resistance,
- Should have high water dissociation rates,
- Should have low co-ion transport rate,
- Should have high ion selectivity,
- Presence of strong acids and bases, they should have good chemical and thermal stability (Strathmann, 2010).

Application of bipolar membranes

Bipolar membranes can be applied to food processes in which chemical, enzymatic, and microbiological stabilization strongly depend on pH (Bazinet et al., 1998).

The application of ED with bipolar membranes has a rising prospective in chemical, biotechnology and water treatment industry as a consequence of cost-effective properties of them (Strathmann, 2010).

Applications of bipolar membranes can be listed as:

- Enzymatic browning inhibition in apple juice,
- Soy protein isolates production,
- Citric acid production,
- Vitamin C production (Tanaka, 2007)
- Acid and bases production from salts
- Acid recovery from fermentation
- SO_2 removal from flue gas (Strathmann, 2010).

In addition all above applications, bipolar membranes can be used for production of organic acids (Bailly, 2002; Quoc et al., 2011; Lameloise and Lewandowski, 2012). As well treatment of industrial effluents has received demanding examination attention (Graillon et al., 1996; Rehouma et al., 2013; Wei et al., 2011; Wang et al., 2014).

Application problems of bipolar membranes

Main problems are encountered in membrane system are low stability of IEM in the course of using acids and bases and high costs of the bipolar membranes establishment.

In addition to these problems, precipitation of multivalent ions, contamination of the products by salt ions and salt leakage through the bipolar membrane can be come across (Strathmann, 2010).

1.2.3.7 Electrodialysis applications

Among the other ion-exchange membrane technologies, traditional ED is undoubtedly the most significant one. At the beginning, it was produced in order to desalinate of brackish water to obtain drinkable water and nowadays it is likewise used for whey demineralization or fruit juice deacidification (Kawahara, 1992). Additional it is used to obtain table salt from concentrating of seawater, which is common especially in Japan or Korea owing not to have mineral salt deposits.

Industrial applications of ED, status of applications, limitations and key problems are listed in Table 1.3 (Strathmann, 2004).

Table 1.3 : Application of electrodialysis (Strathmann, 2004).

Industrial Applications	Status of Applications	Limitations	Key Problems
Brackish water desalination	Commercial	Concentration of feed and costs	Scaling, costs
Boiler feed water production	Commercial	Product water quality and costs	Costs
Waste and process water treatment	Commercial	Membrane properties and costs	Membrane fouling
Ultra-pure water production	Commercial	Product water quality and costs	Membrane biofouling
Demineralization of food products	Commercial or pilot phase	Membrane selectivity and costs	Membrane fouling, product loss
Table salt production	Commercial	Costs	Membrane fouling
Concentration of reverse osmosis brine	Pilot phase	Costs	Waste disposal

1.2.3.8 Factors affecting electrodialysis process

In order to assess ED results for different conditions, bellowed parameters calculated for each experiment.

Special software from the MemBrain s.r.o (Stráž p. Ralskem Czech Republic) was used for each calculation.

α is transport of water [H_2O quantity in grams transferred together with 1 g of salt].

$$\alpha = \frac{m_D,0 * DMf - m_D,final * DMd}{m_s,D,0 - m_s,D,final} \quad (1.1)$$

$$ms, D, 0 = c_s, FD, 0 * m_{FD}, 0 \quad (1.1a)$$

$$ms, D, final = c_s, FD, final * m_{FD}, final \quad (1.1b)$$

Where $m_{D,0}$ is mass of diluate before ED in kg, $m_{D,final}$ is mass of diluate after ED in kg, DM_F is dry matter of feed (g/kg), DM_D is dry matter of diluate (g/kg), $c\_s_{,FD},0$ is concentration of salt into the diluate at the beginning of experiment (g/kg), $m\_{FD},0$ is weight of salt into the diluate at the beginning of experiment (g), $c\_s_{,FD}$,final is concentration of salt into the diluate at the end of the experiment (g/kg), $m\_{FD,final}$ is weight of salt into the diluate at the end of the experiment (g).

C_F is capacity of ED [kg $_{\text{processed feed}}$ .m^{-2}.h^{-1}].

$$CF = \frac{m_D,0}{t_{final} * I_{ef} * W_{ef} * N} \quad (1.2)$$

$$ms, D, 0 = c_s, FD, 0 * m_{FD}, 0 \quad (1.2a)$$

Where c_$s_{,FD,0}$ is concentration of salt into the diluate at the beginning of experiment (g/kg), m_$_{FD,0}$ is weight of salt into the diluate at the beginning of experiment (g), t_{final} is time of ED in total (minutes), I_{ef} is ED current (A), W_{ef} is ED power (W), N is number of membranes in ED.

C_{D_DM} is the capacity of ED [kg $_{\text{produced dry matter}}$ .m^{-2}.h^{-1}].

$$CD_{DM} = \frac{m_D,final * DMd}{t_{final} * I_{ef} * W_{ef} * N} \quad (1.3)$$

Where $m_{D,}$final is mass of diluate after ED in kg, DM_D is dry matter of diluate (g/kg), t_{final} is time of ED in total (minutes), I_{ef} is ED current (A), W_{ef} is ED power (W), N is number of membranes in ED.

Δm_{Ash} is ash content change in diluate [%].

$$\Delta mAsh = \frac{m_D,0 * AshF - m_D,final * AshD}{m_D,0 * AshF} \quad (1.4)$$

Where $m_{D,0}$ is mass of diluate before ED in kg, Ash_F is ash content of feed (g/kg), Ash_D is ash content of diluate (g/kg).

J is flux of salt [g.m^{-2}.h^{-1}] (Strathmann, 2001).

$$Ji = \sum_k Lik\ Xk \quad (1.5)$$

Where L is phenomenological coefficient relating the fluxes to the driving forces, X is generalized driving force, i is volume, k is electrical charge.

W is power of ED which represent how many volts are used to processed for 1 kg of feed (W_h/kg_F).

$$W = \frac{W\_ef\ (w)}{M_{FD}(kg)}\ (W/kg) \quad (1.6)$$

Where W_{ef} is ED power (W) and M_{FD} is mass of feed (kg).

1.2.3.9 Problems of electrodialysis

ED has a great prospective and it has been developing date back 15 years with a 15% (Srikanth, 2004), in food industry application of it still not very prolonged. The major problems of ED can be named as clean ability, membrane fouling/scaling, stack design, membrane investment and replacement expanses, and other membrane processes challenging mainly as NF and ion exchange resins.

Soluble and insoluble impurities such as salts, microorganisms, organic and colloidal compounds are the reasons for membrane plugging and scaling problem, which can be prevented or minimized by applying additional membrane processes to feed such as MF, UF, NF or ion exchange resins.

Membrane shelf time can be prolonged to decay if feed is cleared with other membrane processes or it has a low current density, and stacks are well designed, otherwise shelf time of membrane would not be more than year.

By time cell voltage value, which changes between 0,8–1,5 V/cell intervals, is tend to be increased. As a consequence of this increase membranes should be replaced to restrain the electric consumption.

Membrane stack design is one of the main problems encountered. It is needed to be solved by improved understanding of mass transfer mechanisms, which is limited and needed further improvement together with other key factors like cell voltage, current density and efficiency (Gillery et al., 2002).

The current density and flux of ions passing through the membranes is defined by electric potential difference, which is the driving force of the ED system, and it directly affects the operation and development costs. When the current density is higher, the membrane surface area will become smaller and it decreases charge of the operation and development processes. On the contrary, the electric power consumption will increase (Fidaleo and Moresi, 2006).

1.2.3.10 Electrodialysis of whey

Whey as valuable organic material is obtained during manufacturing of cheese. As as well as whey, permeates which is attained after UF process should be efficiently processed. For instance powder infant formulas are formulated together with cow milk however; total ash and casein content of the breast milk and cow milk differ

than each other. As a consequence of this reason powdered milk is formulated together with whey and cow milk. On the other hand ED should be conducted to whey before standardization because of the high ash content in the dry matter (DM) of permeate and whey (Ideue, 1986; Tomita, 2004).

Demineralization by ED permits to produce lactose higher purity without using any chemical additives. It is crucial because a small salt concentration in a solution of lactose have influenced its crystallization rate (Goryachiy, 2007).

1.2.4 Ultrafiltration

UF is a membrane separation technique that separation process take place under the influence of pressure which is range between 0,2-4 bar. It has pore size 10–1000 A°, and retaining capability in molecular weight range of 300–10,00,000 Da. UF is applied for separation, concentration and diafiltration and is important due to its ability to work under mild operation conditions since it minimize degradation, deactivation and denaturation processes (Charcosset, 2012).

In the membrane process, there are three main components are named as feed, product (permeate) and reject (retentate). Because of the semipermeable structure, the separation process undergoes concurrent retention and releasing of permeate with the flow rate (Singh, 2015).

1.2.4.1 Dairy application of ultrafiltration

UF is mainly used for cheese making process. Retentate of milk are used for cheese manufacture, specialty milk products and total protein isolates however permeate samples are used for lactose manufacture and in fermented products (see Figure 1.8).

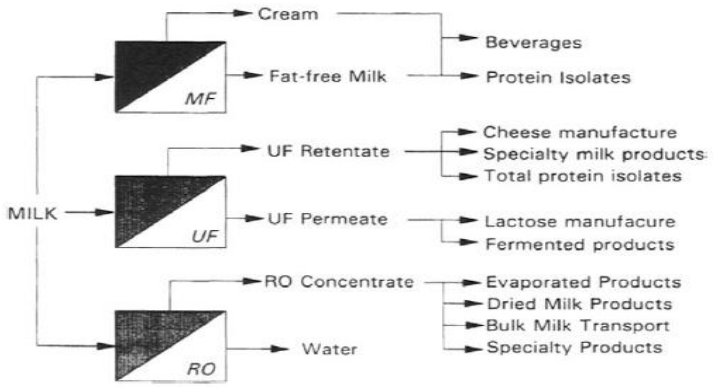

Figure 1.8 : Milk in membrane processes (Scott, 1998).

1.2.4.2 Ultrafiltration of whey

As a one of the most commonly used membrane technology, UF is used especially in dairy technology for concentration of milk, whey and implementation and purifying proteins (Kazemimoghadam and Mohammadi, 2007; Ogunbiyi et al., 2008).

Whey can be separated into protein concentrates, lactoglobulin and lactalbumin by UF and has several benefits such as increasing the protein content from 10-12% to 35%, 50% or 80% in dry basis (Scott, 1998). However, main problems in UF of whey to be encountered are fouling of membrane because of proteins and salts, which lead to pore blocking, concentration polarization or cake formation (Zumbusch et al., 1998). It can be solved by pretreatment processes to feed like pH treatment or using MF and centrifuge.

Whey fractioning by UF is shown in Figure 1.9 (Scott, 1998).

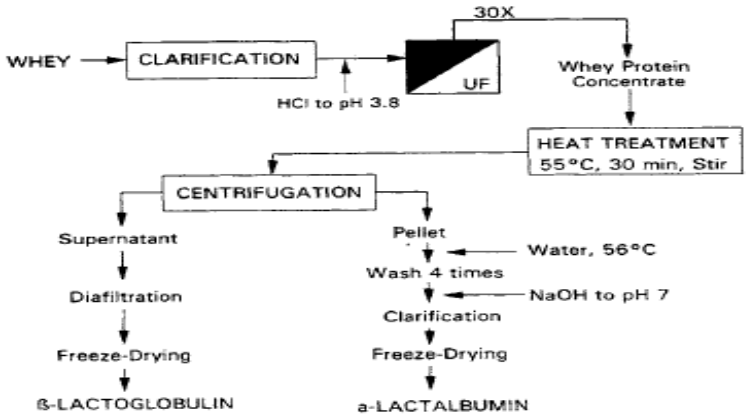

Figure 1.9 : Whey fractioning by ultrafiltration (Scott, 1998).

1.3 Hypothesis

ED with cation-anion exchange membranes is applied at negative and positive polarity for four different sweet dry whey concentrations as 3.5, 7, 10 and 14 % and factors as J_{avg}, C_F, C_{D_DM}, Δm_{ASH}, α and W, which influence efficiency and process of ED of whey, are calculated. It can be seen for all samples ED experiment takes shorter time at negative current than positive ones. Additionally, it is seen the higher the whey percentage is the longer the desalination processes occur.

Before ED, UF is applied at 7% whey concentration. It is aimed to test permeate and retentate solution in ED which are obtained after UF process. Permeate and retentate solution are sampled and tested for ash, protein and dry matter analysis. As a result

of these test it is seen as retentate has higher in dry matter, ash and protein contents than permeate due to high salt concentration. In addition, permeate and retentate samples have lower ash and dry matter content but higher in protein content than other ED's samples. However dry matter, ash and protein result of whey after ED is lower than the whey before ED. In ED permeate takes shorter time in ED according to retentate; because of the low protein content.

It is shown that retentate and permeate solution successfully applied in ED and can be desalinated. It can be concluded that UF before ED can be effectively adapted to food industry according to their low W values, and they are open to new inquiry and improvement.

2. MATERIAL AND METHOD

At the following titles, used materials and methods will be described.

2.1 Materials

Used chemicals and used materials will be explained below.

2.1.1 Used chemicals

Rennet whey powder (Moravia Lacto a.s., Jihlava, Czech Republic)
Nitric acid (Penta Ltd., Czech Republic)
Sodium sulfate (Penta Ltd., Czech Republic)
Sodium hydroxide (Penta Ltd., Czech Republic)
Trichloroacetic acid (Penta Ltd., Czech Republic)
Sodium chloride (Penta Ltd., Czech Republic)

2.1.2 Used instruments

ED unit P EDR-Z/4x with heterogeneous RALEX membranes (Mega Inc., Czech Republic)
Conductivity-pH meter pH / cond 340i (WTW, Germany)
Electric laboratory furnace LE 5.11 (Lac Ltd., Czech Republic)
Drying EcoCELL (Brno medical equipment SpA, Czech Republic)
Ultrafiltration unit ARNO 700 with tubular ceramic (TAMI-Industries, Germany)
Kjeltec 8420 Auto Sampler System 1030 (Foss Analytical, Denmark)

2.2 Methods

Applied methods will be explained at the following titles.

2.2.1 Determination of dry matter

In drying oven, sea sand was poured into the aluminum dishes, and dishes with their lids alongside after mixing rods were pre-heated 1 hour in drying oven at 102 ± 2 °C.

After 1 hour, lids were placed into the dishes and immediately transferred to the desiccator. Dishes were allowed to cool to room temperature (at least 30 min). 5 g of samples were weighed into the dishes in analytical accuracy. Dishes together with lids were placed into the drier at 102 ± 2 °C for 3 hours. After 3 hours, dishes together with lids were placed into desiccator and cool to room temperature. Cooled dishes were weighed and this procedure was repeated until the difference in mass between two following weighing does not exceed 1 mg.

Calculation of the dry matter of the sample was carried out according to the formula:

The total solids content, expressed as a percentage by mass, W_s is equal to:

$$Ws = \frac{m2-m0}{m1-m0}\ x\ 100 \qquad (2.1)$$

Where m_0 is the mass, in grams, of the dish, lid and sea sand; m_1 is the mass, in grams, of the dish, lid and sample portion; and m_2 is the mass, in grams, of the dish, lid and dried sample portion.

Measurements were performed two times; results are presented as average values (ISO 6731).

2.2.2 Determination of ash content

Porcelain crucibles placed into ash furnace oven at 550°C. After 4 hours, crucibles transferred to the desiccator and allowed to cool to room temperature). 5 g of samples were weighed into the crucibles in analytical accuracy. Thereafter crucibles were placed into the programmable furnace for drying and pre-ashing steps increases the temperature by 50 °C/h up to 550 °C and maintains the temperature of the oven at 550 °C for 6 hours (ISO 8070:2007).

After 6 hours, crucibles were placed into desiccator to cool to room temperature. Cooled crucibles were weighed and ash content calculated as:

$$Wp = \frac{mp - mk}{mv - mk}\ x\ 100 \qquad (2.2)$$

Where Wp ash content in the sample in %, mp weight of the sample after ashing in g, mk crucible weight in g and mv sample weight before ashing in g.

Measurements were performed two times; results are presented as average values.

2.2.3 Determination of protein content by the Kjeldahl method

The total protein content was determined by the Kjeldahl method using Kjeltec 8420 Auto Sampler System. Mineralization digestion was carried out in tubes with added sulfuric acid, 5 ml of hydrogen peroxide and 2 tablets of catalyst ($K_2SO4 + CuSO_4$).

Mineralization blocks allow simultaneous mineralization up to 20 samples and regulated gradual warming to a temperature of 420°C.

Distillation of the ammonia takes place in a distillation unit. To complete distillation of ammonia from the mineralizer will occur in about 2 to 4 minutes.

Subsequently distillation is followed by automatic titration with 0.1 M HCl (ISO 8968-3, 2007).

Measurements were performed two times; the results are presented as average values.

2.2.4 Determination of pure protein

5 ml of sample was weighed in a 150 ml beaker with an analytical accuracy. A sample is precipitated by addition of 5 ml of a 25 wt% solution of trichloroacetic acid (TCA), and stirred by rod.

After 15 minutes, solution was poured to the pre-wetted filter paper with 12,5 wt% TCA.

Subsequently, precipitate was washed three times with 10 ml of 12.5 wt% TCA. The filter paper and the precipitate inserted into to the digestion tube to determine the pure protein content.

The nitrogen content of the sample, W_N expressed as a percentage by mass, is calculated according to the following equation:

$$Wn = \frac{1{,}4007\ x\ (Vs - Vb) x\ Mr}{m} \qquad (2.3)$$

Where Vs is the volume (ml), 0,1 $mol.l^{-1}$ HCl solution used in the determination, Vb is the volume (ml), 0,1 $mol.l^{-1}$ HCl solution used in the blank test, Mr is the exact molarity of HCl solution, and m is the numerical value of the mass, in grams, of the test portion (ISO 8968-5, 2001).

2.2.5 Determination of crude protein

In order to calculate the crude protein content, Wp, expressed as a percentage by mass, following equation is used:

$$Wp = Wn\ x\ 6{,}38 \tag{2.4}$$

Where W_N is the nitrogen content of the sample expressed as a percentage, 6,38 is the generally accepted multiplying factor to express the nitrogen content as crude protein content (ISO 8968-5, 2001).

Measurements were performed two times; results are presented as average values.

2.2.6 Determination of pH

Determination of the pH of the samples was performed using a pH meter with a glass electrode.

2.2.7 Determination of conductivity

The conductivity of the samples was measured using a conductivity meter pH / Cond 340i.

2.2.8 Electrodialysis

ED was carried out on an ED unit P EDR-Z/4x, which is made of several basic parts as ED volume:

- Tightening plate with integrated electrodes
- Anion - cation exchange membranes
- Grids and distributor
- Bolts
- Trays of diluate, concentrate and electric solutions
- Pumps (centrifugal sealless)
- Rotameters
- Cells for pH and conductivity probe
- Electric source (2A, 24V)

For the ED experiments ED with anion - cation exchange module is shown in Figure 2.1.

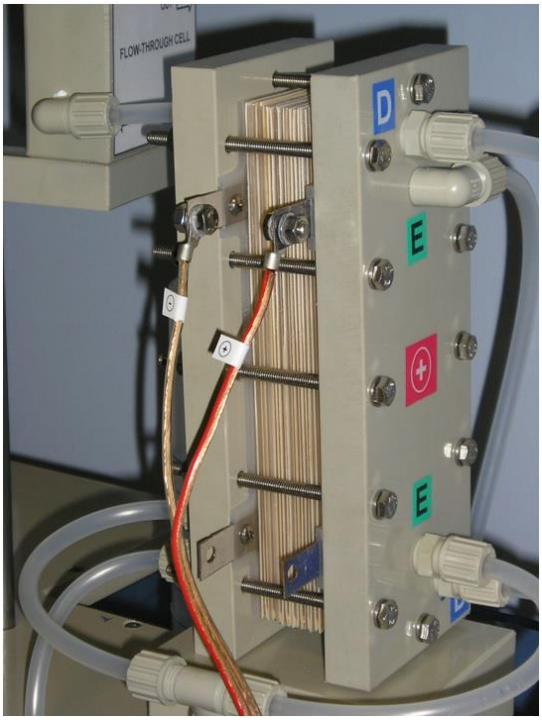

Figure 2.1 : Electrodialysis module EDR-Z/10-0.8.

Before each experiment, the device was washed with distilled water 3 times for 15 minutes. For the diluate part, whey solution was used with different concentration. For the concentrate part HNO_3 solution was prepared together with distilled water and its pH should be 2. The electrical solution was used a solution of $NaNO_3$ at a concentration of 10 $g.l^{-1}$. Volumes of diluate and concentrate were ± 1000 ml solution and for electrical solution ± 250 ml.

Before and after ED, in order to check the membranes working properly or not, a saline test for a model solution Na_2SO_4 (concentration) was prepared and conducted. For that purpose, Na_2SO_4 solution was prepared 5 $g.l^{-1}$ and tested to %95 of desalination. At the end of the test, J_{avg} values are calculated by using macro excel document.

During the experiment changes in conductivity, pH, and temperature were recorded 5 minutes intervals.

The experiment was conducted at a constant voltage of 16 V. The ED was terminated after reaching 95% desalination of the diluate. At the end of the each experiment 45 ml of diluate was collected in order to determine ash content and dry matter.

After each ED usage, below procedure is followed for purification of the membranes:

- 5% NaOH solution for 45 minutes,
- Demineralized water 3 times for 15 minutes,
- 5% HNO_3 solution for 45 minutes,
- Demineralized water 3 times for 15 minutes.

2.2.9 Ultrafiltration

UF unit is used from ARNO 700 with tubular ceramic TAMI-Industries membranes type Clover. 7% of whey solution was prepared for UF. The experiment was conducted at a constant pressure of 2 Bar. 50 kDa membrane was used for UF of whey solution (see Figure 2.2). The UF was terminated after obtaining 300 grams permeate. Concentration factor during UF was 2, because of obtaining 3 liters of permeate and 3 liters of retentate from the 6 liters of feed.

At the end of the each experiment 45 ml of feed, permeate and retentate were collected in order to determine ash content, dry matter and protein analysis.

Correspondingly permeate and retentate samples collected ± 1000 ml for ED experiment.

Figure 2.2 : Ultrafiltration unit ARNO 700.

3. RESULTS AND DISCUSSION

At the following titles, obtained results will be explained and discussed.

3.1 Whey Properties

Sweet whey with a different concentration was used for ED and UF processes was analyzed to characterize in order to determine ash, dry matter and protein content. Table 3.1 shows the analysis of dry matter, ash

Table 3.1 : Whey properties.

Whey Concentration	Dry Matter Results %	Ash Results %	Protein Results %
3,5 % Feed	2,99±0,01	0,22±0,01	0,33±0,02
7 % Feed	5,23±0,03	0,45±0,01	0,49±0,01
10 % Feed	8,66±0,02	0,70±0,02	0,84±0,02
14 % Feed	11,55±0,01	0,90±0,03	1,16±0,03
Ultrafiltration Feed 7 %	6,02±0,02	0,47±0,01	0,66±0,04
Retentate Feed	6,34±0,04	0,52±0,02	0,69±0,03
Permeate Feed	5,24±0,04	0,49±0,03	0,11±0,03

Table 3.1 shows that the higher the whey percentage is the more dry matter, ash and protein content is obtained.

As a result of UF processes, retentate's protein content is higher than the permeate and feed. Moreover, permeate has the highest ash content according to ash in dry matter results among them.

3.2 Saline Test Model Solution Results

Before and after ED, prepared a saline test for a model solution Na_2SO_4 (concentration) is tested and its J_{avg} and Δm_{ASH} values are calculated (see Table 3.2).

Table 3.2 : Saline test's results.

Model Solution	Current	J_{avg} ($g.m^{-2}.h^{-1}$)
Before ED	+	1196,46
Before ED	-	1334,46
After ED	+	1369,85
After ED	-	1525,17

3.3 Electrodialysis Results

The results for samples, which are collected after ED, are shown in Table 3.3. Results are calculated for different whey concentration and +/- current.

Additionally, ED results of ultrafiltrated samples are shown in Table 3.4.

Table 3.3 : Electrodialysis results.

Whey Concentration	Current	J_{avg} ($g.m^{-2}.h^{-1}$)	C_F ($kg.m^{-2}.h^{-1}$)	C_{D_DM} ($kg.m^{-2}.h^{-1}$)	Δm_{ASH} (%)	α (g/g solid)	W/m_F (Wh/kg_F)
3,5 % Diluate	+	80,88	20,50	0,53	94,46	4,58	1,38
3,5 % Diluate	-	88,14	21,50	0,54	95,56	5,21	1,60
7 % Diluate	+	164,50	15,44	0,67	91,59	3,24	2,91
7 % Diluate	-	170,37	16,87	0,82	93,41	3,54	2,97
10 % Diluate	+	252,39	15,36	1,09	89,15	1,44	3,65
10 % Diluate	-	263,32	15,76	1,11	90,29	1,53	3,82
14 % Diluate	+	287,84	11,29	1,12	79,67	0,57	4,72
14 % Diluate	-	303,40	11,71	1,15	75,24	0,70	5,77

J_{avg} is the flux, which shows the number of ions passing through membrane in one hour. ED results show that the higher the whey percentage is the more J_{avg} is obtained. It is because of the higher mineral content from 3,5 to 14% of whey. Additionally, for all of the samples, flux values are higher in negative current than

positive one since at negative current, ED works faster, and it increases the number of ions passing through membrane.

C_F is the capacity of ED, which is calculated according to the kg of feed processed in one hour. C_F is decreasing when the percentage of whey increasing. The more the salt concentration is the membrane fouling and scaling is happened which decreasing capacity of ED.

C_{D_DM} is the capacity of ED, which is calculated according to the kg of dry matter produced in one hour. C_{D_DM} is parallel to the whey percentage, it is increasing when the percentage of whey increasing since the dry matter content is higher from 3,5 to 14 % of whey. In addition, all of the negative current's values have higher results.

Δm_{ASH} is the ash content percentage change in diluate. Since Δm_{ASH} is contrast with whey percentage, from 3,5 to 14% of whey, ash content is increasing so Δm_{ASH} is decreasing.

α is the grams of transferred water together with one gram of salt through membranes. It is decreasing with increased whey percentage. When whey percentage is increasing also ED capacity is decreasing. Owing to this fact, transference of water with salt through membrane is also decreasing.

W is the power consumption, which is calculated with the electric consumption dividing to kg of processed feed. When whey percentage is increased required power for desalination also W is increased. As a result W is parallel to whey percentage. Additionally, it can be seen that both of the ultrafiltrated samples' W values are small enough to be operated for food industry.

Table 3.4 : Electrodialysis results of ultrafiltrated samples.

Whey Concentration	Current	J_{avg} ($g.m^{-2}.h^{-1}$)	C_F ($kg.m^{-2}.h^{-1}$)	C_{D_DM} ($kg.m^{-2}.h^{-1}$)	Δm_{ASH} (%)	α (g/g solid)	W/m_F (Wh/kg_F)
Retentate Diluate	+	114,79	11,17	0,52	92,63	5,77	2,97
Retentate Diluate	-	130,57	11,93	0,60	93,66	6,02	3,22
Permeate Diluate	+	190,86	17,03	0,96	91,14	4,58	2,29
Permeate Diluate	-	197,77	18,80	1,13	91,71	5,46	2,56

Permeate has the highest J_{avg} between permeate and 7 % of whey solution due to its high salt concentration. Moreover, retentate has the lowest value because of the UF process it has lower salt content.

C_F is higher in permeate since it can be processes faster than retentate. As a result of high protein content of retentate, the rate of membrane fouling and scaling is more than retentate and 7% of whey solution.

C_{D_DM} of permeate has higher value than retentate meanwhile the dry matter content is higher in permeate solution. In addition, negative current's values have higher results.

Δm_{ASH} value of permeate is lower than the retentate's value since Δm_{ASH} is contrast with salt concentration.

α is decreasing with increased salt content. Ever since permeate has more salt than retentate, its ED capacity is lower than permeate. The more the salt content is it is harder to transfer the water with salt through membrane.
W of retentate is higher than permeate since it is rich in protein which requires more energy for ED.

3.3.1 Calibration curves

According to dry matter, ash and conductivity results of different concentration whey from feed (7%) to 95% desalted whey; calculation curves is drawn and shown in Figure 3.1 and 3.2 for statistical analysis.

Table 3.5 : Results of whey from feed (7%) to 95% desalted whey.

Whey	Conductivity (mS/cm)	ASH (g/kg)	Dry Matter (g/kg)	ASH/DM
Feed (7%)	5,25	3,97±0,01	57,02±0,02	6,97
12,5% desalted whey	4,59	3,52±0,02	56,92±0,01	6,18
25% desalted whey	3,93	2,77±0,01	57,08±0,01	4,86
50% desalted whey	2,63	2,06±0,03	56,65±0,02	3,64
75% desalted whey	1,31	1,61±0,01	55,78±0,01	2,89
95% desalted whey	0,26	0,73±0,02	54,97±0,01	1,32

It can be seen in Table 3.5; from 7% of whey to 95% desalinated whey, conductivity, ash content and dry matter is decreasing since salt concentration is decreasing.

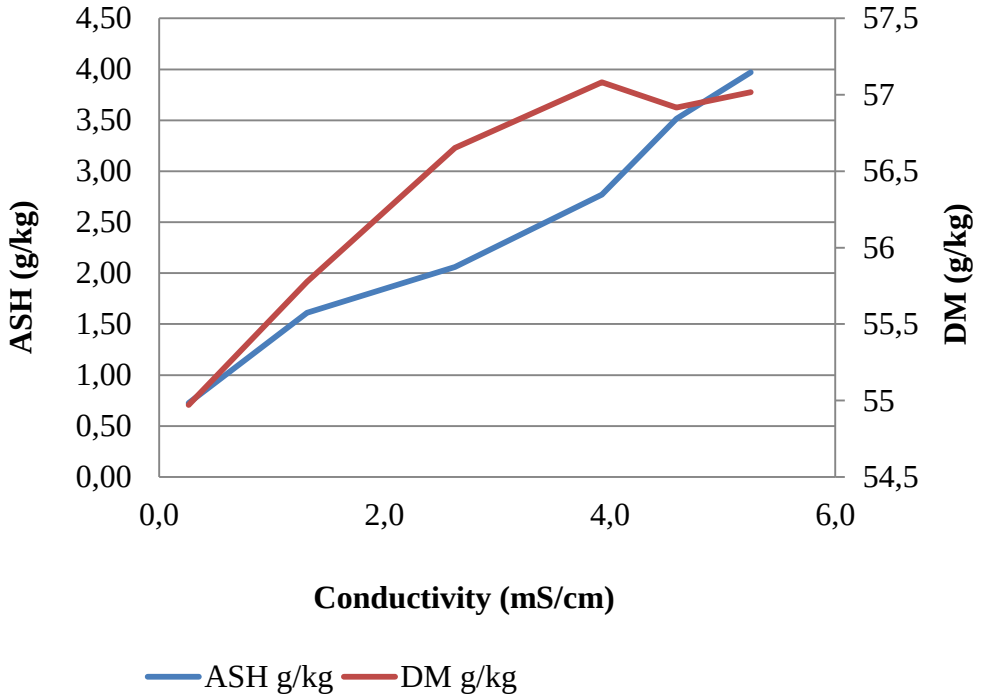

Figure 3.1 : Ash and dry matter results.

For ash:

$$y = 0{,}0319x^2 + 0{,}4412x + 0{,}7344 \quad (3.1)$$

$$R^2 = 0{,}9834 \quad (3.2)$$

where x is conductivity (mS/cm) at 25°C and y is ash (g/kg).

For dry matter:

$$y = -0{,}1149x^2 + 1{,}0419x + 54{,}676 \quad (3.3)$$

$$R^2 = 0{,}9916 \quad (3.4)$$

where x is conductivity (mS/cm) at 25°C and y is dry matter (g/kg).

For ash in dry matter:

$$y = 1{,}0708x + 1{,}1008 \quad (3.5)$$

$$R^2 = 0{,}9768 \quad (3.6)$$

where x is conductivity (mS/cm) at 25°C and y is ash in dry matter (g/kg).

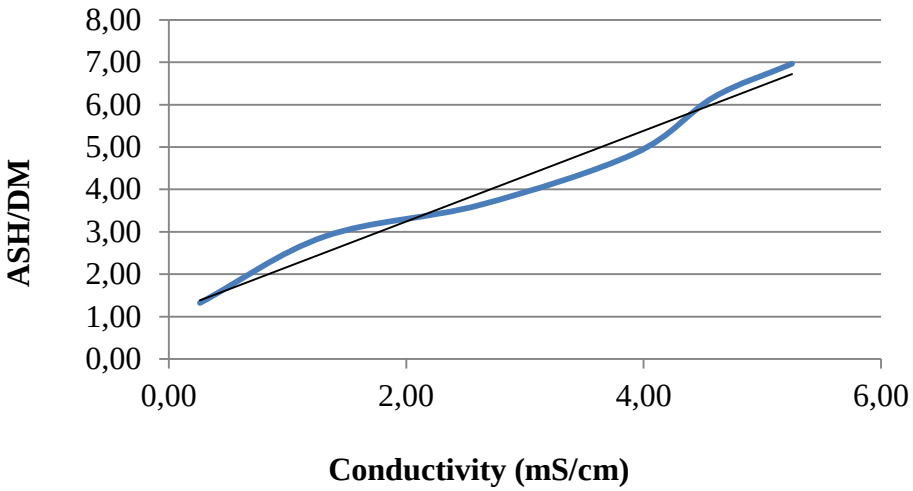

Figure 3.2 : Calibration curve of ash in dry matter.

3.2.2 Dry Matter, ash and protein results of electrodialysis

In Table 3.6, from ED for different concentration diluate samples dry matter, ash and protein results are shown.

Table 3.6 : Dry matter, ash and protein results.

Diluate Sample Name	Current	Dry Matter %	Ash %	Protein %
3,5%	+	2,63±0,04	0,01±0,01	0,31±0,01
3,5%	-	2,68±0,01	0,01±0,01	0,31±0,01
7%	+	5,08±0,03	0,03±0,01	0,33±0,01
7%	-	5,13±0,01	0,04±0,01	0,33±0,01
10%	+	7,13±0,04	0,04±0,02	0,83±0,04
10%	-	7,35±0,01	0,05±0,01	0,87±0,01
14%	+	10,15±0,01	0,23±0,01	1,06±0,01
14%	-	10,11±0,03	0,19±0,03	1,06±0,01
Permeate	+	4,40±0,01	0,04±0,01	0,04±0,02
Permeate	-	4,50±0,01	0,03±0,02	0,05±0,01
Retentate	+	5,37±0,02	0,04±0,01	0,86±0,02
Retentate	-	5,56±0,01	0,04±0,02	0,87±0,01

Table 3.6 shows that dry matter, ash and protein contents are increasing with increased whey concentration.

Moreover, for ultrafiltrated samples, retentate has higher in dry matter, ash and protein contents than permeate due to high protein content. In addition, permeate and retentate samples have lower ash and dry matter content but higher in protein content than other ED's samples.

Dry matter, ash and protein result of whey after ED is lower than the whey before ED. It can be said that ED is applied successfully and desired ash and dry matter contents are obtained.

3.2.3 Changes of conductivity and pH during electrodialysis

All of the ED's results are shown in Figures from 3.3 to 3.14 and for each concentration diluate and concentrate solutions are graphed conductivity versus time.

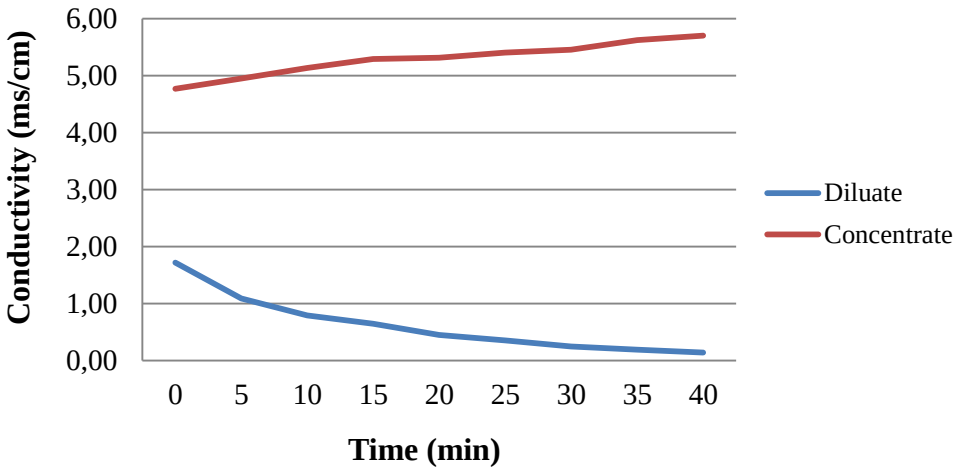

Figure 3.3 : Changes of conductivity of diluate and concentrate during ED of 3,5% whey at positive current.

40 minutes is requires for 95,45% desalination of 3,5 % whey at positive current.

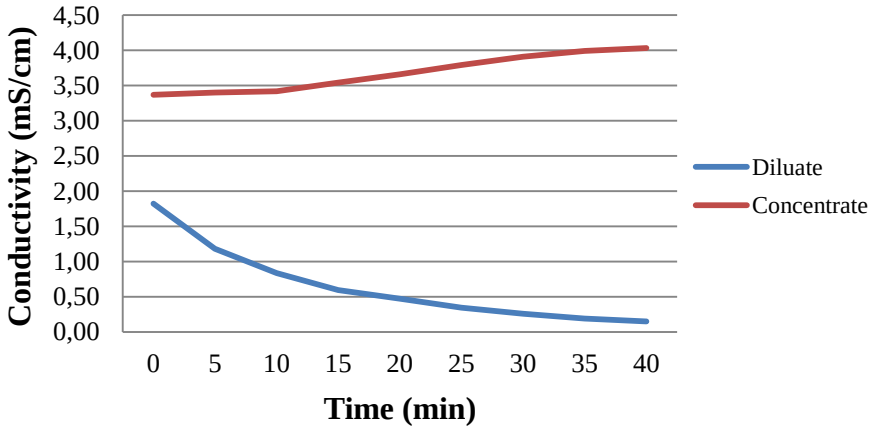

Figure 3.4 : Changes of conductivity of diluate and concentrate during ED of 3,5 % whey at negative current.

40 minutes is requires for 94,83% desalination of 3,5 % whey at negative current.

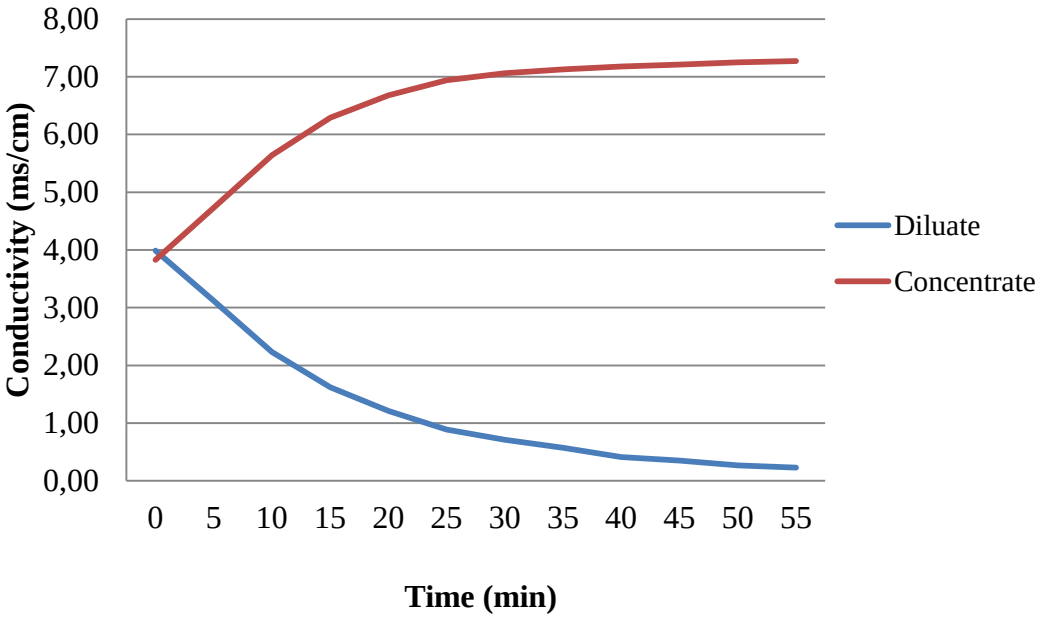

Figure 3.5 : Changes of conductivity of diluate and concentrate during ED of 7 % whey at positive current.

55 minutes is requires for 95,21% desalination of 7 % whey at positive current.

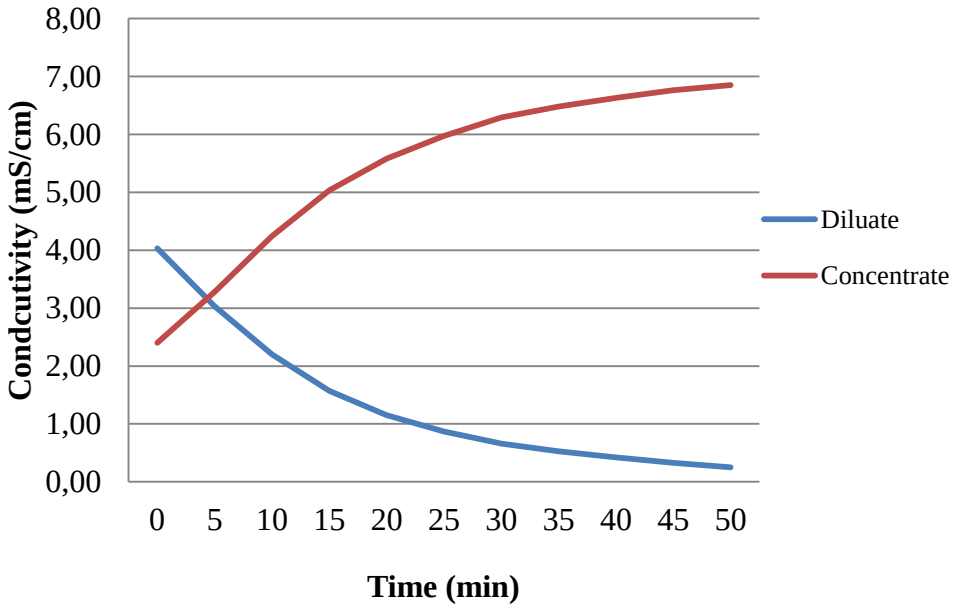

Figure 3.6 : Changes of conductivity of diluate and concentrate during ED of 7 % whey at negative current.

50 minutes is requires for 95% desalination of 7 % whey at negative current.

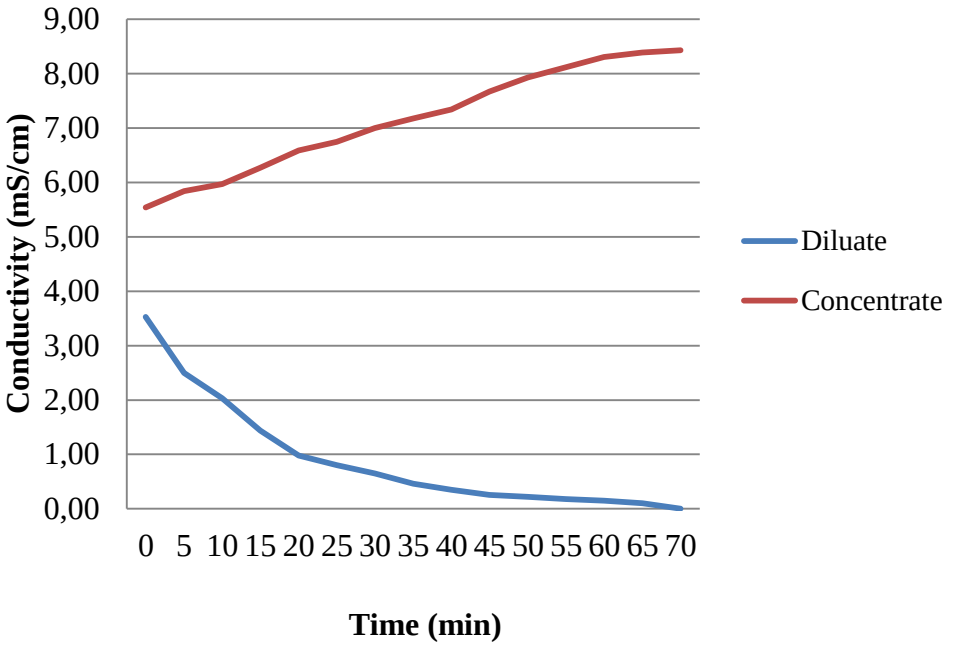

Figure 3.7 : Changes of conductivity of diluate and concentrate during ED of 10 % whey at positive current.

70 minutes is requires for 99,01% desalination of 10 % whey at positive current.

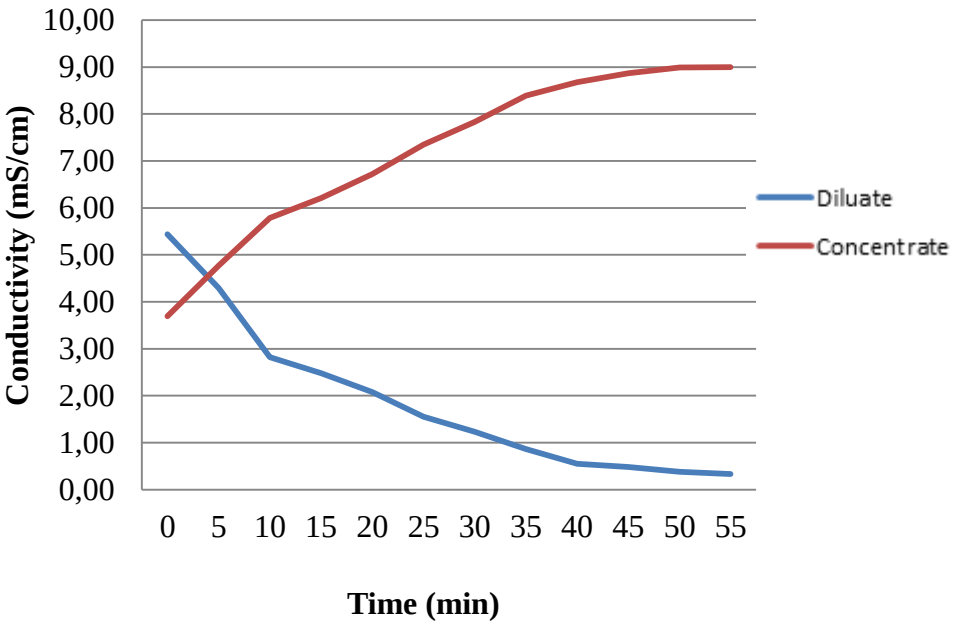

Figure 3.8 : Changes of conductivity of diluate and concentrate during ED of 10 % whey at negative current.

55 minutes is requires for 95,01% desalination of 10 % whey at negative current.

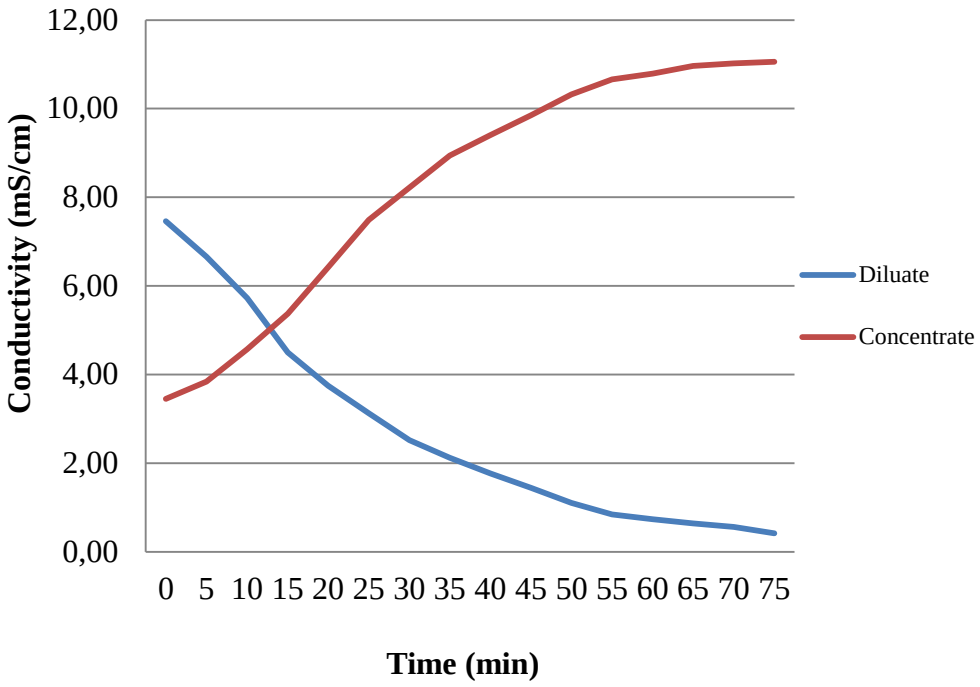

Figure 3.9 : Changes of conductivity of diluate and concentrate during ED of 14 % whey at positive current.

75 minutes is requires for 94,85% desalination of 14 % whey at positive current.

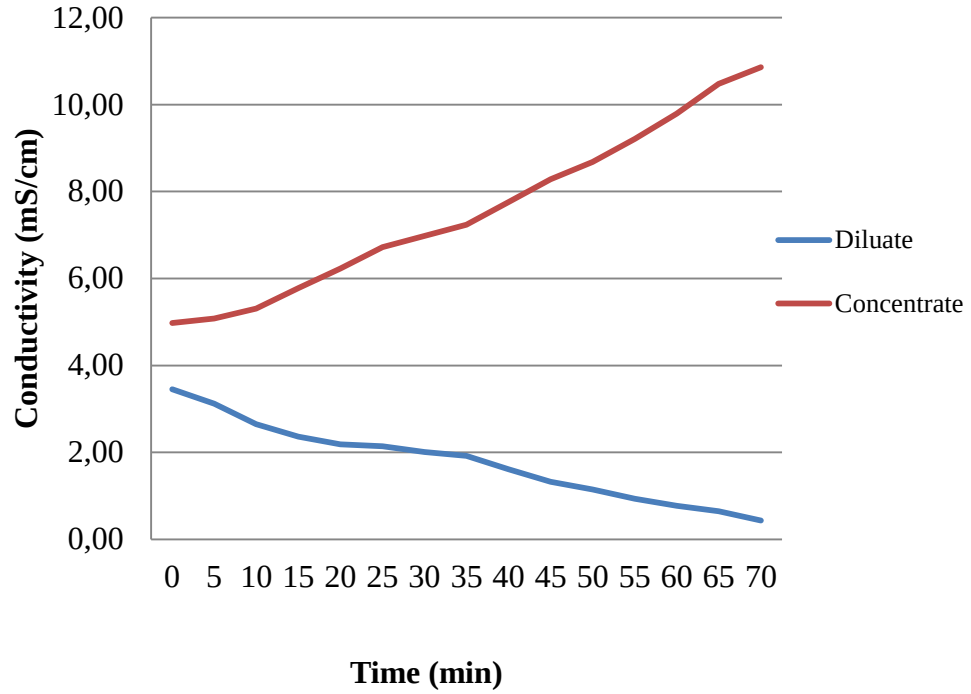

Figure 3.10 : Changes of conductivity of diluate and concentrate during ED of 14 % whey at negative current.

70 minutes is requires for 94,97% desalination of 14 % whey at negative current.

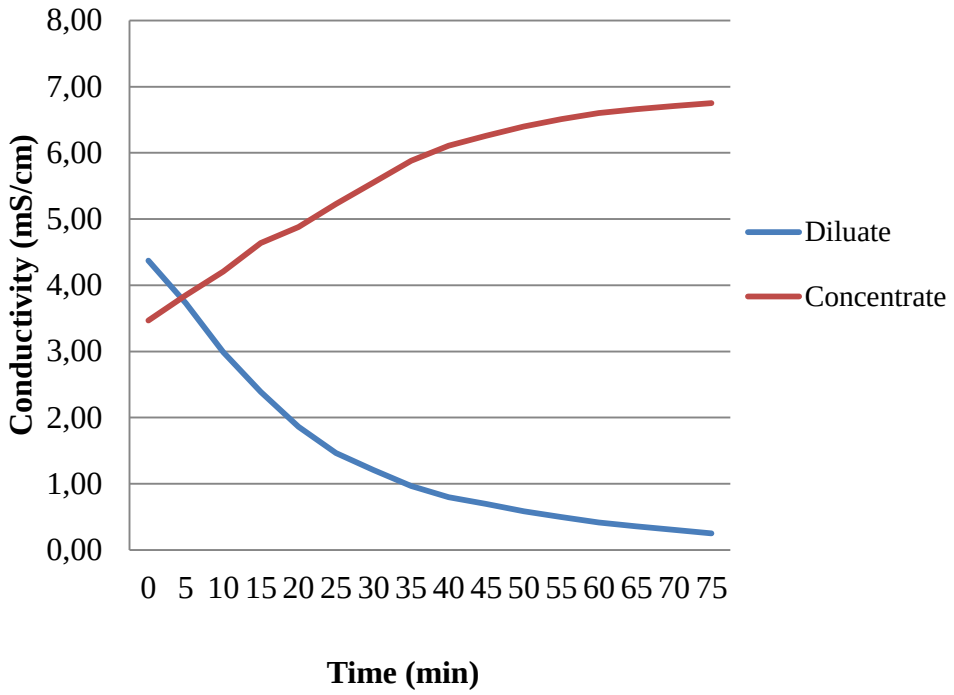

Figure 3.11 : Changes of conductivity of retentate during ED at positive current.

75 minutes is requires for 95,22% desalination of retentate at positive current.

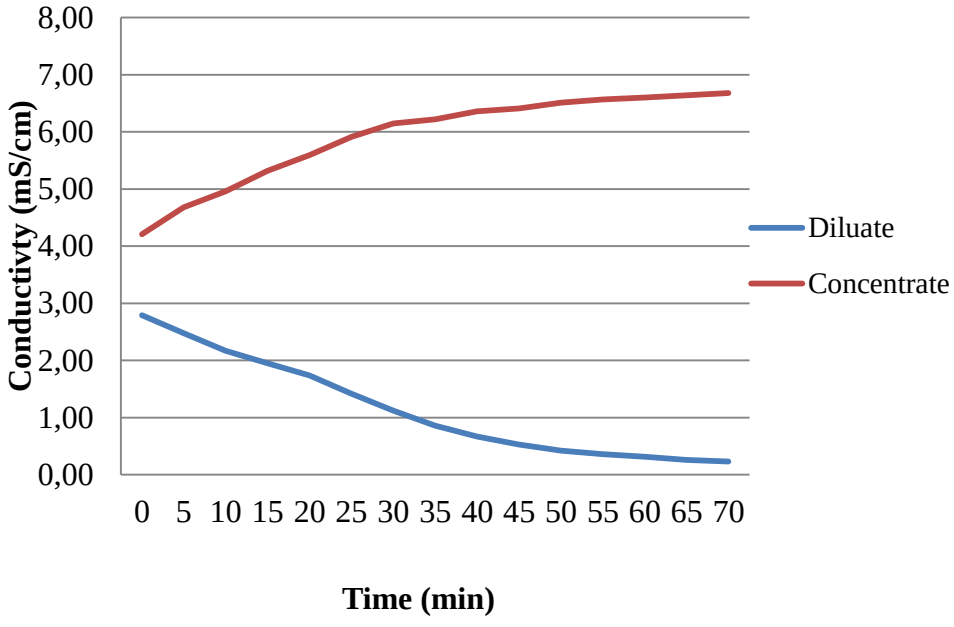

Figure 3.12 : Changes of conductivity of retentate during ED at negative current.

70 minutes is requires for 95,45% desalination of retentate at negative current.

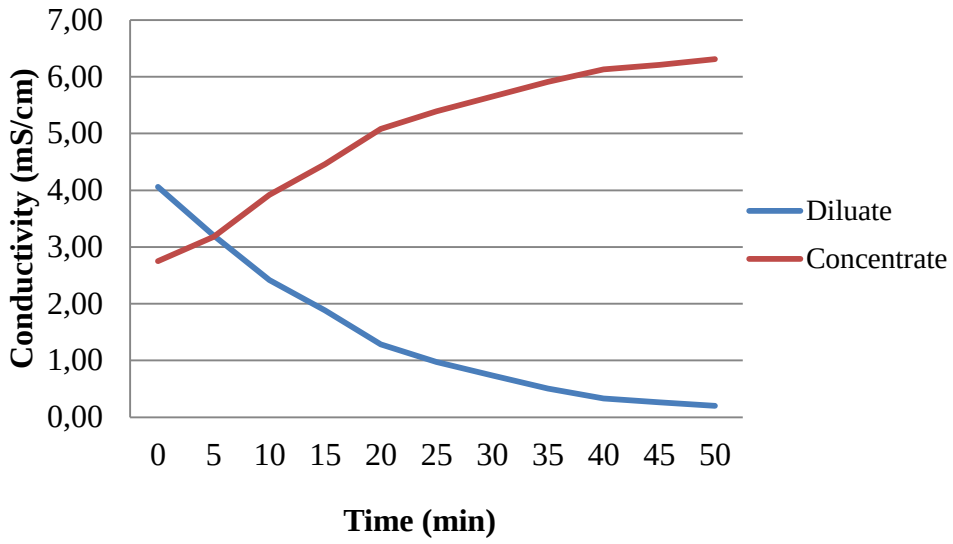

Figure 3.13 : Changes of conductivity of permeate during ED at positive current.

50 minutes is requires for 95,99% desalination of permeate at positive current.

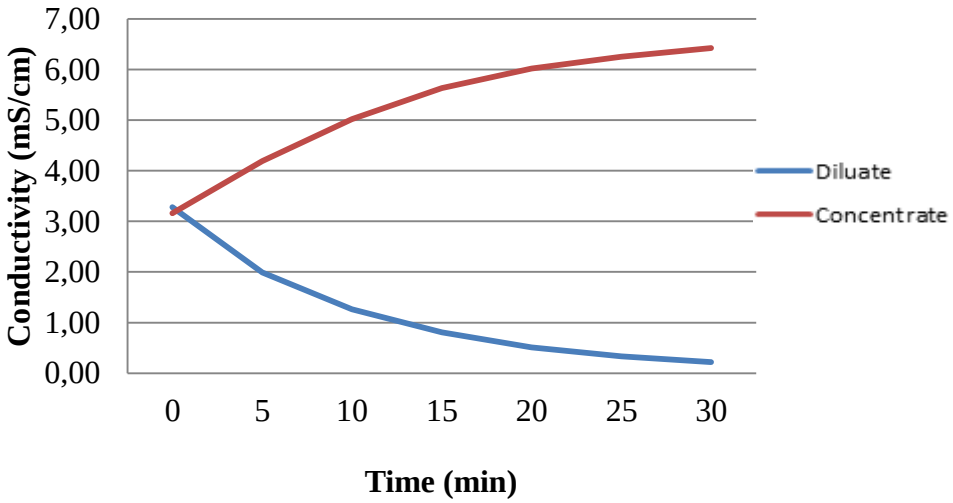

Figure 3.14 : Changes of conductivity of permeate during ED at negative current.

30 minutes is requires for 95,57% desalination of permeate at negative current.

It can be seen from Figure 3.3 to 3.14; ED experiment takes shorter time at negative current than positive ones.

Additionally, it is seen the higher the whey percentage is the longer the desalination processes occur. While 3,5 % of whey requires 40 minutes for desalination, 14 % of whey is needed 75 minutes at positive current.

For the ultrafiltrated samples, permeate takes shorter time in ED according to retentate; because of the low protein content.

At 2010, Lenka Diblíková, Ladislav Čurda and Karolína Homolová were examined ED is an effective way to demineralize the solutions of sweet whey and whey with 1% w/w of NaCl added. They inoculated pasteurized skimmed cow's milk with a starter culture (4% w/w) and after cooling, they added 2 mL of $CaCl_2$ and 7 mL of liquid rennet Laktochym (1:5,000, Milcom a.s., Praha, Czech Republic). After coagulation, cheese grains were removed from solution and microfiltration is applied to liquid whey by using filtration unit ARNO 700 (Mikropur a.s., Hradec Králové, Czech Republic) with ceramic membranes (TAMI-Industries, Hermsdorf, Germany). After microfiltration, a part of whey solution was salted with a NaCl as 1% w/w. All of the salted and non-salted solutions were ED by using a lab-scale ED unit ED-Z mini MemBrain s.r.o. (Strá z pod Ralskem, Czech Republic) and they were tested for dry matter, protein, ash, freezing point, pH and conductivity analysis. In addition the drop of main whey cations (K+, Na+, Ca2+, Mg2+) was measured by capillary electrophoresis PrinCE-C750 (Prince Technologies B.V., Emmen, The Netherlands).

According to ED's result, it is seen that desalination of normal whey took 50 min where salted whey took 65 min. Whey with salted and non-salted solutions can be desalted to a 95% degree successfully. In ED process, ash content, dry matter and the freezing point are decreased cause of the migration of ions to the concentrate. As a result of ion transport, conductivity of normal way was increased to 16.26 mS/cm and for salted whey was increased to 33.60 mS/cm. Additionally ED pH values decreased due to some cations were replaced by H^+ ions.

It can be summarize ED was observed to be an economic, time and energy saving method to achieve demineralization of whey effectively. ED process has a great potential in the future especially in dairy industry and should be investigated for further operations.

Michael Greiter and his collegues were aim to compare the ED process with IE resins in 2002. For this purpose, in a technical plant they were assessed the cumulative energy usage including production of the regeneration agents for the IEs and treatment of wastewater. In the plan for a feed solution, $45m^3$ nanofiltered, three times concentrated and desalted whey is used for each day.

In IE whey can be desalted as 99% where in ED 90% only. To compare enery demand of two process, it was seen in IE 0.15 kWh for pumping, 25.33 kWh for the

production of the regenerants, and 9.75 kWh for the reduction of the organic charge/m^3 whey; for the ED process it was 4.2 kWh for pumping, 5.38 kWh for the electric current through the ED cells, and 3.16 kWh for the reduction of the organic charge/m^3 whey.

According to process yields, for IE it was 3.7 m^3 wastewater including 36.3 kg ash and an organic charge of 26 kg chemical oxygen demand (COD)/m^3 whey however in ED, it was 1.25 m^3 for wastewater with 8.1 kg ash and an organic charge of 8.4 kg COD/m3 whey. As a result of these data it can be said that IE not only generated 4–4.5 times more salt than ED but also generated 3 times more wastewater. To sum, comparing demineralization of whey with ED and IE process in a technical plant, it was seen that ED can be a good technique not only economic point of view but also environment issues which is very important especially for European Union.

On another artical, according to Lenka Diblíková and her colleagues Ladislav Čurda and Jan Kinčl's research at 2013, they investigated the performance of ED using a lab-scale ED unit for demineralization process by preparing ten model solutions. They were prepared solutions with a fresh whey obtained from a local dairy (Moravia Lacto a.s., Jihlava, Czech Republic) at 7% (w/w) ratio and also reconstituted whey solutions were prepared by using distilled water at a 7, 14 and 21 % (w/w) ratio. Raw whey material microfiltrated on a filtration unit ARNO 700 (Mikropur a.s., Hradec Králové, Czech Republic) with ceramic membranes (TAMI-Industries, Hermsdorf, Germany).

The utilization of cheese whey in food products is restricted by its high salt content; demineralization should be applied before any other treatment. Owning to fact that they were also aim to test the performance of demineralization process of highly salted whey by using ED, sodium chloride, which is the most common salt type in cheese industry, was added to solutions (7%, w/w, fresh and reconstituted whey) in the range of 1,2 and 3% (w/w). All of the prepared ten solutions were ED by using a lab-scale ED unit ED-Z mini MemBrain s.r.o. (Stráz pod Ralskem, Czech Republic) and they were tested for dry matter, protein, ash, freezing point, pH and conductivity analysis. Besides that main whey cations (K^+, Na^+, Ca^{2+}, Mg^{2+}) was measured by capillary electrophoresis PrinCE-C750 (Prince Technologies B.V., Emmen, The Netherlands).

As the results of ED process investigated, it can be seen that in all 10 prepared solutions, the overall salt content decreased to 90-99%. K^+ and Na^+ were the fastest removed ions in all solutions and they can be removed as 83-100%. However Ca^{2+} and Mg^{2+}content decreased by 61-96%. From the all it can be seen mineral salts are successfully removed from whey even though they are concentrated and extremely salted.

4. CONCLUSIONS AND RECOMMENDATIONS

ED is applied at negative and positive polarity for four different whey concentrations as 3.5, 7, 10 and 14 %. Before ED, whey solutions are sampled and tested for ash, protein and dry matter analysis. As a result of these test it can be said that the higher the whey percentage is the more dry matter, ash and protein content is obtained.

ED is applied at negative and positive polarity for four different whey concentrations as 3.5, 7, 10 and 14 %. Before ED, whey solutions are sampled and tested for ash, protein and dry matter analysis. As a result of these test it can be said that the higher the whey percentage is the more dry matter, ash and protein content is obtained.

Before ED, UF is applied at 7% whey concentration. It is aimed to test permeate and retentate solution in ED which are obtained after UF process. Before ED process, permeate and retentate solution are sampled and tested for ash, protein and dry matter analysis. As a result of these test it is seen as retentate has higher in dry matter, ash and particularly in protein contents than permeate due to high salt concentration. In addition, permeate and retentate samples have lower ash and dry matter content but higher in protein content than other ED's samples. However dry matter, ash and protein result of whey after ED is lower than the whey before ED. As a conclusion, it is shows that ED is applied successfully and desired ash and dry matter contents are obtained.

Together with 3.5, 7, 10 and 14 % of whey and retentate, permeate solutions ED is conducted. It can be seen for all samples ED experiment takes shorter time at negative current than positive ones. Additionally, it is seen the higher the whey percentage is the longer the desalination processes occur. While 3,5 % of whey requires 40 minutes for desalination, 14 % of whey is needed 75 minutes at positive current. For the ultrafiltrated samples, in ED permeate takes shorter time in ED according to retentate; because of the low protein content.

For statistical analyses of ED process, calibration curves are drawn for 7 % of whey in order to calculate variables of ED process in macro excel document. From feed to 12.5, 25, 50, 75 and 95 %, desalinated whey is collected in order to check the change in dry matter, ash and protein content. It can be seen in from 7% of whey to 95% desalinated whey, conductivity, ash content and dry matter is decreasing since salt concentration is decreasing.

ED results show that the higher the whey percentage is the more J_{avg} is obtained because of the higher mineral content from 3,5 to 14% of whey. Furthermore, for all of the samples, flux values are higher in negative current than positive one since at negative current, ED works faster, and it increases the number of ions passing through membrane. For ultrafiltrated samples, permeate has the highest J_{avg} between permeate and 7 % of whey solution due to its high salt concentration. Besides, retentate has the lowest value because of the UF process it has lower salt content.

C_F is decreasing when the percentage of whey increasing. The more the salt concentration is the membrane fouling and scaling is happened which decreasing capacity of ED. C_F is higher in permeate since it can be processes faster than retentate. As a result of high protein content of retentate, the rate of membrane fouling and scaling is more than retentate and 7% of whey solution.

C_{D_DM} is parallel to the whey percentage, it is increasing when the percentage of whey increasing since the dry matter content is higher from 3,5 to 14 % of whey. As well, all of the negative current's values have higher results. C_{D_DM} of permeate has higher value than retentate meanwhile the dry matter content is higher in permeate solution. Besides, negative current's values have higher results.

Subsequently Δm_{ASH} is contrast with whey percentage, from 3.5 to 14% of whey; ash content is increasing so Δm_{ASH} is decreasing. Δm_{ASH} value of permeate is lower than the retentate's value since Δm_{ASH} is contrast with salt concentration.

α is decreasing with increased whey percentage. When whey percentage is increasing also ED capacity is decreasing. Owing to this fact, transference of water with salt through membrane is also decreasing. α is decreasing with increased salt content. Ever since permeate has more salt than retentate, its ED capacity is lower than permeate. The more the salt content is it is harder to transfer the water with salt through membrane.

When whey percentage is increased required power for desalination also W is increased. As a result W is parallel to whey percentage. In addition, it can be seen that both of the ultrafiltrated samples' W values are small enough to be operated for food industry. W of retentate is higher than permeate since it is rich in protein which requires more energy for ED.

It can be concluded that as a first aim of this thesis, sweet dry whey is treated by ED with cation-anion exchange membranes and factors such as J_{avg}, C_F, C_{D_DM}, Δm_{ASH}, α and W which influence efficiency and process of ED of whey are calculated. As a second objective of this thesis, ED process of retentate and permeate solutions, which are obtained after UF process are evaluated. It is shown that retentate and permeate solution which are collected after UF, successfully applied in ED. Without encountering any problem, retentate and permeate solutions can be desalinated. According to their W values, it can be said that UF before ED can be effectively adapted to food industry and they are open to new research and development.

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APPENDICES

APPENDIX A: ED tables

APPENDIX A

Table A.1 : ED results of 3,5 % whey concentration at positive current.

						Mass of D (kg)		Mass of C (kg)	
Date	29.02.2016	Current	Stack		Before	0,9842		0,9836	
Experiment	3,5%W	POZ	Normal		After	0,9605		0,9878	
t [min]	U_total [V]	U_stack [V]	I [A]	κ_D @25°C [mS/cm]	κ_K @25°C [mS/cm]	T_D [°C]	T_K [°C]	pH_D [-]	pH_K [-]
0	16,00	13,22	0,38	3,08	5,09	20,80	20,80	7,00	2,46
5	16,00	13,33	0,27	1,72	4,77	21,20	21,20	6,77	2,62
10	16,00	13,88	0,20	1,09	4,95	21,70	21,70	6,49	2,78
15	16,00	13,80	0,15	0,79	5,13	21,80	21,80	6,18	2,91
20	16,00	13,70	0,11	0,65	5,29	21,90	22,00	5,95	3,01
25	16,00	14,20	0,09	0,45	5,31	22,30	22,20	5,54	3,17
30	16,00	14,14	0,07	0,36	5,40	22,30	22,40	5,26	3,28
35	16,00	14,40	0,05	0,25	5,45	22,50	22,60	4,93	3,50
40	16,00	14,30	0,05	0,19	5,62	22,70	22,80	4,72	3,63
45	16,00	13,83	0,03	0,14	5,70	22,80	23,00	4,55	3,79

Table A.2 : ED results of 3,5 % whey concentration at negative current.

						Mass of D (kg)		Mass of C (kg)	
Date	02.03.2016	Current	Stack		Before	0,9685		0,9471	
Experiment	3,5%W	NEG	Normal		After	0,9513		1,0014	
t [min]	U_total [V]	U_stack [V]	I [A]	κ_D @25°C [mS/cm]	κ_K @25°C [mS/cm]	T_D [°C]	T_K [°C]	pH_D [-]	pH_K [-]
0	16,00	13,00	0,35	2,90	4,02	21,40	21,30	6,95	2,55
5	16,00	13,20	0,26	1,82	3,37	21,80	21,90	6,83	2,82
10	16,00	14,30	0,26	1,18	3,40	22,20	22,20	6,62	3,13
15	16,00	13,80	0,24	0,84	3,42	22,30	22,40	6,26	3,37
20	16,00	14,20	0,12	0,59	3,54	22,70	22,70	5,91	3,65
25	16,00	14,30	0,09	0,47	3,66	22,70	22,80	5,66	3,80
30	16,00	14,40	0,08	0,34	3,79	23,00	23,00	5,35	4,02
35	16,00	14,00	0,07	0,26	3,91	23,10	23,30	5,08	4,18
40	16,00	14,20	0,05	0,19	3,99	23,30	23,40	4,84	4,31
45	16,00	14,30	0,05	0,15	4,03	23,60	23,60	4,68	4,38

Table A.3 : ED results of 7 % whey concentration at positive current.

						Mass of D (kg)		Mass of C (kg)	
Date	19.04.2016	Current	Stack		Before	0,9882		0,9657	
Experiment	7%W	POZ	Normal		After	0,9772		1,0325	
t [min]	U_total [V]	U_stack [V]	I [A]	κ_D @25°C [mS/cm]	κ_K @25°C [mS/cm]	T_D [°C]	T_K [°C]	pH_D [-]	pH_K [-]
0	16,00	12,80	0,55	4,80	3,27	22,50	22,40	6,46	2,74
5	16,00	12,80	0,51	3,99	3,83	22,80	22,70	6,44	2,80
10	16,00	12,80	0,44	3,12	4,73	23,20	23,10	6,38	2,86
15	16,00	12,80	0,34	2,23	5,64	23,60	23,60	6,25	2,91
20	16,00	12,80	0,26	1,62	6,29	23,90	23,90	6,07	2,96
25	16,00	12,80	0,20	1,21	6,68	24,20	24,20	5,82	3,02
30	16,00	12,80	0,15	0,89	6,94	24,50	24,50	5,52	3,13
35	16,00	12,90	0,12	0,71	7,06	24,60	24,70	5,30	3,22
40	16,00	12,90	0,10	0,57	7,13	24,80	24,80	5,08	3,34
45	16,00	13,30	0,09	0,41	7,18	25,00	25,00	4,89	3,47
50	16,00	13,50	0,07	0,35	7,21	25,20	25,20	4,68	3,63

Table A.4 : ED results of 7 % whey concentration at negative current.

						Mass of D (kg)		Mass of C (kg)	
Date	19.04.2016	Current	Stack		Before	0,9899		0,9558	
Experiment	7%W	NEG	Normal		After	0,9368		0,9499	
t [min]	U_total [V]	U_stack [V]	I [A]	κ_D @25°C [mS/cm]	κ_K @25°C [mS/cm]	T_D [°C]	T_K [°C]	pH_D [-]	pH_K [-]
0	16,00	13,30	0,43	5,00	2,31	23,30	23,30	6,98	2,44
5	16,00	13,40	0,42	4,03	2,40	23,90	23,90	6,95	2,87
10	16,00	13,40	0,41	3,03	3,28	24,30	24,30	6,91	3,64
15	16,00	13,50	0,36	2,20	4,24	24,60	24,60	6,82	4,09
20	16,00	13,60	0,28	1,57	5,03	24,90	24,90	6,67	4,35
25	16,00	13,70	0,22	1,15	5,58	25,20	25,20	6,47	4,53
30	16,00	13,70	0,18	0,87	5,97	25,30	25,30	6,25	4,70
35	16,00	13,80	0,15	0,66	6,29	25,50	25,50	5,98	4,83
40	16,00	13,80	0,12	0,53	6,48	25,70	25,70	5,79	4,94
45	16,00	13,90	0,10	0,42	6,63	25,80	25,80	5,61	5,05
50	16,00	13,90	0,09	0,33	6,76	25,90	25,90	5,41	5,16
55	16,00	13,90	0,07	0,25	6,85	26,00	26,00	5,30	5,24

Table A.5 : ED results of 10 % whey concentration at positive current.

						Mass of D (kg)		Mass of C (kg)	
Date	02.03.2016	Current	Stack		Before	1,0048		0,9821	
Experiment	10%W	POZ	Normal		After	0,9705		0,9804	
t [min]	U_total [V]	U_stack [V]	I [A]	κ_D @25°C [mS/cm]	κ_K @25°C [mS/cm]	T_D [°C]	T_K [°C]	pH_D [-]	pH_K [-]
0	16,00	13,20	0,60	5,05	5,35	21,20	21,10	6,70	1,97
5	16,00	11,70	0,44	3,53	5,54	22,00	22,00	6,68	2,14
10	16,00	12,30	0,45	2,50	5,84	22,30	22,20	6,66	2,31
15	16,00	13,10	0,34	2,03	5,97	22,50	22,40	6,63	2,46
20	16,00	12,70	0,34	1,44	6,27	22,70	21,60	6,58	2,62
25	16,00	12,72	0,26	0,98	6,59	23,10	22,90	6,47	2,99
30	16,00	12,34	0,23	0,80	6,75	23,20	23,10	6,34	3,31
35	16,00	12,60	0,17	0,65	7,00	23,30	23,30	6,23	3,56
40	16,00	12,50	0,13	0,46	7,18	23,60	23,40	6,10	3,79
45	16,00	13,10	0,13	0,35	7,34	23,70	23,70	5,92	4,00
50	16,00	13,30	0,12	0,26	7,67	23,80	23,70	5,81	4,12
55	16,00	13,20	0,10	0,22	7,93	23,90	23,80	5,65	4,27
60	16,00	13,70	0,09	0,18	8,12	24,00	24,00	5,51	4,38
65	16,00	14,00	0,08	0,15	8,31	24,10	24,00	5,39	4,46
70	16,00	13,70	0,07	0,10	8,39	24,10	24,20	5,26	4,54
75	16,00	13,90	0,07	0.05	8,43	24,20	24,20	5,17	4,62

Table A6 : ED results of 10 % whey concentration at negative current

						Mass of D (kg)		Mass of C (kg)	
Date	02.03.2016	Current	Stack		Before	1,0084		0,9495	
Experiment	10%W	NEG	Normal		After	0,9664		1,0001	
t [min]	U_total [V]	U_stack [V]	I [A]	κ_D @25°C [mS/cm]	κ_K @25°C [mS/cm]	T_D [°C]	T_K [°C]	pH_D [-]	pH_K [-]
0	16,00	13,30	0,52	6,61	4,21	21,00	20,90	6,75	2,52
5	16,00	13,30	0,51	5,44	3,69	21,70	21,80	6,75	2,86
10	16,00	13,60	0,50	4,29	4,77	22,30	22,90	6,75	3,46
15	16,00	13,50	0,49	2,82	5,79	22,90	23,00	6,66	4,29
20	16,00	13,60	0,33	2,48	6,21	23,00	23,10	6,64	4,44
25	16,00	13,50	0,31	2,08	6,72	23,20	23,30	6,58	4,59
30	16,00	13,30	0,24	1,55	7,35	23,60	23,70	6,44	4,75
35	16,00	13,30	0,19	1,23	7,83	23,80	23,90	6,29	4,91
40	16,00	13,40	0,15	0,87	8,39	24,10	24,20	5,98	5,13
45	16,00	13,20	0,10	0,55	8,68	2,44	24,50	5,51	5,38
50	16,00	13,30	0,08	0,48	8,87	24,50	24,50	5,41	5,44
55	16,00	13,40	0,08	0,38	8,99	24,70	24,80	5,23	5,57
60	16,00	13,30	0,07	0,33	9,00	24,70	24,90	5,16	5,63

Table A.7 : ED results of 14 % whey concentration at positive current.

						Mass of D (kg)		Mass of C (kg)	
Date	03.03.2016	Current	Stack		Before	0,9994		0,9296	
Experiment	14%W	POZ	Normal		After	0,9681		1,0321	
t [min]	U_total [V]	U_stack [V]	I [A]	κ_D @25°C [mS/cm]	κ_K @25°C [mS/cm]	T_D [°C]	T_K [°C]	pH_D [-]	pH_K [-]
0	16,00	12,60	0,47	8,15	4,27	21,70	21,40	6,74	2,18
5	16,00	12,40	0,47	7,46	3,45	21,90	22,00	6,75	2,45
10	16,00	12,50	0,47	6,66	3,84	22,30	22,30	6,75	3,13
15	16,00	12,50	0,48	5,73	4,57	22,50	22,50	6,75	3,76
20	16,00	12,90	0,46	4,50	5,37	22,90	22,90	6,72	4,35
25	16,00	12,70	0,41	3,75	6,42	23,20	23,20	6,69	4,56
30	16,00	12,90	0,38	3,13	7,49	23,40	23,40	6,67	4,72
35	16,00	12,70	0,30	2,52	8,22	23,50	23,60	6,61	4,88
40	16,00	12,90	0,27	2,12	8,94	23,80	23,70	6,55	5,00
45	16,00	12,90	0,22	1,77	9,40	23,90	23,80	6,48	5,14
50	16,00	12,90	0,19	1,45	9,85	23,90	23,90	6,37	5,28
55	16,00	12,70	0,15	1,10	10,32	24,00	24,00	6,21	5,50
60	16,00	12,90	0,11	0,85	10,66	24,10	24,20	5,99	5,71
65	16,00	12,70	0,09	0,74	10,79	24,20	24,20	5,87	5,82
70	16,00	12,80	0,09	0,64	10,96	24,20	24,20	5,74	5,93
75	16,00	12,70	0,08	0,56	11,02	24,20	24,30	5,66	5,99
80	16,00	13,10	0,07	0,42	11,06	24,30	24,30	5,53	6,07

Table A.8 : ED results of 14 % whey concentration at negative current.

						Mass of D (kg)		Mass of C (kg)	
Date	03.03.2016	Current	Stack		Before	1,0234		0,9662	
Experiment	14%W	NEG	Normal		After	0,9855		1,0489	
t [min]	U_total [V]	U_stack [V]	I [A]	κ_D @25°C [mS/cm]	κ_K @25°C [mS/cm]	T_D [°C]	T_K [°C]	pH_D [-]	pH_K [-]
0	16,00	13,10	0,41	8,55	4,00	21,10	21,00	6,72	2,53
5	16,00	13,10	0,45	5,73	4,09	22,40	22,40	6,75	4,19
10	16,00	12,70	0,51	4,28	4,71	22,70	22,90	6,74	4,35
15	16,00	13,00	0,51	3,45	4,98	22,90	23,00	7,73	5,20
20	16,00	13,20	0,45	2,65	5,31	23,40	23,50	6,69	6,00
25	16,00	13,30	0,39	2,36	5,78	23,50	23,70	6,66	6,40
30	16,00	13,60	0,37	2,19	6,23	23,80	24,00	6,63	7,03
35	16,00	13,90	0,27	2,01	6,98	24,00	24,10	6,46	7,76
40	16,00	13,60	0,24	1,92	7,24	24,10	24,20	6,42	8,03
45	16,00	13,80	0,20	1,61	7,76	24,10	24,30	6,39	8,98
50	16,00	13,90	0,18	1,33	8,28	24,20	24,30	6,31	5,45
55	16	13,9	0,15	1,15	8,68	24,20	24,3	6,27	5,56
60	16	13,6	0,12	0,94	9,21	24,30	24,4	6,20	5,84
65	16	13,8	0,12	0,78	9,79	24,30	24,4	5,9	5,98
70	16	13,9	0,11	0,65	10,48	24,30	24,4	5,74	6,08
75	16	13,9	0,1	0,43	10,86	24,40	24,4	5,47	6,23

Table A.9 : ED results of retentate at positive current.

						Mass of D (kg)		Mass of C (kg)	
Date	08.03.2016	Current	Stack		Before	1,0183		0,9943	
Experiment	UF Retentate	POZ	Normal		After	0,9717		1,0051	
t [min]	U_total [V]	U_stack [V]	I [A]	κ_D @25°C [mS/cm]	κ_K @25°C [mS/cm]	T_D [°C]	T_K [°C]	pH_D [-]	pH_K [-]
0	16,00	13,20	0,43	5,23	4,20	20,00	20,40	7,00	2,17
5	16,00	13,10	0,37	4,37	3,47	21,20	21,30	6,98	2,35
10	16,00	13,30	0,36	3,73	3,85	21,70	21,70	6,96	2,70
15	16,00	13,01	0,32	2,99	4,21	22,30	22,40	6,91	3,26
20	16,00	12,90	0,29	2,39	4,64	22,60	22,60	6,87	3,58
25	16,00	13,20	0,22	1,86	4,88	23,30	23,20	6,69	4,14
30	16,00	13,30	0,17	1,46	5,23	23,60	23,70	6,60	4,29
35	16,00	13,20	0,15	1,21	5,55	23,90	23,80	6,47	4,42
40	16,00	13,40	0,10	0,97	5,88	24,20	24,20	6,31	4,56
45	16,00	13,00	0,10	0,80	6,11	24,40	24,40	6,15	4,67
50	16,00	13,20	0,09	0,70	6,26	24,60	24,70	6,03	4,75
55	16,00	12,90	0,07	0,59	6,40	24,80	24,70	5,88	4,84
60	16,00	13,10	0,07	0,50	6,51	25,00	24,90	5,75	4,92
65	16,00	13,20	0,06	0,42	6,60	25,10	25,00	5,61	5,00
70	16,00	13,30	0,05	0,36	6,66	25,20	25,30	5,50	5,06
75	16,00	13,20	0,05	0,31	6,71	25,30	25,20	5,41	5,11
80	16,00	13,20	0,04	0,25	6,75	25,30	25,20	5,28	5,18

Table A.10 : ED results of retentate at negative current.

						Mass of D (kg)		Mass of C (kg)	
Date	08.03.2016	Current	Stack		Before	1,0124		0,9708	
Experiment	UF Retentate	NEG	Normal		After	0,9694		0,9927	
t [min]	U_total [V]	U_stack [V]	I [A]	κ_D @25°C [mS/cm]	κ_K @25°C [mS/cm]	T_D [°C]	T_K [°C]	pH_D [-]	pH_K [-]
0	16,00	13,20	0,45	5,05	4,50	20,40	20,50	6,81	2,66
5	16,00	13,10	0,44	3,98	3,35	21,40	21,40	6,78	2,95
10	16,00	13,20	0,43	3,21	3,84	22,10	21,20	6,76	3,02
15	16,00	13,20	0,39	2,48	4,68	22,50	22,60	6,69	3,34
20	16,00	13,30	0,35	2,17	4,96	22,60	22,70	6,65	3,58
25	16,00	13,30	0,28	1,95	5,32	22,70	22,80	6,61	4,21
30	16,00	13,40	0,22	1,74	5,59	22,80	22,90	5,78	4,67
35	16,00	13,10	0,17	1,12	6,15	23,10	23,20	5,41	5,16
40	16,00	13,10	0,13	0,86	6,22	23,50	23,60	5,39	5,21
45	16,00	13,30	0,12	0,67	6,36	23,80	23,90	5,28	5,29
50	16,00	13,50	0,10	0,53	6,41	24,20	24,30	5,22	5,34
55	16,00	13,4	0,1	0,42	6,51	24,40	24,5	5,14	5,41
60	16,00	13,1	0,09	0,36	6,57	24,60	24,7	4,98	5,47
65	16,00	13,2	0,08	0,32	6,60	24,70	24,8	4,93	5,5
70	16,00	13,4	0,07	0,26	6,64	24,80	24,9	4,87	5,55
75	16,00	13	0,06	0,23	6,68	24,90	25	4,76	5,61

Table A.11 : ED results of permeate at positive current.

						Mass of D (kg)		Mass of C (kg)	
Date	08.03.2016	Current	Stack		Before	0,9993		0,9527	
Experiment	UF Permeate	POZ	Normal		After	1,0146		1,0741	
t [min]	U_total [V]	U_stack [V]	I [A]	κ_D @25°C [mS/cm]	κ_K @25°C [mS/cm]	T_D [°C]	T_K [°C]	pH_D [-]	pH_K [-]
0	16,00	12,29	0,40	4,99	3,38	17,20	17,40	7,15	2,20
5	16,00	12,27	0,34	4,06	2,75	18,90	18,80	7,17	2,87
10	16,00	12,13	0,32	3,20	3,18	19,70	19,70	7,15	3,72
15	16,00	11,44	0,29	2,42	3,92	20,30	20,30	7,12	4,41
20	16,00	11,63	0,26	1,88	4,46	20,80	20,80	7,07	4,75
25	16,00	12,90	0,20	1,29	5,08	21,50	21,50	6,96	5,12
30	16,00	12,74	0,15	0,98	5,39	21,80	21,80	6,87	5,38
35	16,00	12,71	0,14	0,74	5,65	22,20	22,20	6,75	5,60
40	16,00	12,70	0,12	0,51	5,91	22,40	22,40	6,48	5,88
45	16,00	12,90	0,08	0,33	6,13	22,90	22,90	6,10	6,13
50	16,00	13,03	0,07	0,26	6,21	23,00	23,00	5,95	6,24
55	16,00	13,21	0,06	0,20	6,31	23,10	23,10	5,74	6,33

Table A.12 : ED results of permeate at negative current.

						Mass of D (kg)		Mass of C (kg)	
Date	08.03.2016	Current	Stack		Before	1,0005		0,9953	
Experiment	UF Permeate	NEG	Normal		After	0,9630		1,0035	
t [min]	U_total [V]	U_stack [V]	I [A]	κ_D @25°C [mS/cm]	κ_K @25°C [mS/cm]	T_D [°C]	T_K [°C]	pH_D [-]	pH_K [-]
0	16,00	13,30	0,45	4,97	4,14	19,20	20,00	6,85	2,64
5	16,00	13,10	0,45	3,28	3,16	21,00	20,90	6,83	3,41
10	16,00	13,20	0,41	1,99	4,19	21,70	21,80	6,77	4,35
15	16,00	13,20	0,31	1,26	5,02	22,30	22,30	6,68	4,66
20	16,00	13,40	0,23	0,81	5,63	22,60	22,80	6,48	4,85
25	16,00	13,50	0,18	0,51	6,02	22,90	23,00	6,16	5,00
30	16,00	13,40	0,12	0,34	6,25	23,20	23,40	5,81	5,10
35	16,00	13,20	0,10	0,22	6,42	23,60	23,70	5,41	5,20

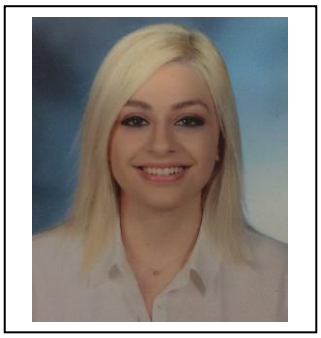
Table A.13 : ED results of model solution (Na_2SO_4) at positive current.

						Mass of D (kg)		Mass of C (kg)	
Date	18.04.2016	Current	Stack		Before	0,9600		0,9973	
Experiment	Model solution	POZ	Normal		After	0,8889		1,0392	
t [min]	U_total [V]	U_stack [V]	I [A]	κ_D @25°C [mS/cm]	κ_K @25°C [mS/cm]	T_D [°C]	T_K [°C]	pH_D [-]	pH_K [-]
0	16,00	12,80	1,21	11,44	12,48	24,00	24,00	2,53	2,91
5	16,00	12,60	1,07	9,31	14,30	24,30	24,30	2,70	2,83
10	16,00	12,79	0,89	6,65	16,40	24,70	24,70	2,95	2,77
15	16,00	12,90	0,73	4,71	17,80	24,80	24,80	3,18	2,74
20	16,00	12,90	0,58	3,21	18,87	24,80	24,80	3,37	2,73
25	16,00	12,90	0,39	1,91	19,68	24,80	24,80	3,48	2,72
30	16,00	13,20	0,26	1,03	20,20	24,80	24,80	3,63	2,72
35	16,00	13,30	0,14	0,56	20,50	24,80	24,80	3,68	2,72

Table A.14 : ED results of model solution (Na_2SO_4) at negative current.

						Mass of D (kg)		Mass of C (kg)	
Date	18.04.2016	Current	Stack		Before	0,9785		1,0316	
Experiment	Model solution	NEG	Normal		After	1,0316		0,9281	
t [min]	U_total [V]	U_stack [V]	I [A]	κ_D @25°C [mS/cm]	κ_K @25°C [mS/cm]	T_D [°C]	T_K [°C]	pH_D [-]	pH_K [-]
0	16,00	13,13	1,21	12,06	12,07	22,60	22,50	2,85	3,25
5	16,00	12,80	1,11	10,60	14,71	22,90	22,90	2,77	3,21
10	16,00	12,80	1,08	7,72	16,68	23,40	23,40	2,23	3,52
15	16,00	12,80	0,92	5,22	18,19	23,70	23,80	2,26	3,91
20	16,00	12,80	0,61	2,78	19,58	24,10	24,20	2,28	4,24
25	16,00	12,90	0,37	1,50	20,30	24,30	24,40	2,26	4,27
30	16,00	12,80	0,15	0,76	20,60	24,50	24,50	2,27	4,31
35	16,00	12,90	0,11	0,43	20,80	24,60	24,70	2,29	4,33

CURRICULUM VITAE



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