

Chapter 5

Recovery of High-Added Value Compounds from Dairy and Winery Agro-Food Industries Using Electrodialysis



X. Vecino, M. Reig, and J. L. Cortina

Abstract Electrodialysis (ED) is an established technology, which can separate ionic species applying an electrical potential. ED is widely used in water desalination for drinking water production, seawater concentration for table salt production, acid and base production from its corresponding inorganic or organic salt and the recovery of by-products from industrial effluents. However, ED is a promising and eco-friendly technology to treat agro-food streams or agricultural wastes and by-products, generated from agro-food industries, following the frame of circular economy on the management of these residues (reduce, reuse, recycle and reprocess) and in line with the industrial symbiosis principles.

This chapter presents an overview of the electro-membrane technology from the agro-industries context, as well as the application of this technology in the agro-food industries for the recovery of high-added value compounds. Among the agro-industries, dairy and winery sectors are studied in detail. These industries are selected due to their importance at Southern European and state (Spain) level as one

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of the largest industrial producers (in terms of tonnes of product). In the dairy industry, ED is mostly applied as a demineralization step of milk whey, however ED can be used for production of protein fractions, lactose recovery or lactic acid removal. Regarding winery sector, the main application of ED is in the tartaric acid stabilization of wine; but also, it can be used for tartaric acid and potassium recovery from vinasses. In this sense, the ED applications in the above-mentioned agro-industries have been summarized in this chapter.

Keywords Ion-exchange membrane · Monopolar membranes · Bipolar membranes · Selective membranes · Resource recovery · Agro-industries · Dairy · Winery

1 Introduction

1.1 Electrodialysis Principles and Applications

Electrodialysis (ED) is a membrane separation technique based on electrical potential as main driving force. For that, ion-exchange membranes (IXMs) are placed between two electrodes, forming an ED stack. ED is based on selective passage of ions through IXMs, depending on their functional group charge, due to Donnan repulsion (Doble 2016; Baker 2012). There are two IXM types: (i) cationic exchange membranes (CEMs) and (ii) anionic exchange membranes (AEMs). CEMs are negatively charged (containing $-\text{SO}_3^-$, $-\text{POO}_2^-$, or $-\text{COO}^-$ groups) and allow cations transference, while blocking anions passage through them. On the other hand, AEMs are positively charged (due to $-\text{NR}_4^+$, NR_3H^+ or NH_2^+ groups) and allow anions passage, although obstruct cations transport. In other words, IXMs permit contra-ions (opposite charge) passage, while hindering co-ions (same charge) transport through them (Strathmann 2010).

To conduct electrodialysis, AEMs and CEMs are placed alternatively between a cathode and an anode, separated by spacer gaskets. Thus, by a voltage applied between both electrodes, it is possible to separate ions from an aqueous solution and uncharged compounds, obtaining two new streams: (i) diluate and (ii) concentrate (see Fig. 5.1) (Al-Amshawee et al. 2020).

As can be seen in Fig. 5.1, membranes work as barrier for co-ions, while allowing contra-ions migration through them. Then, when feed solution, containing a solute electrolyte (MX), is introduced into the ED stack and voltage is applied between anode and cathode, ion migration occurs. Cations (M^+) from feed solution are attracted by the cathode, whereas anions (X^-) are attracted by the opposite electrode, the anode. Therefore, cations move towards the negatively charged electrode, crossing CEMs (negatively charged), but not AEMs (positively charged). At the same time, anions move towards the anode, passing through AEMs, but not CEMs. Hence, ionic species are removed from the feed solution producing a diluate

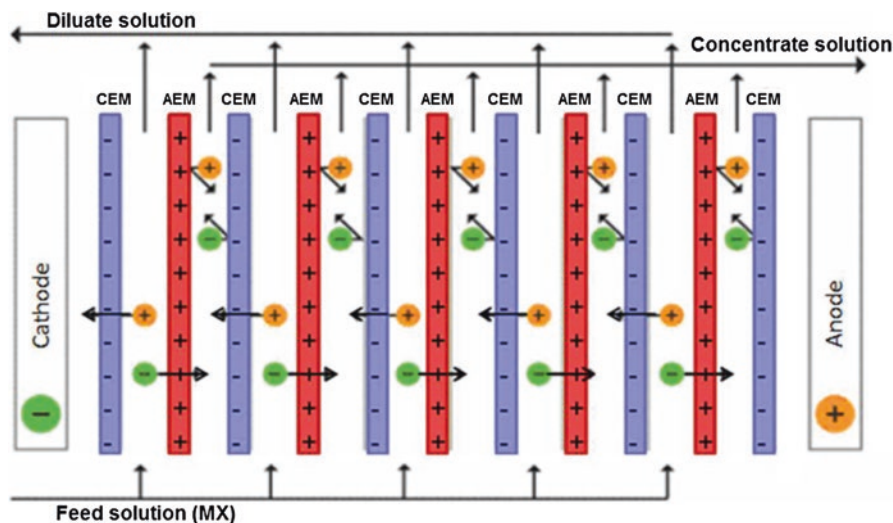


Fig. 5.1 ED membrane stack layout including the pairs of cation (CEMs) and anion exchange (AEMs) membranes and the electrodes at the extremes of the stack. (Adapted from (Arema 2017))

compartment, while a concentrate compartment is also obtained, recovering the ionic species from the feed solution. Moreover, an electrode rinse solution must circulate through both electrodes compartments, although it does not interact with the diluate and concentrate streams (Baker 2012; Strathmann 2010; Al-Amshawee et al. 2020). The electrode rinse solution keeps constant its concentration and composition over the ED procedure, since no ion transport occurred in this compartment, unless internal leaks appeared. However, solutions containing chloride ions are not recommended, due to chlorine gas formation in the electrodes compartment during the ED process. Indeed, Na_2SO_4 is one of the most widely used electrolyte (Campione et al. 2018; Reig 2016a).

As abovementioned, ion migration is the main transport phenomenon that takes place in an ED process. Nevertheless, undesired transport phenomena also take place, reducing the ED efficiency (Strathmann 2010; Pabby et al. 2009; Valdez Salas and Schorr Wiener 2012). Figure 5.2 represents all the mass transport phenomena that happen through IXMs during ED trials.

As shown in Fig. 5.2, three undesired phenomena occurred, apart from ion migration. In fact, ion migration implies electro-osmosis due to ion solvation. Then, water migration flux also occurred from the diluate to the concentrate compartment of the ED stack, diluting the final concentrated solution. Furthermore, ion diffusion and osmosis appeared after a period of ED operation, due to ion concentration gradient between both compartments. The former is the back diffusion or diffusion flux of ions from the concentrate to the diluate compartment, whereas the latter implies water transport from diluate to concentrate compartment, also known as osmosis flux.

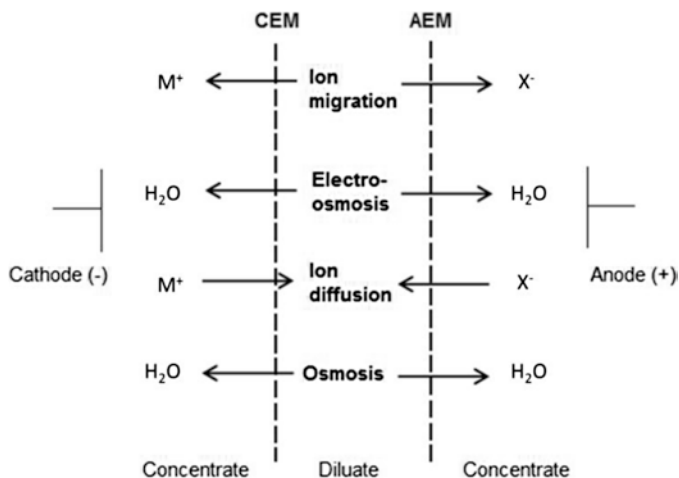


Fig. 5.2 Mass transport phenomena inside the ED stack: (i) ion migration, (ii) electro-osmosis, (iii) ion diffusion, and (iv) osmosis. (Adapted from (Reig 2016a))

An ED set-up usually consists of an ED stack, a power supply, pumps, piping and sensors. The main part is the ED stack, which is composed of IXMs, spacers and electrodes. As observed in Fig. 5.1, AEMs and CEMs are placed alternatively between electrodes. Each group of one AEM and one CEM is named cell pair. Besides, several IXMs configurations can be used for ED processes: CEM-AEM-CEM, AEM-CEM-AEM, cathode-CEM-AEM-anode or cathode-AEM-CEM-anode.

IXMs can be made by different materials, such as polyester, polyethylene, polyetheretherketone (PEEK) or polysulphone, among others (Al-Amshawee et al. 2020). Moreover, different membranes areas are used, depending on the application scale. Usually, IXMs of laboratory units have areas from 0.01 to 0.06 m², up to membrane areas of 1 m² at industrial scale (Von Gottberg 1998; Demircioglu et al. 2003; Tanaka 2015).

On the other hand, spacers can be also made from different materials, such as polypropylene/plexiglass, or polypropylene/silicone and they have the same area than the used IXMs. Besides, their thickness range is between 0.42 and 10 mm (Al-Amshawee et al. 2020).

Finally, electrodes from an ED stack can be made of titanium, titanium coated with ruthenium oxide, titanium plated with iridium, titanium coated with titanium and ruthenium oxides (70(%)RuO₂/30(%)TiO₂), platinum-plated iridium, steel 314 or graphite (Scarazzato et al. 2015; Szczygiełda and Prochaska 2017).

On the other hand, power supply is also an important device for ED tests. In fact, ED experiments do not start until current is applied between both electrodes, since ions are not attracted by the electrodes and they do not cross membranes without current applied. Thus, voltage and current should be enforced to the ED stack. However, depending on the number of cell pair, a maximum voltage could be

applied without damaging the membranes. For standard ED, 2.5 V should be considered as voltage drop in the electrodes compartment and increasing this value by 1 V for each cell pair. On the other hand, if other ED-based technologies are used, such as ED with bipolar membranes (BPMs) or selective ED, 2.5 V should be considered for electrode compartments voltage drop plus 1.5 V per each cell trio (combination of three different IXMs) plus 1 V per each bipolar membrane placed inside the stack (Ghyselbrecht et al. 2013).

There are many ED suppliers worldwide, although the main-know providers are Suez WTS, (formerly General Electric(GE) and before Ionics Inc.), Eurodia, and MEGA a.s (Valero et al. 2011).

ED technique appeared in the early 1950s for brackish water desalination applications. From then, several thousands of ED plants have been installed worldwide for water and wastewater desalination, since around 80–95% of the feed brackish water is recovered as clean water. However, a concentrate stream (5–20 times higher than the initial one) is also obtained by ED, named brine (Baker 2012). Hence, salt recovery from seawater appeared as another ED application, by valorising the ED brines (Campione et al. 2018; Reig et al. 2014). This application is widely used in Japan for table salt production.

The two already described applications are the most used by ED. Nevertheless, other applications appeared over the years, such as transition metals removal from electroplating rinse waters and hydrometallurgical processes (Zimmermann et al. 2020), energy production (Tian et al. 2020), lithium recovery (Li et al. 2019), organic acids production (Huang et al. 2007) or integrated with other technologies for increasing solutions concentration, for example in agricultural field (Vecino et al. 2020) or as part of a zero liquid discharge (ZLD) scheme (Muhammad Yaqub 2019). Finally, it is worth mentioning that ED has been also used in food industry applications (salt removal from cheese whey or soy, tannic acid removal from wine, citric acid removal from fruit juice, among others) (Baker 2012; Xu 2005).

The main drawback of conventional ED systems is membrane scaling and fouling due to colloid or insoluble salts precipitation on the IXMs. In order to prevent this issues, anti-scaling chemicals must be added to feed solution or pH adjustment must be done, together with regular membrane cleaning procedures (Strathmann 2010; Fidaleo and Moresi 2006).

Therefore, another operation mode appeared in the early 1970s: the electrodiagnosis reversal (EDR), designed by Ionics Inc. (USA). The main difference between conventional ED and EDR is that conventional ED systems operate unidirectional (diluate and concentrate compartments in the stack are fixed, since polarity of the electrodes is constant), whereas EDR systems are able to change – reverse – the polarity of the electrodes. Thus, by EDR the polarity of direct current applied to the electrodes is reversed time by time, exchanging also the diluate and the concentrate chambers. Thus, the precipitation accumulated on the membranes are flushed from the IXMs during the switching polarity periods, avoiding scaling or colloid obstructions on the IXMs surface. Nevertheless, the lifetime of the electrodes is reduced due to changes in polarity by EDR process (Baker 2012; Karimi and Ghassemi 2016; Murray 1995).

Apart from ED and EDR, there other ED-based technologies, that are gaining increasing attention, because of their promising possibilities of added-value compounds recovery and reuse. For instance, monovalent-selective membranes can be combined with AEMs and CEMs for separating monovalent from divalent ions, by electrodialysis (SED) (Zhang et al. 2012a), monovalent electrodialysis (mEDR) (Atkinson 2018), or electrodialysis methathesis (EDM) (Bond and Veerapaneni 2011). Besides, acid and bases can be produced from its corresponding salts by electrodialysis with bipolar membranes (EDBM) (Koter and Warszawski 2000; Huang and Xu 2006).

In fact, ion fractioning has a potential interest for several industries, such as wastewater treatment (Reig et al. 2016a, 2018, 2019; Tran et al. 2015) or recovery of added-value from agro-food residue (Barros et al. 2019; Zhang et al. 2011; Vecino et al. 2020). For that, standard IXMs are not enough, since they can only separate ions with different charge sign (positive or negative), but they do not distinguish between different ions charges (monovalent or divalent). However, monovalent-selective membranes can make differentiation in different charge ions transport. For instance, monovalent-selective cationic membranes (MVCs) allow monovalent cations passage, while blocking the divalent cations transfer when current is applied between both electrodes (Reig et al. 2018). On the other hand, monovalent-selective anionic membranes (MVAs) allow monovalent anions to cross them, while impeding the divalent anions passage (Zhang et al. 2012a). Therefore, by using different combinations of standard IXMs and selective IXMs (SED, mEDR or EDM), it is possible to achieve two differentiated streams, one rich in divalent

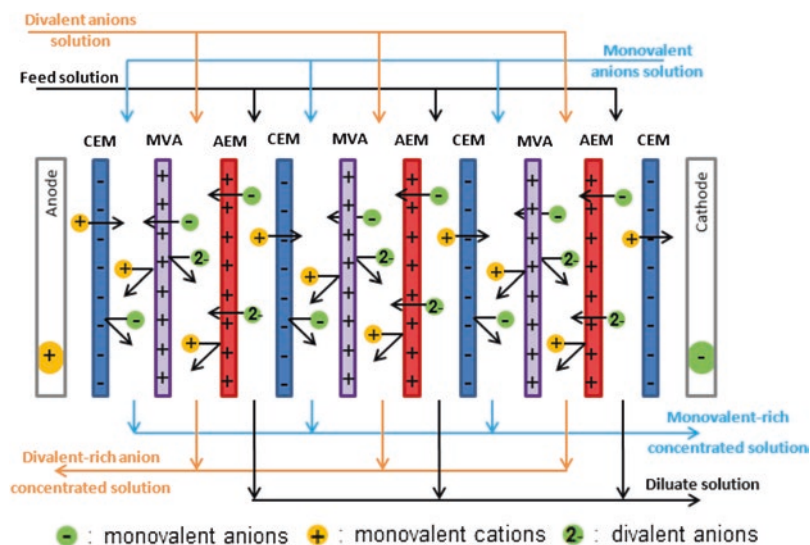


Fig. 5.3 SED membranes scheme for different charge anions separation, where AEMs and CEMs are conventional anionic and cationic ion-exchange membranes and MVAs are monovalent-selective anion exchange membranes. (Adapted from (Reig et al. 2016a))

ions and another monovalent ions-rich. For instance, Fig. 5.3 shows an example of SED for monovalent and divalent anions separation and concentration. Moreover, a diluate stream is also obtained.

Selective ED has been also applied in different fields, such as high salinity wastewater desalination (Zhang et al. 2012a), chloride and sulphate separation and concentration from industrial wastewater (Reig et al. 2016a), phosphate concentration from municipal wastewater (Tran et al. 2015), toxic metallic and non-metallic species removal from metallurgical process waters (Reig et al. 2018, 2019) or ZLD circuits (Bond and Veerapaneni 2011), among others.

For selective ED, transport phenomena are the same as when using ED. Nevertheless, other transport phenomena occurred due to membrane selectivity (using MVCs and MVAs), such as dielectric exclusion (Yaroshchuk 2000), size exclusion, charge differences and/or hydrophilicity differences between mono-charged and double-charged ions, or other membrane characteristics (Zhang et al. 2012a).

For EDBM applications, BPMs are combined with CEMs and AEMs for acid and base production. A bipolar membrane is formed by a cation selective layer (negatively charged) and an anion selective layer (positively charged), with a contact region between them. This layered structure with an interfacial layer permits water splitting when current is applied, producing protons and hydroxyl ions (Pourcelly 2002; Mukiibi and Feathers 2009; Koseoglu-Imer and Karagunduz 2018). Figure 5.4 shows an EDBM membrane disposition between two electrodes.

As can be seen in Fig. 5.4, when a feed electrolyte solution (MX) is introduced into the EDBM system and current is applied, water splitting is produced in the

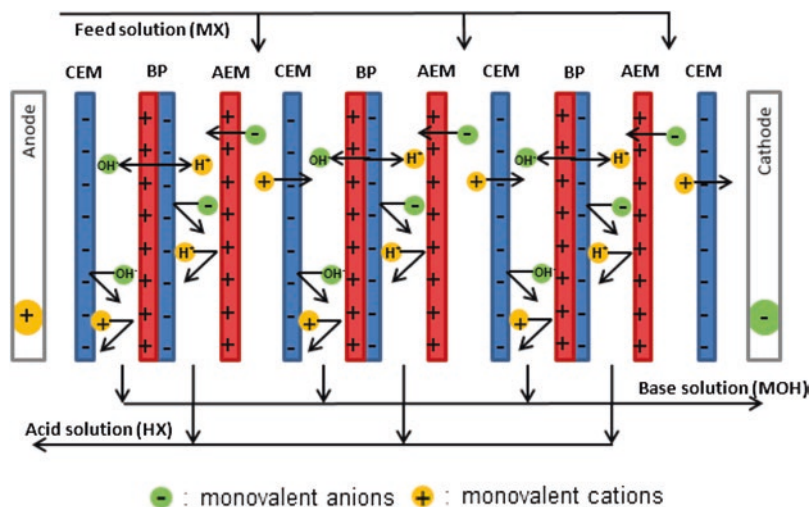


Fig. 5.4 EDBM membranes scheme for acid and base production, where AEMs and CEMs are conventional anionic and cationic ion-exchange membranes and BPMs are bipolar membranes. (Adapted from (Reig 2016b))

membrane interface (H^+ and OH^- are generated by the BPM and released through its cationic and anionic layer, respectively). Then, protons and cations moved towards the cathode, crossing the CEMs, but not the AEMs, whereas hydroxyl ions and anions move towards the anode, crossing AEMs, but not CEMs ones. Thus, cations (M^+) and OH^- are retained in the basic compartment, forming a base solution (MOH), while anions (X^-) and H^+ are retained in the acidic compartment, producing an acid solution (HX). Moreover, a diluate stream is obtained between CEMs and AEMs (not shown in Fig. 5.4) (Pourcelly 2002; Wiśniewski et al. 2004).

EDBM technique has been also used in different fields, although its main purpose is to produce acids and bases from its corresponding salt. Some applications are brines valorisation (Reig et al. 2016b, c), environmental protection (Ibáñez et al. 2004) or chemical and food processing (Fidaleo and Moresi 2006; Vecino et al. 2020). In fact, EDBM is an alternative technique for residues valorisation by chemicals production. For that reason, its implementation is growing in many industries, such as agro-food fields (Bazinet et al. 1998; Wang et al. 2018).

1.2 *An Overview of Agro-Food Industries*

Agriculture, food, and their combination as “agro-food”, are essential sectors for human communities. In fact, agro-food is not only an important driver of economic growth in several EU countries, but also a key thematic for new industrial value chains under the Horizon 2020 program as the Bio Based Industries (BBI) Action (European Commission 2017). Agricultural products comprise three main categories: (i) animals and animal products (such as live animals, meat, fish, crustaceans and aquatic invertebrates, dairy produce, eggs, honey, and other animal origin products); (ii) crop products; and (iii) foodstuffs (Castro-Muñoz et al. 2016). In Spain, the agricultural production (EU-28 total share, 2018) was mainly highlighted in permanent crops (35.1%) and fresh vegetables (23.3%), followed by different kinds of meat, such as pig (19.0%), poultry (10.8%) and bovine (8.4%), as well as cereals (8.3%); finally the low partakes were for raw milk and root crops with 4.9% and 2.9%, respectively (European Union 2019). On the other hand, the food and beverages industries were composed mainly by (i) bakery and farinaceous products (51.7%), proceeded by (ii) meat and meat products (12.2%) and beverages (10.2%), and (iii) prepared meals and dishes, food preparations and dietetic food, sugars, cocoa, tea and coffee (9.4%). Besides, dairy products and fruit and vegetables were 4.4% and 4.2%, respectively of the EU-28 companies. In addition, there was another group that involved vegetable and animal oils and fats, grain mill and starch products, prepared animal feed and fish and fish products (7.9%). Among the EU-28 total share, Spain had the 9.3% of the food and beverages enterprises and 8.2% of persons employed in this sector (European Union 2019). In consequence, the agro-food trade in Europe reached a value of 254€ billion in 2018. Thus, EU-28 achieved the first position as the largest global exporter and second biggest importer of

agro-food products, reaching a value of 138€ billion and 116€ billion, respectively. In regard with the EU-28 agro-food exports, the overview of products included wines and vermouth (the main category); spirits and liqueurs; infant food; food preparations; chocolate; and pasta and pastry. On the other hand, the tropical fruit, coffee and fresh or dried fruits; products that are mainly used for animal feed (e.g. oilcakes and soybeans); and products which are used as ingredient in further processing (e.g. palm oil) comprised the EU-28 agro-food imports (European Commission 2018a). Among the above-mentioned agro-industries, this chapter is focused on dairy and winery sectors. Both industries are important sectors in the EU, as well as in Spain. In fact, dairy industry represents the second major agro-food industry in Europe (Di Berardino 2019). In 2018, the EU produced 172.2 million tonnes of raw milk on farms, 97% of which was from cows (166.7 million tonnes); followed by milk from ewes (2.8 million tonnes), goats (2.3 million tonnes) and buffalos (0.3 million tonnes). The vast majority of raw milk was delivered to dairies (160 million tonnes); whereas only 12.2 million tonnes of milk was used on farms, either being consumed by the farmer and his/her family, sold directly to consumers, used as animals feed or processed directly. From 160 million tonnes of milk delivered to dairies, 156 million tonnes were milk from cows, being the rest a combination of ewes', goats' and buffalos' milk. From the milk used by the dairies, 0.4 and 0.2 million tonnes were raw milk imported and exported, respectively. On the other hand, with the milk used in dairy industry, 118.4 million tonnes of fresh and manufactured products can be obtained. Fresh products comprise drinking milk products (30.1 million tonnes) and other fresh products (15.7 million tonnes). From the 30.1 million tonnes of drinking milk, 12.6 million tonnes are skimmed milk and a further 17.3 million tonnes are whole milk. In regard with manufactured products, they can be divided mostly into whey (54.8 million tonnes), followed by cheese (10.3 million tonnes), milk powder (3.1 million tonnes), butter (2.4 million tonnes) and other manufactured products (2.0 million tonnes). In addition, Spain represented the 4.6% (eighth position), of the EU-28 total share from the 156.0 million tonnes, collection of cows' milk by dairies (European Union 2019).

On the other hand, the EU is a big player on the world's wine market. Indeed, between 2014 and 2018 it accounted for 65% of global production, 60% of consumption and 70% of exports, with 45% of the wine-growing areas in the world (European Union 2019). Particularly, 13% of the global area is occupied for Spain wine production (7.4 million of hectares), which are mainly destined for the production of wine grapes (table grapes or dried grapes) (IOV 2019), whereas Spain represented 26% of grapes for wines from the total harvested production of grapes in the EU-28 total (European Union 2019).

From the world production of grapes in 2018 (77.8 million of tons), the majority was used for wine grape (57%), preceded by table grape (36%) and dried grape (7%). In this context, the global production of wine was 292 million of hectolitres in 2018 (including sparkling and special wines and excluding juice and musts), being Spain the third largest wine producer (44.4 Mhl) just behind Italy and France (IOV 2019).

Apart from that, agro-food industries are responsible of a large part of global greenhouse gas (GHG) emissions. The agricultural sector produced 426,473 ktonnes of CO₂ eq of GHG (not including land use), about 10% of the EU's total GHG emissions (in 2015) (Eurostat 2019); whereas the entire food supply chain generated 26% of global GHG emissions (~13.7 billion metric tons of CO₂ eq). Additionally, food production caused ~32% of global terrestrial acidification and ~78% of eutrophication in 2018 (Poore and Nemecek 2018). Indeed, the implemented traditional agricultural practices result in high productivity but are strongly dependent on natural resources, such as water, nutrients (e.g. phosphorus), and fossil fuels. Actually, it has been estimated that between 30 and 50% of all food produced around the world is food losses or food wastes. In addition, agro-food industries, such as both dairy and winery industries, produce huge amounts of wastes. In the case of dairy industry, whey, dairy sludge and wastewaters (from processing, cleaning and sanitary steps) are the main wastes generated, having the latter the major environmental impact of the sector. Indeed, around 6–10 m³ of wastewater per m³ of processed milk is generated by dairy industries. Furthermore, processing of dairy products, from milk fermentation or by-products from the processing, can result in wastes, that could be used in the preparation of other dairy products, like whey concentrates from cheese whey. Commonly, dairy wastes (sludge and effluents) have high level of suspended solids and organic matter, high content of nutrients (nitrogen and phosphorous), high concentration of dissolved organic components (e.g. lactose, minerals, fat and whey protein), fatty acids, oil and greases. Furthermore, they can contain residues of the cleaning products used in utensils and equipment cleaning (e.g. detergents and biocides). However, the main by-product of dairy industry is whey, which is a source of food protein and produced during cheese and casein manufacturing. Due to the milk whey composition (lipids, carbohydrates, soluble vitamin, minerals as well as proteins), it has already been integrated for human consumption in many products (Ahmad et al. 2019; Reig et al. 2021).

Regarding to winery industry, during the winemaking process different kind of residues are produced. For instance, vineyard pruning wastes (also named trimming vine shoots) from harvest step, grape stalks from the de-stemmed of grapes, bagasse (also called grape marc or grape pomace) from pressing steps, wine lees obtained after different decanting steps, sediments obtained during clarification step, and wastewater generated from vinification lees (Devesa-Rey et al. 2011). In fact, Oliveira and Duarte (Oliveira and Duarte 2016) provided that from 1 ton of processed grape around 0.13 t of grape marc, 0.06 t of wine lees, 0.03 t of stalks and 1.65 m³ of wastewater are generated. Additionally, to recover ethanol and produce distilled beverages, the grape marc and wine lees must be sent to alcohol distilleries companies according to the European Council Regulation (EC) 479/2008 on the common organization of the wine market. During the distillation process, a liquid waste called vinasses is produced (Devesa-Rey et al. 2011; Pérez-Bibbins et al. 2015). Distilled vinasses are an environmental challenge if they are not treated properly, since they are acidic effluents with a large amount of organic matter and high solid content from dead yeast, grape pulp, skin and seeds. Vinasses are also composed by acids, sugars, phenols, proteins, lipids, as well as significant nutrients

(such as nitrogen, phosphorous, and potassium) (Devesa-Rey et al. 2011; Vlyssides et al. 2005).

In view of the aforementioned, a change for sustainable agricultural practices, in the agro-food system, is necessary to reduce the environmental impacts. The interdependency between infrastructure, production, distribution and environmental resources would allow the agro-food sustainability (Matthews 2017). Thus, the global interest about environmental protection in food, about several aspects such as climate change, resource depletion, human health risk or ecosystem damage are now considered as a priority by both society and governments in industrialised countries, as well as social and environmental organisations, businesses and academics (Scherer et al. 2020; Rico et al. 2020). In fact, the priority order in waste prevention and management legislation and policy should be as follows: (1) prevention; (2) preparing for re-use; (3) recycling; (4) other recovery, e.g. energy recovery; and (5) disposal, according to the Directive EU 2018/851 on waste (European Commission 2018b). Traditional direct disposal and treatment techniques, such as landfilling or incineration, are still vastly used but they are not suitable options; while re-use and recycling are the most favored choices (Capson-Tojo et al. 2016). The seventh EU Environmental Action Programme until 2020 (European Union 2013) identified waste prevention and management as one top priorities, being the main objective that the economic growth would not result in a disproportionate increase in waste generation. The huge impact that these residues have on the environment is not only due to the GHG emissions, related to climate change or to the loss of resources, but also to the complex mix of materials that compose them.

The households and processing sectors are the ones that generated the most food waste, being the 72% of EU-28 food waste (Stenmarck et al. 2016). For instance, the Spanish economy produced 132.1 million tonnes of waste in 2017, 2.3% more than the previous year. Among them, 3.2 million corresponded to hazardous waste (1.6% more than in 2016) and 128.9 million to non-hazardous waste (2.0% more), meaning the 2.4% and the 97.6% of the waste generated, respectively. By sectors, animal and vegetal wastes were generated mostly by agriculture, livestock, forestry and fishing (5.6 million tonnes). Regarding of the total generated waste to final waste treatment in Spain, 53.9% ended up in landfill, 38.9% was recycled, 3.7% was reused in backfilling operations and 3.5% was incinerated (Instituto Nacional de Estadística 2019). However, in line with the demands from the European Commission, industries should give a second thought to their current residues disposal practices. Instead of burning the wastes generated in their production chains or sending to landfill, it would be more interesting to develop strategies to transform them into valuable by-products. Because of that, the agricultural waste and by-products generated from the agro-food industries require a change from a linear to a circular economy concept that create innovative ways of valorisation to convert these waste materials into high-value products (Devesa-Rey et al. 2011; Donner et al. 2020). For that reason, apart from the classical 3Rs (reduce, reuse and recycle) of the waste management strategies, it has been introduced a fourth R, "Reprocess", which consists on the development of completely new processes to reuse the wastes as resources (Melikoglu et al. 2013).

Therefore, not only agro-food products must be considered added-value products, but also generated waste from the agro-food processing industry should be utilized to reduce the environmental impact and to increase the potential benefits for the industries (Castro-Muñoz et al. 2020). Thus, process technologies for conventional process improvement and for producing novel products have been developed for both purposes: (i) agro-food products treatment and (ii) agro-food waste recovery. Among them, membrane technology has been successfully employed in agro-food processing and valorisation (Daufin et al. 2001; Lipnizki 2010a, b; Castro-Muñoz and Fíla 2018; Castro-Muñoz et al. 2018, 2019, 2020). In fact, membrane technologies are well-known as clean technologies for agro-food treatment and they are economically feasible when either the waste management is high-cost or when a high-quality product is desired. For example, in the dairy industry, membrane technologies have been implemented in the milk and dairy processing chains, such as milk reception, cheese making, whey protein concentration, fractionation of protein hydrolysates, waste stream purification and effluents recycling and treatment (Di Berardino 2019; Daufin et al. 2001; Tavares and Malcata 2016; Reig et al. 2021). It is worth mentioning that the innovative application of membrane technology in the dairy industry was the conversion of whey into refined proteins for commercial use by ultrafiltration process (UF). In this sense, the top pressure-driven membrane processes in the dairy industry are microfiltration (MF) and UF, followed by nanofiltration (NF) and reverse osmosis (RO) (Lipnizki 2010a, b; Conidi et al. 2020). Regarding electro-membrane technologies, ED is commonly applied in the demineralization of whey (Daufin et al. 2001; Vecino et al. 2020).

On the other hand, must correction by RO (in terms of sugar content) was the first potential application in the wine production and lately for alcohol reduction in wine; then MF was used for clarification of wine after fermentation; the rejuvenation/lifting of old wine has been carried out with RO and DF (diafiltration); pervaporation (PV) has been used for recovery of wine aromas (Castro-Muñoz 2019); and the well-known application of ED in the wine industry is as a stabilizing stage in the tartaric precipitation in wines (Daufin et al. 2001; Lipnizki 2010a, b; Vecino et al. 2020).

2 Applications for Agro-Food Sectors

The agro-food industry encompasses diverse and complex activities whose challenge includes a wide range of processes and operations as food activities from the agricultural to our table. However, as aforementioned, agro-industries also produced huge amount of wastes. Thus, ED is a promising and eco-friendly technology to treat agro-food products streams, as well as agricultural wastes and by-products, generated from agro-food industries, following the frame of circular economy on the management of these residues and in line with the industrial symbiosis. Therefore, as represented in Fig. 5.5, the following chapter sections cover an overview of the application of ED technology in agro-food products treatment as well as in the recovery of by-products/wastes generated from two of the major agro-food

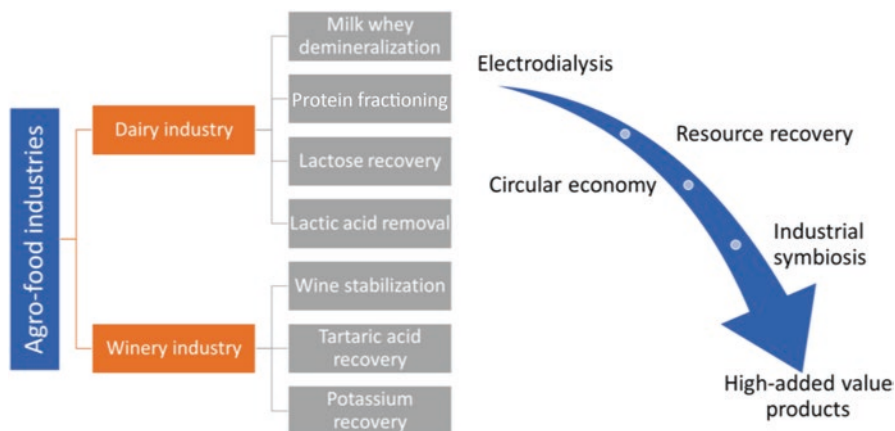


Fig. 5.5 Electrodiagnosis technology for high-added value compounds recovery from agro-food industries – chapter overview

industries: dairy and wine. In this case, sections were divided to show in detail the recovery of high-added value compounds by ED from the above-mentioned agro-food industries.

2.1 Dairy Industry

The major by-product from dairy industry, mainly of cheese and casein manufactures, is whey. Whey is a yellow-green liquid fraction drained from the curd, which can be easily acidified. Thus, two whey types can be distinguished: sweet whey ($\text{pH} \geq 5.6$) and acid whey ($\text{pH} \leq 4$) (Fidaleo and Moresi 2006; Wang et al. 2018). The main difference between cow milk and whey composition is lactic acid content in sweet whey (0.03–0.04% w/w) and acid whey (0.42–0.49% w/w), in comparison with cow milk, which does not contain lactic acid (Fidaleo and Moresi 2006). Furthermore, another whey type can be found, namely salty whey. This whey, which is salt-rich (50–60%), is produced in the cheese salting process and it is usually treated by UF. By this technique is possible to recover the whey proteins, although a high amount of minerals is retained in the UF permeate, making this stream not suitable neither for human, nor for animals feed and presenting environmental concerns if disposed directly, without any treatment. For this reason, salty whey is a waste by-product that increases disposal costs for dairy industries (Talebi et al. 2019). Thus, the main restriction for dairy by-products (mainly whey and ultrafiltration permeates) commercialization is the high minerals content in cow milk (3.37% w/w raw proteins, 3.9% w/w fat, among others). Moreover, due to whey biochemical oxygen demand (BOD_5) content (31–35 kg/m^3), it cannot be discharged into sewage. Furthermore, if whey is desalted, then it could be used in food production

(Fidaleo and Moresi 2006). For these reasons, the main ED application (with monopolar and bipolar membranes) in dairy industries is whey treatment (Tavares and Malcata 2016; Chen et al. 2018), mainly milk effluents demineralization, whey deacidification or alkalisation, as well as proteins and caseinates production (Bazinet 2005). However, due to its high lactic acid content, acid whey processing is blocked at industrial scale and it is one of the main challenge in dairy industries treatment (Kravtsov et al. 2020a; Dufton et al. 2020). Indeed, whey demineralization can be carried out by ED, achieving a demineralization rate from 50 to 95% (Daufin et al. 2001).

On the other hand, ED has many advantages for whey treatment, such biological substances demineralization, substance separation, minimal valuable components (proteins, lactose, among others) losses, no chemical addition, low energy consumption (< 1 kWh/kg ash removed, when demineralization degree is 50%–75% (Ahlgren 1972)) and high industrial capacity (easy operation equipment, modular design, processed at ambient temperature, automation, among others). Thus, ED is an economic and efficient method for whey processing (Tavares and Malcata 2016). In this case, although standard ED, with monopolar membranes, is the most common used technology in dairy industries, EDBM is also used for whey treatment (Kravtsov et al. 2020a, b; Dufton et al. 2018; Merkel et al. 2018) and proteins production (caseins and caseinates) (Mikhaylin et al. 2018; Masson et al. 2018). Table 5.1 summarises the main operational conditions, such as flow rates or electricity inputs into the EDBM stack, as well as the ion-exchange membranes used, the EDBM set-up, the main treated streams, the studied parameters and also the main results obtained in each study.

As can be seen in Table 5.1, Mikhaylin et al. (2018) and Masson et al. (2018) studied the effect of an EDBM system for proteins production from an ultrafiltrated milk fraction. In both cases, an EDBM set-up Model MP, from ElectroCell Systems AB Company was equipped with food grade Neosepta membranes (CMX-SB, AMX-SB and BP-1) with 100 cm² membrane area. Moreover, constant current of 2 A was applied in both cases and NaCl was used as additional stream. The former work (Mikhaylin et al. 2018) developed a life cycle assessment in order to study caseinate powder production from skim milk, whereas the latter (Masson et al. 2018) studied the advantages of UF, followed by EDBM for caseins separation, in comparison with conventional chemical acidification. The more outstanding result in both studies was NaOH production by EDBM, which could be used to solubilize the casein obtained during the EDBM process and be able produce caseinates. Thus, not only the skim milk stream was acidified during the process, but also caseinates were produced without the need of chemical addition, such as NaOH. Then, sodium caseinate powder could be produced on-site. Finally, it is worth to mention that by both methods (EDBM and conventional acidification with HCl) it was possible to produce whey. However, when using EDBM, the obtained whey was already demineralized and also some co-products were obtained: a lactose enriched solution and a Ca²⁺/Mg²⁺-rich solution.

On the other hand, Dufton et al. (2018) and Kravtsov et al. (2020b) studied acid whey demineralization and deacidification by EDBM (Table 5.1). The former work

Table 5.1 EDBM applications in dairy industries: review of applications and process performances

	Feed solutions		CEM AEM BPM	EDBM set-up Membranes	Operational conditions		Main results	Ref	
	Diluate stream (treated solution)	Concentrate stream I Concentrate stream II Electrode rinse			Flow rates (L/h)	Constant voltage or current Temperature Experimental time (min)			
Milk protein production	0.5 L – UF milk fraction (containing mainly lactose and minerals)	0.5 L – 2 g/L NaCl – 0.5 L – 20 g/L NaCl	Food grade Neosepta CMX-SB (Astom) Food grade Neosepta AMX-SB (Astom) Food grade Neosepta BP-1 (Astom)	Model MP (ElectroCell Systems AB Company) 100 cm ² ; 3CEM, 1 AEM, 1 BPM	Diluate and concentrate: 9 Electrode solution: 30	20 mA/cm ² – 100	Life cycle assessment (LCA) Caseinate powder production from skim milk	Skim milk acidification Casein production Co-products: Demineralized whey, lactose – rich and Ca/ Mg-rich solution NaOH to solubilize the casein	(Mikhaylin et al. 2018)
Production of calcium- and magnesium- enriched caseins and caseinates	0.5 L – UF pasteurized skim milk (Pur filter; Parmalat, Toronto, Canada).	0.5 L – 2 g/L NaCl – 0.7 L – 30 g/L NaCl	Food grade Neosepta CMX-SB (Astom) Food grade Neosepta AMX-SB (Astom) Food grade Neosepta BP-1 (Astom)	Model MP (ElectroCell Systems AB Company) 100 cm ² ; 3CEM, 1 AEM, 1 BPM	Diluate: varied Concentrate: 12 Electrode solution: 36	2 A – –	Separation of caseins by conventional chemical acidification vs UF + EDBM	pH decreases from 6.7 to 4.8 Caseins content: Ca = 0.22%; Mg = 0.011% Caseinates content: Ca = 0.18%; Mg = 0.010% Caseinates production by solubilizing Caseins with NaOH from EDBM	(Masson et al. 2018)

(continued)

Table 5.1 (continued)

Main purpose	Feed solutions			EDBM set-up Membranes	Operational conditions		Studied parameters	Main results	Ref
	Diluate stream (treated solution)	Concentrate stream I Concentrate stream II Electrode rinse	CEM AEM BPM		Flow rates (L/h)	Constant voltage or current Temperature Experimental time (min)			
Neutralization of the desalinated whey after ED (pH correction of milk whey)	1 kg – Nanofiltrated acidic milk whey (NFW) from curd producing (MADETA milk factory, Czech Republic) + ED treated stream (ED70 and ED90)	Conf 1: 0.5 kg – tap water – 0.25 kg – 10 g/L NaNO ₃ Conf 2: 0.5 kg – 13.56 mS/cm 0.5 kg – 7.47 mS/cm 0.25 kg – 10 g/L NaNO ₃	Food-grade CM-PP (MemBrain Ltd.) Food-grade AM-PP (MemBrain Ltd.) Heterogeneous food-grade BM-PP (MemBrain Ltd.)	Pilot unit EDR-Z/10–0.8 (MemBrain Ltd.) 64 cm ² ; BM-AM-BM (conf 1)-10 cell pairs; BM-AM-AM-BM (conf 2) – 20 AM, 11 BM	Diluate and concentrate: 58 Electrode solution: 50	20 V (conf 1); 25 V (conf 2) 15 ± 3 °C 150 (NFW); 120 (ED70); 30 (ED90)	Milk whey samples from ED demineralization Two EDBM configurations	EDBM capacity (kg/m-h): NFW = 8.2–14.8; ED70 = 7.6–10.4; ED90 = 31.0–49.3 pH increase: NFW = 4.8 to 5.7; ED70 = up to 6.3; ED90 = up to 6.7 Lactic acid concentration (g/L): NFW = 16.8 to 7.6; ED70 = 12.9–6.1; ED90 = 2.6–1.4 Wh/kg feed: NFW = 11.3–7.4; ED70 = 3.8–6.5; ED90 = 1.0–1.9	(Merkel et al. 2018)
Lactic acid deacidification and demineralization	2000 mL – AW from a dairy processing plant owned by Parmalat-Canada (Canada)	2000 mL – 5.5 g/L KOH 2000 mL – 5.5 g/L NaCl 2000 mL – 20 g/L Na ₂ SO ₄	Commercial food-grade CEM (Astom) Commercial food-grade AEM (Astom) BPM (Astom)	Laboratory-scale cell (Model MP, ElectroCell Systems AB Company) 100 cm ² ; BACBAC configuration	Diluate and concentrate (I and II): 240 Electrode solution: 240	100 A/m ² Around 20 °C 180	EDBM vs ED	Lactic acid deacidification: 44% AW demineralization: 67%	(Duffon et al. 2018)

Acid whey demineralization and deacidification	300 mL – AW from the cottage cheese production at the MKS dairy plant (Russia). Two types: 1) AW: 7% total solids 2) concentrated AW: 20% total solids	300 mL – Main water, conductivity 0.43–0.48 mS/cm – 300 mL – 20 g/L NaNO ₃	Heterogeneous RALEX CMH-PES (MEGA a.s.) Heterogeneous Ralax AMH-PES (MEGA a.s.) BM 2.0 (MEGA a.s.)	Laboratory-scale unit P EDR-Z (MEGA a.s.) 64 cm ² ; 21 membranes	Diluate and concentrate: 70 Electrode solution: 100	12.5 V demineralization; 25 V deacidification 22 ± 2 °C 65 (AW); 100 (concentrated AW)	AW and concentrated AW treatment	Degrees of AW demineralization: 70% (AW) and 90% (concentrated AW) Energy consumption: 76 kWh/t dry matter (70%) and 45 kWh/t dry matter (90%)	(Kravtsov et al. 2020b)
Alkalinization of acid whey in terms of lactic acid removal	800 mL – AW from a dairy processing plant owned by Parmalat-Canada (Canada)	800 mL – 0.5 mS/cm KCl – 1 L – 20 mS/cm NaNO ₃	CMX (Neosepta) AMX (Neosepta) BP-1 (Tokuyama corporation)	Laboratory-scale cell (Model MP, ElectroCell Systems AB Company) 100 cm ²	Diluate and concentrate: 24 Electrode solution: 54	10 V Room temperature (22 ± 2 °C) 140 (conf 1); 80 (conf 2)	Two cell configurations: 1) 2 CEM, 1 AEM, 1 BPM; 2) 2 AEM, 1 BPM	AW alkalinization: pH up to 6.5 Lactic acid removal: 26% (conf 1), 24% (conf 2) Demineralization: 34% (conf 1), 24% (conf 2) 28 kWh/kg (conf 1), 21 kWh/kg (conf 2)	(Kravtsov et al. 2020a)

AW: Acid whey

treated an acid whey from a dairy processing plant owned by Parmalat-Canada, whereas the second one treated an acid whey from the Cottage cheese production at the MKS dairy plant in Russia. In this case, different EDBM set-up, with different membranes (from Astom and MEGA, respectively) and different membrane areas (100 and 64 cm², respectively) were used. Moreover, the first study used constant current, whereas the latter kept voltage constant during the EDBM process. In both cases, the main purpose was achieved, with a demineralization degree of 67%, lactic acid deacidification of 44% (Dufton et al. 2018), and 70% to 90% acid whey demineralization with an energy consumption varying from 45 to 76 kWh/t dry matter, depending on the initial acid whey concentration (Kravtsov et al. 2020b).

Finally, Merkel et al. (2018) and Kravtsov et al. (2020a) studied EDBM for acid whey alkalisation in terms of lactic acid removal. The first work treated an acid whey from a dairy processing plant (Canada), whereas the second work used three different feed streams: a nanofiltrated acid whey (NFW, Czech Republic) from curd processing, and two desalinated ED streams (ED70 and ED90). Different membranes (Membrain (Merkel et al. 2018), Neosepta (CEMs and AEMs) and Tokuyama Corporation (BPMs) (Kravtsov et al. 2020a)) with different membrane areas (64 and 100 cm², respectively) were used. In both studies, constant voltage was applied and different EDBM configurations were tested. Results showed that acid whey pH increased up to 5.7 (when treating NFW), 6.3 (for ED70 feed stream), 6.7 (for ED90 input), and 6.5 (when using acid whey from Canada), whereas lactic acid was removed (from 46 to 55% (Merkel et al. 2018) and values around 25% (Kravtsov et al. 2020a)). Moreover, as reported in Table 5.1, energy consumption results depended on the initial feed stream and also EDBM configuration.

On the other hand, as above-mentioned, ED is widely used for whey treatment in dairy industry applications, mainly for demineralization purposes or lactic acid removal, as summarized in Table 5.2.

As summarized in Table 5.2, acid whey, sweet whey and salty whey have been processed by ED, mainly for lactic acid removal and demineralization purposes. Indeed, Chen et al. (2016) and Talebi et al. (2020) studied lactic acid reduction by ED when treating raw acid whey samples and ultrafiltrated fresh raw acid whey solutions, respectively, with a FuMA-Tech ED module, Neosepta IXMs and applying constant voltage. The aim of removing lactate or reducing its concentration from acid whey allows to recover proteins and lactose for sale, by processing the acid whey without lactic acid, as sweet whey. There were some differences between both studies: the former was carried out at lab scale (36 cm² active membrane area) and two working temperatures were studied (5 and 45 °C), whereas the latter was carried out at pilot scale (100 cm² of active membrane area) and ED was studied in combination with other membranes techniques, such as UF or NF. In this case, UF was used as a pre-treatment for protein removal and NF as a pre-treatment to achieve greater lactic acid removal levels. The first work showed that best results were obtained at high temperature (45 °C), achieving 80% lactic acid removal, 90% minerals removal and 3 times less ED experimental time than at 5 °C, and with an energy consumption of 0.014 kWh/kg acid whey when achieving 90% demineralization. On the other hand, the second work obtained the best results when

Table 5.2 Whey treatment by ED in dairy industries: review of applications and process performances

Main purpose	Feed solutions		CEM AEM	ED set-up Membranes	Operational conditions		Studied parameters	Main results	Ref
	Diluate stream (treated solution)	Concentrate stream Electrode rinse			Flow rates (L/h)	Constant voltage Temperature Experimental time (min)			
Removal of lactic acid from acid whey	1.2 L – raw acid whey samples from a dairy processing company (Australia)	1.2 L – 5.5 g/L NaCl 1.2 L – 20 g/L Na ₂ SO ₄	Neosepta CMB (Astom) Neosepta AHA (Astom)	FTED-40 module (FuMA-Tech GmbH) 36 cm ² ; 2 cell pairs	Diluate and concentrate: 7.5 Electrode solution: 60	7 V 5 and 45 °C 180	Temperature pH increase (4.6–6)	45 °C: more lactate ions Removal 45 °C: 3 times shorter ED than at 5 °C, to achieve the same lactate removal 80% lactate ions removal; 90% minerals removal 0.014 kWh/kg whey → 90% demineralization	(Chen et al. 2016)
Removal of lactic acid and minerals from acid whey	5 L – UF fresh raw acid whey (Tatura Milk Industries, Australia)	Tap water 20 g/L Na ₂ SO ₄	Neosepta CMB (Astom) Neosepta AHA (Astom)	FT-TS40 module (FuMA-Tech GmbH) 100 cm ² ; 10 cell pairs	Diluate, concentrate, electrode solution: 200	24 V 35 ± 4 °C 60	(i) UF + ED (ii) UF + NF + ED (iii) UF + dia-NF + ED	Best results: UF + dia-NF + ED maximum lactic acid removal: 88% lowest lactic acid/lactose ratio: 0.017 g/g 7.8 kWh/t feed	(Talebi et al. 2020)

(continued)

Table 5.2 (continued)

	Feed solutions		CEM AEM	ED set-up Membranes	Operational conditions		Studied parameters	Main results	Ref
	Diluate stream (treated solution)	Concentrate stream Electrode rinse			Flow rates (L/h)	Constant voltage Temperature Experimental time (min)			
Main purpose (i) Sweet whey demineralization (ii) Salty whey permeate demineralization (lactose and salt separation)	(i) 2 L – skimmed sweet whey from dairy companies (Australia) (ii) 4 L – UF salty whey permeate from dairy companies (Australia)	(i) 2 L- salty whey permeate or 0.1 M NaCl (ii) 1 L – salty whey permeate or 0.1 M NaCl	CMB and CIMS (Astom) AHA and ACD (Astom)	FTED-40 unit (FuMA-Tech GmbH) 36 cm ² ; 3 cell pairs	Diluate and concentrate: 30 Electrode solution: 60 (i) 44 ± 2 °C (ii) 26 ± 2 °C (i) 180 (ii) 480	Sweet demineralization using UF salty whey as concentrate stream Monovalent-selective IXM for salty whey demineralization	(i) 75% demineralization 7.4 kWh/t whey (ii) 6.5–22–33% demineralization at 5, 10 and 15 V 0.9–2.1–3.6 kWh/t NaCl at 5, 10 and 15 V Ca concentration (concentrate stream) half reduced with selective IXMs	(Talebi et al. 2019)	
Demineralization of whey	2.2 kg – Nanofiltrated acidic milk whey (NFW) from curd producing (MADETA milk factory, Czech Republic)	0.5 kg – Tap water 0.25 kg – 10 g/L NaNO ₃	Heterogeneous food-grade CM-PES (MemBrain Ltd.) Heterogeneous food-grade AM-PES (MemBrain Ltd.)	Pilot unit EDR-Z/10–0.8 (MemBrain Ltd.) 64 cm ² ; 10 cell pairs	Diluate and concentrate: 58 Electrode solution: 50 12 V 15 ± 3 °C 348 (ED70); 438 (ED90)	ED with 70% (ED70) and 90% (ED90) of demineralization	ED capacity (kg/m ² ·h): ED70 = 5.8; ED90 = 4.2 pH increased (4.5 to 5.0) Lactic acid concentration decreased (17 to 3 g/L) Wh/kg feed: ED70 = 5.7; ED90 = 10.0	(Merkel et al. 2018)	

Lactic acid deacidification and demineralization	2000 mL – AW from a dairy processing plant owned by Parmalat-Canada (Canada)	2000 mL – 5.5 g/L NaCl 2000 mL – 20 g/L Na ₂ SO ₄	Commercial food-grade CEM (Astom) Commercial food-grade AEM (Astom)	Laboratory-scale cell (model MP, ElectroCell systems AB company) 100 cm ² ; CACAC configuration	Diluate and concentrate: 240 Electrode solution: 240	100 A/m ² ~ 20 °C 180	ED vs EDBM	Lactic acid deacidification: 44% AW demineralization: 67%	(Dufion et al. 2018)
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AW: acid whey

combining UF, dia-NF and ED, since the dia-NF retentate was 3.5 times more concentrated than the UF permeate. Thus, it was possible to remove 88% lactic acid with an energy consumption of 7.8 kWh/t feed.

Talebi et al. (2019) had also studied the demineralization of sweet whey and salty whey permeate treatment from dairy companies by ED. The aim of this study was not only to recover the demineralize lactose-rich stream – which could be used for lactose powder production, but also to produce a concentrated salt solution – which could be used in the chlor-alkali industry. For sweet whey demineralization, ultrafiltrated salty whey was used as concentrate solution using a 36 cm² membrane area module from FuMA-Tech. In this case, standard IXMs from Astom were used at constant voltage to obtain 75% of demineralization with an energy consumption of 7.4 kWh/t whey. For salty whey processing, monovalent-selective IXMs from Astom were used. These membranes selectively separate monovalent ions, such as sodium and chloride ions from divalent ions, such as calcium. Then, it was possible to generate a pure salt concentrate stream and a lactose-rich stream, with nutritional content of calcium. Moreover, demineralization values from 6.5 to 33% were achieved at different constant voltages (5–15 V) with an energy consumption ranging from 0.9 to 3.6 kWh/t NaCl.

Finally, Merkel et al. (2018) and Dufton et al. (2018) studied acid whey demineralization by ED. The first work utilized MemBrain heterogenous food-grade IXMs to demineralize nanofiltrated acidic milk whey from curd producing by an EDR pilot with 64 cm² of membrane area at constant voltage. Results showed that the conductivity decreased (lactic acid concentration varied from 17 to 3 g/L), whereas the pH increased (from 4.5 to 5.0) over time. On the other hand, the second work was carried out to study lactic acid deacidification and acid whey demineralization by commercial food-grade membranes from Astom (100 cm² of active membrane area) and a laboratory ED cell from ElectroCell Systems. In this case, constant current density was applied obtaining lactic acid deacidification of 44% and acid whey demineralization of 67%.

Furthermore, ED has been studied and applied for other applications, such as skimmed milk demineralization, desalination of ultrafiltrated milk permeate, demineralization of nanofiltrated retentate when treating lactose-free milk production or preparation of low-lactose milk powder. These examples have been summarized in Table 5.3.

As can be seen in Table 5.3, to the best of our knowledge, Andrés et al. (1995) were the first proposing the use of selective IXMs for commercial skimmed milk demineralization. In this case, they compared the results when using (i) standard IXMs (from Stantech) and (ii) selective IXMs (from Tokuyama). Moreover, different stacks were used: (i) a 100 cm² laboratory unit with 10 cell pairs and (ii) a 1568 cm² semi-pilot scale unit with 4 cell pairs, both tested at constant voltage. Results showed slightly better demineralization percentages when using standard ED membranes (45% vs 42% when using selective ED). However, it was possible to obtain quicker monovalent ions removal, when using selective IXMs, achieving higher Ca/Na selectivity (1.6 vs 1.2). Finally, energy consumption was also lower when using the selective membranes (0.94 kWh/kg vs 1.2 kWh/kg).

Table 5.3 ED applications in the dairy industry: review of applications and process performances

Main purpose	Feed solutions		CEM AEM	ED set-up Membranes	Operational conditions		Studied parameters	Main results	Ref
	Diluate stream (treated solution)	Concentrate stream Electrode rinse			Flow rates (L/h)	Constant voltage Temperature Experimental time (min)			
Skimmed milk demineralization with (i) standard ED, (ii) selective ED	Commercial skimmed milk from Central Lechera Asturiana (Spain)	7 g/L Na ₂ SO ₄ 20 g/L Na ₂ SO ₄ (acidified to pH = 2 with H ₂ SO ₄)	(i) SC-1 (Stantech) (ii) Special grade Neosepta CMS (Tokuyama) (i) SA 1 (Stantech) (ii) Special grade Neosepta ACS (Tokuyama)	(i) Backpack laboratory unit (Stantech) (ii) Semi-pilot scale TS-2 IOP (Eurodia) (i) 100 cm ² ; 10 cell pairs (ii) 1568 cm ² ; 4 cell pairs	-	Constant voltage (not exceeding 20–25 mA/cm ²) 24 °C 60	Selective IXMs vs standard IXMs	Demineralization: 45% ED and 42% selective ED Quicker monovalent ions removal with selective ED Ca/Na: 1.57 selective ED and 1.22 ED Current efficiency: 77% selective ED; 63% ED ED: 0.94 kWh/kg selective ED: 1.2 kWh/g	(Andrés et al. 1995)

(continued)

Table 5.3 (continued)

Main purpose	Feed solutions		CEM AEM	ED set-up Membranes	Operational conditions		Studied parameters	Main results	Ref
	Diluate stream (treated solution)	Concentrate stream Electrode rinse			Flow rates (L/h)	Constant voltage Temperature Experimental time (min)			
Desalination of UF milk permeate	(i) UF permeate from skim milk from a dairy plant (Czech Republic) (ii) UF milk permeate, concentrated by RO (3 times)	3% HNO ₃ 10 g/L NaNO ₃	RALE X-CEM RALE X-AEM	P EDR- Z/10-0.8 (MemBrain s.r.o) 64 cm ² ; 10 cell pairs	Diluate and concentrate: 60 Electrode solution: 50	15 V 15 ± 2 °C (i) 65; (ii) 130	Feed sample: (i) UF milk permeate and (ii) concentrate UF milk permeate by RO	(i) 90% desalination by 65 min; 2.21 Wh/ kg (ii) 90% desalination by 130 min; 6.95 Wh/kg (i, ii) final products had similar compositions	(Shakhmo et al. 2019)
Demineralization of NF retentate (lactose-rich)	2 L – NF retentate (NFR) from a lactose- free milk production (Esbjerg, Denmark)	2 L – 2 g/L NaCl 0.25 L – 20 g/L Na ₂ SO ₄	CEM-PES (Membrain) AEM-PES (MemBrain)	P EDR- Z/10-0.8 (MemBrain) 64 cm ² ; 10 cell pairs	Diluate and concentrate: 50 Electrode solution: 60	5, 10, 15 V 25 °C 120	Applied voltage NFR concentration; undiluted, dilution ratios 1.5 and 2	Demineralization rate > 90% Higher demineralization efficiency at 15 V and higher dilution ratio	(Rasmussen et al. 2020)

Preparation of low-lactose milk powder ED: Remove and recover mineral salts from UF permeate	10 L – UF pasteurized milk (Hangzhou Meijian Co., Ltd.)	Ionized water Na_2SO_4	CEM-type II (FUJI film) AEM-type II (FUJI film)	MP type cell (Electro Cell Systems AB) 189 cm ² , 6 cell pairs	Diluate, concentrate, electrode solution: 40	15, 20, 25 V Room temperature 60	UF + ED + NF	Volume reduction: 1 L of concentrate from 10 L of feed More voltage → + salt rejection rate, – desalination time 98% desalination at 30 min	(Zhang et al. 2020)
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Furthermore, as observed in Table 5.3, the newest works (Rasmussen et al. (2020) and Zhang et al. (2020)) carried out during 2020, are focused in lactose-free/low-lactose dairy products, due to the growing lactose intolerance in society. The aim of the first work was to demineralize a nanofiltrated retentate from a lactose-free milk production in Denmark. In this case, the NF retentate, which was lactose-rich, was tested by an ED set-up from MemBrain and standard CEMs and AEMs from the same company, with an active membrane area of 64 cm². Several constant voltages were studied (5, 10 and 15 V) and also different dilution ratios of the NF retentate were tested. In all cases, demineralization percentages above 90% were achieved, without significant changes or lactose losses. Nevertheless, it was concluded that higher demineralization efficiencies were achieved at 15 V and higher dilution ratio of the ED feed.

On the other hand, Zhang et al. (2020) studied ED to remove and recover mineral salts from an ultrafiltrated permeate from pasteurized milk. The idea of the UF pre-treatment was to retain macromolecules (proteins and fats). Then, ED was used for mineral salt removal (desalting) and also as a concentration step of the UF permeate stream (salts were re-added into milk). Finally, NF was also added in the proposed treatment train for lactose recovery. Moreover, to close a circular scheme, NF permeate could be recirculated into the UF stage. For that, CEMs and AEMs from FUJI Film were used in an ED stack from Electro Cell Systems applying several constant voltages (15, 20, 25 V). Results showed a high-volume reduction (from 10 L of feed to 1 L of concentrate) and a maximum desalination percentage of 98% at 30 min of ED operation. Besides, it was demonstrated that higher applied voltage, implied more salt rejection rates and less desalination time. All in all, it can be indicated that the proposed membrane treatment train could be a promising scheme for low-lactose milk powder preparation and lactose recovery.

Finally, ED has been studied for other treatments in dairy industries, such as to separate added-value proteins (e.g. lactoferrin and immunoglobulins from other proteins of crude dairy streams (Wang et al. 2020)) or to increase the *Lactococcus lactis* NZ133 starter culture biomass production by lactate removal in dairy applications (Boonmee et al. 2007). In the former work, poly(vinyl) alcohol membranes were prepared to achieve a high selectivity between lactoferrin and other proteins. Moreover, a crosslinking agent was used to increase the membranes water resistance. Synthetic solutions were tested by ED, mimicking an ultrafiltrated milk, that represented the buffered salt mixture in a common dairy whey. In this case, an ED Gradipore Gradflow BF400 System (Memphasys Limited, Sydney) was used, with an active membrane area of 16 cm². All tanks (diluate, concentrate and electrode rinse) were filled with the same feed solution. Diluate and concentrate stream compartments used 10 mL of ultrafiltrated milk in a recirculation mode at 17 mL/min, whereas the electrode rinse compartment was filled with 1 L of feed solution, which was also recirculated, but at 3.4 L/min. Then, a constant voltage of 100 V was applied between ED electrodes. Results showed that it was possible to isolate large proteins, such as lactoferrin and immunoglobulins from dairy whey by ED. In this case, smaller proteins passed through the membrane, while larger proteins were retained (Wang et al. 2020). Finally, Boonmee et al. (2007) incorporated an ED

system into a batch fermentation grown on 80 g lactose/L. The ED set-up consisted on 3 cell pairs of CEMs and AEMs from BDH Chemicals (Australia), with a membrane area of 100 cm². In this case, 0.25 L of diluate (fermented stream) and initial concentrate solution (tap water) were circulated into the ED stack at 3.6 L/h. Moreover, 0.25 L of 0.1 M H₂SO₄ were used as electrode rinse solution, circulating at 2.85 L/h. Different experiments were carried out at constant voltage of 40 V, or at constant current. Results showed that it was possible to remove lactate ions, which are a growth inhibitory metabolic end-product. Nevertheless, it seems that ED is not the most adequate technology to be incorporated in a fermentation process due to its limitations in lactate ions removal and low increase of the biomass production of the dairy starter culture *Lactococcus lactis* NZ133. Indeed, lower starter culture concentrations were obtained, compared with the conventional fermentation process (Boonmee et al. 2007).

As a summary, ED, selective ED and EDBM have been used for several purposes in dairy industries, such as, whey and milk demineralization, production of protein fractions, lactose recovery or lactic acid removal (Himstedt and Hestekin 2011; Bazinet 2015; O'Mahony and Tuohy 2013; Hestekin et al. 2010).

2.2 Wine Industry

The main application of ED in wine industry is the tartaric acid stabilization in wines (Lasanta and Gómez 2012; Gonçalves et al. 2003; Gómez Benítez et al. 2003; Soares et al. 2009; Bories et al. 2011; Corti and Paladino 2016; Henriques et al. 2019). The wine instability is caused by some sediments of tartaric salts, potassium bitartrate (KHT) and less frequently calcium tartrate (CaT), when the wine is bottled and stored at low temperatures. The precipitation of both tartaric salts occurs during the alcohol fermentation of wines, producing a supersaturated solution in them. Thus, the crystals deposits are neither desirable for wine production, nor for consumers (Low et al. 2008). Hence, the most widely used and traditional technique for wine stabilization is the cold treatment. It consists in cooling the wine to a temperature close to its freezing point and storing it between 3 days and 3 weeks, being 1 week the most often. However, it is a time-spending and energy-consuming method (Lasanta and Gómez 2012). In addition, cold treatment does not allow a precise control of the final KHT concentration and the wine quality can be affected by simultaneous polysaccharides and polyphenols precipitation together with the KHT salts (Gonçalves et al. 2003; Soares et al. 2009). For that reason, ED is proposed as a suitable membrane technique not only to remove KHT and tartaric acid (H₂T) in almost the same way observed in the conventional cold stabilization process, but it also permits a specific reduction degree of organics acids (e.g. lactic and malic acids) as well as cationic species (Mg²⁺, Ca²⁺, and Na⁺) (Fidaleo and Moresi 2006). In this context, some ions from wines, such as potassium, calcium and tartrate were extracted by ED, which helps to reduce the over saturation level of tartaric acid salts (Daufin et al. 2001). In fact, Gonçalves et al. (Gonçalves et al. 2003)

highlighted that calcium removal is crucial to achieve tartrate stability in terms of both KHT and CaT. Nevertheless, in the ED treatment other molecules like polyphenols (anthocyanins and tannins), polysaccharides, amino acids and volatile compounds were unaffected (Daufin et al. 2001).

Tartaric stabilization of wines by ED is industrially conducted. To achieve the required level of K^+ , the wine is circulated in the diluate compartments of the ED stack. Under an electrical field influence, the organic anions (containing tartrate, lactate and maleate) move towards to the anode permeating through the AEMs, whereas the cations (principally K^+ , but also Mg^{2+} , Ca^{2+} , and Na^+) drive towards to the cathode crossing the CEMs. The concentrate compartments are filled with a salt solution (e.g., NaCl or KHT). In addition, a slight decrease in the pH of wine is commonly observed (0.15–0.25 pH units), since the anions permeation is slower than for the cations (Mondor et al. 2012).

The main advantage to use ED is that the energy cost of tartaric stabilization is very low; since the total electricity consumption is between 0.5 and 1 kWh/m³ of treated wine (including pumping). Comparing with traditional methods, in which refrigeration is used, the energy required is about 10 times less by ED process. Additionally, the sensorial and organoleptic properties in wines were not modified by the ED process (Daufin et al. 2001). For instance, Gonçalves et al. (Gonçalves et al. 2003) commented that the organoleptic characteristics of wines (colour, aroma and taste) showed no differences when wines were treated by ED or conventional cold stabilization processes. For that reason, ED is a process implemented in several industrial treatment units of different capacities (4000–10,000 L/h) in France, Italy and Spain since 1997 (Daufin et al. 2001).

Forsyth (2010), from the Australian Wine Research Institute, made a comparison between ED and cold treatment, as a method to produce potassium tartrate stable wine. Results showed that ED offered considerable assets in power consumption (77 kWh for ED vs 1761–2968 kWh for cold treatment), time taken to process wine (17 h vs 384 h), and in wine losses minimization (136 L vs 424 L). Besides, there was no sensorial difference in the wines treated by ED in comparison with the cold technique. Nevertheless, wastewater volume (7683 L vs 1581 L) and labour requirements (17 h vs 9 h) were higher for ED than for the cold method. This report concluded that, based on the obtained results, ED appeared to offer a sustainable alternative method for tartrate stabilization in wines.

Several examples of tartaric acid stabilization in wines by using ED process are collected in Table 5.4. For practical considerations, to predict the degree of deionization (DD) that renders the wine stable, it is necessary in advance the removal of potassium and bitartrate ions from wine by ED (El Rayess and Mietton-Peuchot 2016). Two assays, based on rapid response conductivity techniques, such as saturation temperature and mini-contact test, can be used to determine the tartaric stability. First, the tartaric acid stability can be determined by the saturation temperature (T_s) in KHT, contained in the wine, being high stability at low values of T_s . The saturation temperature is obtained by measuring the electrical conductivity during a cycle by increasing the temperature of two samples, a control (without KHT) and other adding KHT. The T_s is reached when the conductivity of the two samples is

Table 5.4 ED applications from tartaric acid stabilization in wines: review of applications and process performances

Feed solutions		Operational conditions			Main results	Ref	
Diluate stream (treated solution)	Concentrate stream Electrode rinse	CEM AEM	ED set-up Membranes	Flow rates			Constant voltage or current Temperature Experimental time
24 L of white wine and 14 L of red wine – “Vinho Verde” wines	0.2 g/L NaCl –	CMX Sb (Tokuyama Soda) AMX Sb (Tokuyama Soda)	EUR2C-7P18 unit (Eurodia) 2 dm ² ; 7 cells	–	1 V/cell 25 °C 2.5 h	DD for white wines: 4.7–30.1% DD for red wines: 5.5–20.6% KHT removal: 24% H ₂ T removal: 10.9% (at T _s = 14.8 °C and DD = 14.5%) Ca removal: 39% Lactic and malic acids contents were almost constant	(Concalves et al. 2003)
Sherry wines	– –	– –	– 25 m ²	25 hL/h	– – –	DD for “Fino” wine: 19.6–40.4% DD for “Medium” wine: 20.8–30.1% DD for “Cream” wine: 18.3%	(Gómez Benítez et al. 2003)
3 L – white, rose, red, and fortified wines	3 L – 0.2 mol/L KHT (pH = 3) 3 L – 0.2 mol/L K ₂ SO ₄	CMX Sb (Tokuyama) AMX Sb (Tokuyama)	EUR2C-7P18 unit (Eurodia) 2 dm ² ; 7 cells	180, 250, and 400 L/h for concentrate, diluate, and electrode rinse solution, respectively	1.5 V/cell 25 °C 18 min, 4 h, 24 h and 65 h	DD: 10–30%	(Soares et al. 2009)

Table 5.4 (continued)

Feed solutions		Operational conditions				Main results	Ref
Diluate stream (treated solution)	Concentrate stream Electrode rinse	CEM AEM	ED set-up Membranes	Flow rates	Constant voltage or current Temperature Experimental time		
White, rose, red, and fortified wines	– –	– –	Pilot device: 150 cells (Inra/Eurodia) Industrial unit: Two stacks of 150 cells (semi-automatic Eurodia)	Pilot scale: 15 hL/h Industrial scale: 30 hL/h	– – 7 h	Overall electrical energy consumption at industrial scale: 0.21 kWh/hL Water consumption: 5.5 L/hL wine	(Bories et al. 2011)
10,500 L – White and red wines	– –	– –	Model ED15 (Juclias) 30 m ² ; 150 cells	15 hL/h	– –	DD of white wines: 21–23% DD of red wines: 16–18%	(Corti and Paladino 2016)

the same (Lasanta and Gómez 2012). However, this parameter is not very accurate due to the huge metastability of KHT and the presence of crystal growth inhibitors. Therefore, a test, called mini-contact, has been developed, which consists in cooling a wine sample (0 °C) and measuring its conductivity for 4 h after the addition of KHT (4 g/L) that causes salt precipitation. The difference between conductivity at the beginning and at the end of the experimental time gives an estimate of the DD required to stabilize a wine by ED (Lasanta and Gómez 2012; Soares et al. 2009; Henriques et al. 2019; El Rayess and Mietton-Peuchot 2016). For example, Soares et al. (2009) developed a study to predict the required DD to stabilize wine by ED based on the mini-contact test. Then, they evaluated the tartaric stability of wines by the freezer test, the long-term storage test, and saturation temperature. In the study, the DD was predicted using the mini-contact test, which simulates the cold treatment with seeding, at a bench-scale during 65 h with two different particle size distribution of KHT crystals. Otherwise, freezer test is the traditional method to check when crystalline sediments appear during the thawing of a previously frozen wine sample. Different wine samples with DD between 10% and 30% were obtained by ED. They also observed, with mini-contact assays, that the DD required for tartaric acid stability of the electrodialysis-treated wine was strongly dependent on experimental time and was also influenced by KHT crystal granulometry. Furthermore, it is important to mention that it is necessary to use KHT, with a controlled size distribution, to enhance the mini-contact test repeatability. Afterwards, wines that overcame from the freezer test (with no prefiltration) were stable during 6 months of storage at 6 °C. Recently, Henriques et al. (2019) proposed both active and passive controlled freeze-thawing tests to predict the deionization degree required for tartaric stabilization by ED, and then compared it with the mini-contact test. In this study, the wine was frozen (−20 °C) and thawed (0 °C) in controlled conditions. They showed that freeze-thawing assays gave reproducible results, that were between 5 and 9% higher DD than the corresponding values obtained by the mini-contact test at −4 °C during 4 h. Because of that, they concluded that the controlled passive freeze-thawing test could be a reliable and low-cost alternative to the mini-contact test that can yield in 24 h an estimation of the DD of wines for tartaric stabilization by ED.

In regards with ED applications for tartaric acid stabilization in wines, Gonçalves et al. (2003) studied the KHT removal performance for wine tartaric stabilization by using ED. The wine saturation temperature was used to assess the tartaric stability. The study was carried out in a pilot scale (from Eurodia) with an ED stack composed by 7 cells with 2 dm² effective area (9 cationic CMX Sb and 7 anionic AMX Sb membranes, all from Tokuyama Soda). The wine samples used were two “Vinho Verde” wines, a white and a red, from grapes harvested in 1998 (Portugal). Results showed that the wine saturation temperatures varied linearly with the deionization degree. Regarding with white wines, it was possible to achieve a DD of 14.5% and a tartaric acid removal of 10.9% when the saturation temperature was 14.8 °C and stability up to 0 °C. In addition, the lactic and malic acids contents were kept almost constant, while the calcium content was reduced by 39%. For red wines, the saturation temperature was 9.2 °C, indicating a more stable wine.

Gómez Benítez et al. (2003) compared the efficacy of cold treatment and ED for tartrate stabilization, at industrial scale, of three sherry wines (“Fino”, “Medium” and “Cream”). The difference in the studied wines was the sugar content, being <2 g/L, 40 g/L and 100 g/L of sugar, respectively. Conductivity techniques for rapid tartaric stability control, such as saturation temperature and mini-contact test, were used. They checked that the mini-contact test provided accurate information on stability in comparison with saturation temperature assay. Additionally, in the three studied wines, the cold treatment guaranteed the tartrate stability; while for ED, to obtain a similar stability it was necessary to apply a DD value higher than 26% in the “Fino” wine and lower than 20% for “Medium” and “Cream” wines. Authors also noted that the sulphate content was reduced more than the tartrates, and the sensory characteristics of sherry wines were slightly affected, depending on the applied DD.

Bories et al. (2011) evaluated the environmental impacts of tartaric acid stabilisation processes for wines using ED (pilot and industrial scale) and cold treatment. In the case of ED at industrial scale (30 hL/h), a RO unit was coupled to treat the generated brines in the ED process, and then to recycle the permeates from RO into the ED device. It is worth noting that this ED-RO hybrid process allowed the reduction of 65% of the overall water consumption in comparison with the ED without brine treatment. Comparing ED and cold method, results showed that ED presented less wine loss and minor waste generation because of the filtration step with diatomaceous earth involved in the cold technique. Besides, the overall electrical energy consumption for tartaric stabilisation by ED (2.1 kWh/m³) resulted in eight times lower than the cold stabilisation treatment.

Besides, Daufin et al. (2001) stated that the integration of ED and MF, as one-step process and in a continuous system, is an innovative hybrid process to solve some issues in wine industry as follows: (i) microbiological stability, (ii) clarification, and (iii) tartaric stabilization with an excellent protection against oxidation without any additive.

On the other hand, ED is also proposed as a grape must rectification step for wine production. The conventional process for concentrate grape must production is evaporation, followed by ion-exchange resins for rectification; however in these processes numerous aromas compounds and organic acids are removed (Bazinet and Firdaous 2011). As an example, Correia de Pinho et al. (2006) patented a NF and electrodialysis hybrid process to simultaneously concentrate and partially rectify grape must. In this application, ED can be performed before the NF, after the NF or both, before and after the NF. They proposed the ED before the concentration step by NF, when the processing grape musts presented high potassium bitartrate concentration, to decrease from 10 to 40% of grape must ions. Thus, by reducing the precipitation of tartrate salts, it was possible to avoid NF membranes fouling after the ED stage. Also, ED could be used after the NF concentration step, when the initial concentration of potassium bitartrate in the grape must is low, to control the concentration of organic acids in the final grape must concentrate. It is important to note that the process does not require thermal separation and can be operated at room temperature or at temperature ensuring the preservation of volatile and aromas

compounds. As suggested by the authors, the process can be used for concentration and rectification of pulps and juices of fruits.

ED, with monopolar membranes, is also employed to recover tartaric acid (Zhang et al. 2011; Andrés et al. 1997; Kaláb and Palatý 2012; Eliseeva et al. 2012) and to reduce potassium content (Barros et al. 2019; Decloux et al. 2002) in vinasses as an effective way of wine waste treatment. Furthermore, ED with bipolar membranes, is used to produce acid (e.g. H_2T) and base (e.g. KOH) solutions from the salt (KHT) found in the vinasses (Zhang et al. 2009, 2012b; Vecino et al. 2020). Some examples about the vinasses valorisation by using ED and EDBM are summarized in Table 5.5.

Andrés et al. (1997) studied the ED process as an alternative method to purify and concentrate tartaric acid from IX (ion-exchange) regeneration waters obtained in grape juice industry. In that work, it was possible to reach a final tartrate concentration about 50 g/L from an initial tartrate concentration in the grape juice wastewaters about 5 g/L; so a concentration factor of 10 was achieved after 3.7 h, by using an ED configuration (anode)-CEM-AEM-(cathode) with 10 cell pairs and an effective membrane area of 0.2 m².

Zhang et al. (2011) evaluated the production of tartaric acid by EDM (QianQiu Environmental Protection & Water Treatment Corporation) applying different current densities (300 to 600 A/m²) adding a resin or without adding it in the EDM process. The concentration factor obtained was 3.25 using 2 cell pairs ((anode)-AEM-CEM-(cathode) configuration) in presence or absence of the resin at 300 A/m² with a membrane area of 25 cm² after 5 h.

Kaláb and Palatý (2012) investigated mathematical models to predict tartaric acid concentration in the diluate and concentrate streams by ED with a CEM-AEM-CEM configuration. The IXMs used were Ralex-CMH-PES and Ralex-AMH-PES and were supplied by the Mega Inc. Company (Czech Republic). A concentration factor of 3.8 was achieved for tartaric acid in the concentrate compartment, under a range of current densities from 50 to 130 A/m², by using an ED-set-up with 10 cell pairs.

Eliseeva et al. (2012) studied the ED process of tartaric acid solutions and its salts, achieving tartaric acid percentage removal of 62% from an initial tartaric acid solution. A stack with 7 cell pairs was used, with a membrane area of 20 cm², and a (anode)-AEM-CEM-(cathode) as membrane configuration.

Barros et al. (2019) tested different configurations in the ED process for vinasses desalting and potassium recovery. ED system comprised 2 cell pairs and an effective membrane area of 16 cm². The membranes used in this study were Neosepta® homogenous selective monovalent cation (CMS) and anion (ACS) exchange membranes from Astom Co. (Japan), and non-selective heterogeneous HDX membranes (HDX 100 (cationic) and HDX 200 (anionic)) supplied by Hidrodex®. The vinasses were obtained from a sugarcane juice distillery plant and the configurations were as follows: (anode)-CEM-AEM-(cathode) for both monovalent-selective membranes or both non-selective membranes; and a mix configuration as (anode)-CEM (non-selective)-AEM (non-selective)-CEM (selective)-(cathode). Using all three configurations during 8 h, the maximum removal from raw vinasses was around 90% for K^+ and SO_4^{2-} and about 80% for Ca^{2+} and Mg^{2+} . Additionally, with the ED mixed

Table 5.5 ED and EDBM applications for vinasses revalorization: review of applications and process performances

Main purpose		Feed solutions		CEM AEM	ED set-up Membranes	Operational conditions		Main results	Ref
		Diluate stream (treated solution)	Concentrate stream Electrode rinse			Flow rates	Constant voltage or current Temperature Experimental time		
Potassium tartrate concentration	1L distilled vinasses (synthetic solution)	1 L distilled vinasses (synthetic solution)/1 L distilled vinasses (synthetic solution)	CEM Type II (FujiFilm), PC SK (PCCell)/ AEM Type II (FujiFilm), PC-100D (PCCell)	ED64-4 (PCCell, Germany)/64 cm ; 3 cell pairs	Diluate and concentrate: 15-20 L/h; Electrode rinse: 90-100 L/h	5.5 V/ 23 °C/6 h	KHT desalting = 89.4 %; CF (KHT) = 2.2	Vecino et al. 2020	
Tartaric acid recovery and concentration	45 L – fruit juice wastewater (10 kg/m ³ H ₂ T and 60 kg/m ³ glucose)	2 L – 10 kg/ m ³ H ₂ T 10 L – 20 kg/ m ³ Na ₂ SO ₄ (pH = 2)	SC-1 (Stantech) SA-1 (Stantech)	LT-1 stack (Stantech) 0.2 m ² ; 10 cell pairs	–	120 A/m ² 25 °C 3.7 h	H ₂ T final concentration = 53.2 kg/ m ³ H ₂ T purity ~60%	(Andrés et al. 1997)	
Potassium content reduction	Beet molasses stillage	2 L – 5 g/L NaCl 2 L – 21 g/L KNO ₃	CMXbs AMXsb	EUR 2B IOP30 pilot unit (Eurodia) 0.2 m ² ; 10 cells	–	17 V 19–26 °C 40 min	77.8% of K reduction CF = 7	(Decloux et al. 2002)	

ED

Tartaric acid production	Synthetic solution of KHT (0.5 M)	0.05 M H ₂ T 0.5 M Na ₂ SO ₄	Heterogenous CEM (Qianqiu Environmental Protection & Water Treatment Corporation) Heterogenous AEM (Qianqiu Environmental Protection & Water Treatment Corporation)	– 25 cm ² ; 2 cell pairs	–	300 to 600 A/ m ² – 5 h	H ₂ T CF = 3.25	(Zhang et al. 2011)
Tartaric acid production	Synthetic solution of H ₂ T (0.2 to 0.35 kmol/m ³)	0.05 to 0.35 kmol/m ³ H ₂ T 0.2 kmol/m ³ H ₂ T	Ralex-CMH-PES (Mega) Ralex-AMH-PES (Mega)	ED-Z mini (Mega) 64 x10 ⁻⁴ m ² ; 10 cell pairs	Diluate and concentrate: 65 L/h Electrode rinse: 75 L/h	50 to 130 A/m ² 25 °C –	H ₂ T CF = 3.8	(Kaláb and Palatý 2012)
Tartaric acid production	Synthetic solution of H ₂ T ^a (0.05 mol/L, pH = 1.95) or KHT ^b (0.01 mol/L, pH = 5) or lysine hydrotartrate ^c (0.05 mol/L, pH = 2.75)	– Na ₂ SO ₄	Heterogeneous MK-40 (Shchekinoazot) Heterogeneous MA-41 (Shchekinoazot)	B5-50 unit 20 cm ² ; 7 cell pairs	0.1 cm/s (velocity)	~0.5–5 mA/cm ² ~0.25–2 mA/cm ² ~5–14 mA/cm ² – –	H ₂ T extraction = 62%	(Eliseeva et al. 2012)

(continued)

Table 5.5 (continued)

Vinasses desalting and potassium recovery	1 L – distilled vinasses (raw and ultrafiltrated) from sugarcane juice	– 1 L – 0.03 M Na ₂ SO ₄ (pH = 8)	Selective monovalent NEOSEPTA® CIMS and NEOSEPTA® ACS (ASTOM) Non-selective heterogeneous HDX (Hidrodex®)	– 16 cm ² ; 2 cell pairs	10 L/h for all streams	0–210 A/m ² – 8 h	72% of potassium recovery applying 60 A/m ² (mixed configuration) Energy consumption = 9 kWh/m ³ Current efficiency = 54%	(Barros et al. 2019)
EDBM								
Main purpose	Feed solutions		CEM AEM BPM	EDBM set-up Membranes	Operational conditions		Main results	Ref
	Diluate stream (treated solution)	Concentrate stream I Concentrate stream II Electrode rinse			Flow rates	Constant voltage or current Temperature Experimental time		
Tartaric acid production	1L concentrate vinasses by ED	1 L 0.05 M H T / 1 L 0.05 M KOH/1 L concentrate vinasses by ED	CEM Type II (Fujifilm)/AEM Type II (Fujifilm)/PC-bip (PCCell)	ED64-4 (PCCell, Germany)/64 cm ² ; 3 cell trios	Diluate, acid and base: 15-20 L/h; Electrode rinse: 90-100 L/h	10 V/ 23 °C/16 h	9.9 g/L H T, 69.7% purity and 5.6 g/L KOH, 77.3% purity	Vecino et al. 2020

Tartaric acid production	Synthetic solution of KHT (0.5 M)	0.05 M H ₂ T 0.2 M KOH 0.5 M Na ₂ SO ₄	Homogeneous FT-FKB CEM (FuMA-tech GmbH) Heterogenous AEM (Qianju Environmental Protection & Water Treatment Corporation) FT-BP (FuMA-tech GmbH)	- 25 cm ² ; 1 cell trio	-	300 to 700 A/m ² - 5 h	H ₂ T final concentration = 14 g/L at 300 A/m ² (CF = 2.8) H ₂ T final concentration = 16 g/L at 400 A/m ² (CF = 3.2) H ₂ T final concentration = 20 g/L at 500 A/m ² (CF = 4.0) H ₂ T final concentration = 25 g/L at 700 A/m ² (CF = 5.0)	(Zhang et al. 2009)
Tartaric acid production	Synthetic solution of KHT (0.5 M)	0.05 M H ₂ T 0.05 M KOH Na ₂ SO ₄ (0.5 M)	Heterogenous CEM (Qianju Environmental Protection & Water Treatment Corporation) Heterogenous AEM (Qianju environmental protection & water treatment corporation) FT-BP (FuMA-tech GmbH)	- 150 cm ² ; 4 cell trios	15 L/h	66.7 to 200 A/m ² - 5 h	H ₂ T final concentration = 55 g/L at 66.7 A/m ² (CF = 3.7) H ₂ T final concentration = 95 g/L at 133.3 A/m ² (CF = 4.75) H ₂ T final concentration = 135 g/L at 200 A/m ² (CF = 5.4)	(Zhang et al. 2012b)

CF: concentration factor

configuration, an energy consumption of 9 kWh/m³ was reached for K⁺ recovery (72%) at 60 A/m².

Respect to EDBM examples, Zhang et al. (2009) carried out different assays varying the current density (300–700 A/m²) and adding a resin or without adding it in the EDBM process. The membranes used were heterogeneous AEMs supplied from QianQiu Environmental Protection Water Treatment Corporation (China), and a homogeneous FT-FKB CEM and a FT-BPM commercialized by FuMA-Tech GmbH (Germany). Fixing the current density value at 300 A/m² and using the following configuration (anode)-BPM-AEM-CEM-BPM-(cathode), independently of the presence or absence of the resins, the produced tartaric acid concentration was about 14 g/L after 5 h. Nevertheless, increasing the current density up to 700 A/m², it was possible to reach 25 g/L of H₂T.

In another work, Zhang et al. (2012b) evaluated ion conductive spacers for energy-saving production of tartaric acid instead of conventional spacers. CEMs and AEMs, were supplied by QianQiu Environmental Protection & Water Treatment Corporation, whereas the BPMs were commercially obtained from FuMA-Tech GmbH (Germany). The initial concentration of H₂T and KOH were 0.05 M in the acid and base compartments, respectively, and the configuration used was (anode)-CEM-BPM-AEM-CEM-(cathode). Under the above-mentioned conditions, it was possible to reach a concentration factor of 3.7 regardless of the spacer type (ion conductive or conventional) using a current density of 66.7 A/m² after 5 h. However, by increasing the current to 200 A/m², it was possible to achieve 135 g/L of tartaric acid providing a concentration factor of 5.4.

On the other hand, by using EDBM process the acidification and de-acidification of musts and wines are possible (El Rayess and Mietton-Peuchot 2016; Comuzzo and Battistutta 2018). High pH (about 4) is presented in wines not only because of the deficit of organic acids, but also due to cations excess such as potassium (El Rayess and Mietton-Peuchot 2016). In this context, the natural acidity in musts and wines can be caused by the climatic conditions in the viticulture region or due to oenological practices that lead to a decrease in natural acidity (Castelluci 2010). Several properties such as microbiological stability, physico-chemical, colour stability and organoleptic quality of wines depend on the wine acidity. For that, tartaric acid was proposed as an acidulant to correct the pH of musts and wines, being 1.5 g/L and 2.5 g/L the maximum dosage for them, respectively (El Rayess and Mietton-Peuchot 2016; Moldes et al. 2017).

In 2010, according to the International Organisation of Vine and Wine (OIV), the use of EDBM was accepted as an acidification method to treat musts and wines. The goals of this method consist of: (i) increasing of titratable acidity and actual acidity (decrease of the pH); (ii) obtaining wines with balanced taste characteristics; (iii) promoting a good biological evolution and proper storage of the wine; and (iv) remedying insufficient natural acidity (Castelluci 2010).

The steps of wine acidification process by EDBM are described as follows: when the electric current is applied, the K⁺ ions contained in the must or wine are drawn to the cathode (the negative pole), they pass through the CEM and are stopped by the BPM. The electric current that is applied between the two electrodes splits water molecules into OH⁻ and H⁺ inside the BPM, that is in contact with must or wine.

The OH^- ions migrate to the anode (the positive pole) into the concentrate stream, while the H^+ ions migrate to the cathode and replace the K^+ ions that are extracted from the must or wine in order to conserve the ion equilibrium. EDBM causes acidification (pH decrease) by lowering the potassium content in the wines. For a reduction of pH values, there is a concomitant enhancement in titratable acidity (El Rayess and Mietton-Peuchot 2016). As suggested by the OIV, the total acidity must not exceed 54 meq/L (4 g/L expressed as tartaric acid) when musts and wines are acidified (Castelluci 2010).

On the other hand, the process when the titratable acidity of musts and wines is reduced is called de-acidification process. Yeasts (e.g. *Schizosaccharomyces pombe*) or bacteria (e.g. lactic acid bacteria) lead to de-acidification in wine during the fermentation process. However, the physico-chemical de-acidification implies acid precipitation or ion-exchange processes in a fixed-bed configuration. Calcium carbonate or potassium bicarbonate can be de-acidification agents that involve tartaric acid precipitation as insoluble salts (El Rayess and Mietton-Peuchot 2016). For all, in 2012 it was accepted, by the OIV, the de-acidification of musts and wines using ED with bipolar membranes (Castelluci 2012). The principle of must or wine de-acidification by EDBM is similar to the conventional acidification one, but the anions (e.g. TH^- and T^{2-}) are concerned in this process. The application of the electric current drives the anions to the anode; they pass through the AEMs and are stopped by the BPMs. The anion forms of organic acids are transferred from the feed compartment to the concentrate compartment where they are associated with H^+ ions, missing their ionic form. Thus, the wine is poorer in organic acids, reducing the titratable acidity, and in consequence the must or wine is de-acidified (Mondor et al. 2012). The OIV proposed that the wine from a de-acidified process should contain at least 1 g/L of tartaric acid (Castelluci 2012).

3 Concluding Remarks

In this chapter, the potential of electro dialysis membrane technology for cost-effectively separation process in agro-food industries has been examined. ED is not only used for agro-food streams processing, but also it is applied for agricultural wastes and by-products valorisation, generated from agro-food industries. The reduction, treatment and recycling of agro-food streams is a matter of utter importance nowadays. Society and governments in industrialized countries, as well as social and environmental organizations, businesses and academics have developed an environmental awareness, demanding cleaner production systems from companies as electro dialysis. Among them, ED applications, with monopolar and bipolar membranes, in this chapter are focused on dairy and winery industry sectors. In the case of dairy industry, ED is mostly applied as a demineralization step of milk whey, however ED can be used for production of protein fractions, lactose recovery or lactic acid removal. Concerning winery sector, the main application of ED is in the tartaric acid stabilization of wine; but also, it can be used for tartaric acid and potassium recovery from vinasses.

All in all, ED is proposed as an alternative to traditional processes, having a more sustainable and eco-friendly approach following the frame of circular economy and in line with the industrial symbiosis. ED is a technology with several highlights such as modular design, automatic, easy to operate, food safety, minimum waste production and competitiveness. Additionally, future research may be addressing in: (i) new membrane manufacturing: develop new ion-exchange membranes, improve the performance of conventional technologies by enhancing mass transfer and retarding fouling/scaling; (ii) water transport reduction through the membranes, (iii) membranes development with lower electrical resistance (iv) new membrane stack designs; (v) novel hybrid processes (traditional techniques with membranes); and (vi) economic analysis for agro-food industries.

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