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Winery wastewater treatment for biomolecules recovery and water reuse purposes

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11.1 Introduction

Viniculture is a very important agroindustrial activity worldwide, especially in the Mediterranean region where the three largest wine producing countries are located, namely Italy, France and Spain. Such is the economic importance of this sector that, in the last decade (2011–20), the average global wine production was approximately 270 MhL, from which about 100 MhL were destined for exports (OIV, 2021). According to data from the Food and Agriculture Organization of the United Nations, in 2019 the world export value of wine exceeded US$33 billion (FAO, 2021).

Without a doubt, viniculture plays an important economic role in many countries, but like any agroindustrial activity, it also causes environmental impacts, such as the generation of wastes and wastewaters throughout the production process (Giacobbo et al., 2019; Giacobbo, Bernardes, Rosa, & de Pinho, 2018). Sometimes there can be some difficulty in managing this environmental liability, since more than 60% of these wastes and wastewaters are generated in a short period of up to 3 months during the vintage and in the first racking

Advanced Technologies in Wastewater Treatment. DOI: https://doi.org/10.1016/B978-0-323-88510-2.00001-4

(Devesa-Rey et al., 2011; Oliveira & Duarte, 2016), demanding a greater effort from the treatment system.

In this regard, wineries typically generate about 0.2–4 L of wastewater per liter of wine produced (Welz, Holtman, Haldenwang, & le Roes-Hill, 2016), but this figure can reach 14 L of wastewater per liter of wine produced (Ioannou, Puma, & Fatta-Kassinos, 2015), varying with the dimensions of the facilities, the type of wine produced (e.g., red, white, or special wines), and the winemaking and cleaning technologies (Giacobbo et al., 2013b; Lofrano & Meric, 2016; Oliveira, Costa, Fragoso, & Duarte, 2019). Winery wastewaters mainly originate from cleaning procedures for reception hoods, destemmers, tanks, presses, vats, barrels, floors, and other equipment and surfaces (Costa et al., 2020). Therefore they are predominantly composed of residues of skins, seeds, stems, lees, losses of wines and musts, cleaning products, and filtration aids (Giacobbo, Meneguzzi, Bernardes, & de Pinho, 2017b; Rodrigues et al., 2006).

In fact, winery wastewater has a high pollutant load and can reach values of chemical oxygen demand (COD) and total solids of up to 49 $g\,L^{-1}$ and 18 $g\,L^{-1}$, respectively (Conradie, Sigge, & Cloete, 2014). The wastewater can contain various contaminants, such as sugars, ethanol, glycerol, organic acids, esters, phenolic compounds, and minerals (Conradie et al., 2014; Mosse, Verheyen, Cruickshank, Patti, & Cavagnaro, 2013). Nevertheless, some of these contaminants are value-added compounds liable to recovery, such as phenolic compounds (Giacobbo, Bernardes, & de Pinho, 2013a). Furthermore, the treated wastewater is an important and low-cost resource that can be reused for irrigation, representing a source of water and nutrients for agriculture (Albornoz, Centurião, Giacobbo, Zoppas-Ferreira, & Bernardes, 2020).

Summing up, the recovery of biomolecules and other substances from winery wastewater and the reuse of treated wastewater reduce the environmental impact of wineries and represent a significant advance in terms of sustainability, with gains in environmental and economic issues and promotion of the circular economy (Giacobbo et al., 2017b; Martins, Araújo, Graça, Caetano, & Mata, 2018). On the basis of these considerations, this chapter will present an overview of winery wastewater and its treatment processes aiming at biomolecule recovery and water reuse purposes. The identification of the best processes for the recovery of byproducts, the definition of their sequencing, as well as the selection of the treatment system for this generated wastewater will also be developed in this chapter.

11.2 Winemaking process and wastewater generation

Annually, wineries generate large volumes of wastewater, which depends on the winery dimension, the winemaking technology, and the specific operation that is being performed (Andreottola, Foladori, Ragazzi, & Villa, 2002; Brito et al., 2007; Coetzee, Malandra, Wolfaardt, & Viljoen-Bloom, 2004; Day et al., 2011; Malandra, Wolfaardt, Zietsman, & Viljoen-Bloom, 2003; Oliveira et al., 2019). Winemaking typically involves receiving grapes, crushing and pressing, processing (including maturation and stabilization), and bottling (Fig. 11.1). During each working period, wastewater volumes are generated from crushing and pressing of grapes and rinsing of fermentation tanks, barrels, other equipment (racking operations), and surfaces (Brito et al., 2007; Oliveira & Duarte, 2016; Zacharof, 2017), differing in their composition and quality.

Briefly, the main stages of the winemaking process and contamination sources are as follows:

1. *Grape reception*. The wastewater generated at this stage is mostly related to the washing of equipment and surfaces. It is rich in suspended solids, dissolved sugars, potassium, and sodium (Day et al., 2011; Oliveira et al., 2019).
2. *Crushing and pressing (must production)*. The grapes are pressed to produce must and solid residues (grape pomace), which consists mostly of skins, seeds, and stems (Genisheva, Macedo, Mussatto, Teixeira, & Oliveira, 2012). Wastewater is generated during the prewashing of the fermentation tanks and the washing of the equipment and the production hall. It can also contain must loss as a result of the racking operation. In this stage, the wastewater is rich in dissolved sugars, potassium, and sodium (Day et al., 2011; Oliveira et al., 2019).
3. *Fermentation*. At this stage, wastewater is generated mostly from rinsing of fermentation tanks. It is rich in suspended solids, grape solids, dissolved sugars, wine, potassium, and sodium (Day et al., 2011; Oliveira et al., 2019).
4. *Decanting*. During this process, the wine is decanted from the wine lees (Giacobbo et al., 2019). Wastewater is generated during prewashing and washing of the stabilization tanks and production room and during pump cleaning. At this stage product losses can occur (Vlyssides, Barampouti, & Mai, 2005). The wastewater is rich in suspended solids, grape solids, dissolved sugars, wine, potassium, and sodium (Day et al., 2011; Oliveira et al., 2019).

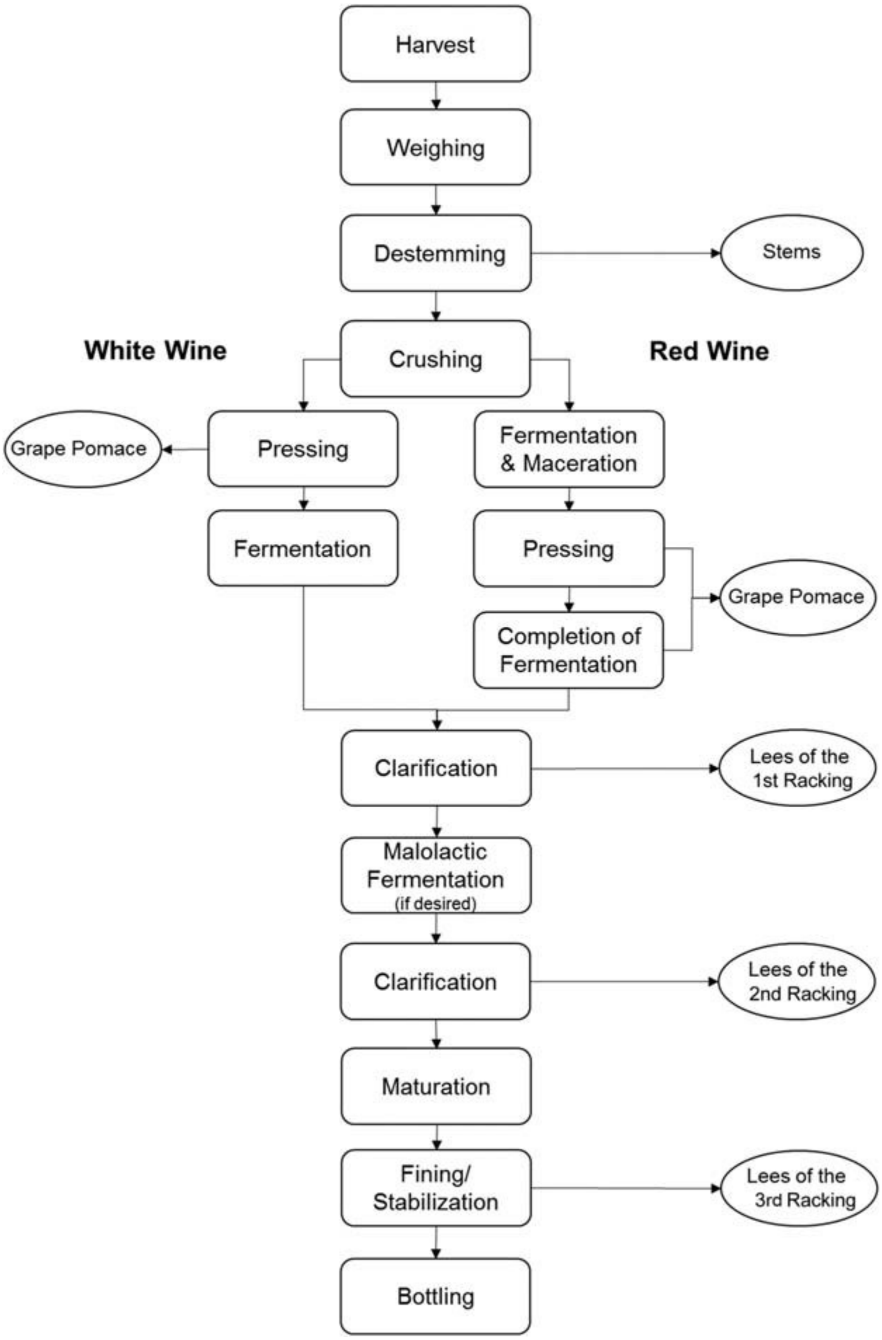

Figure 11.1 Diagram of the winemaking process for white wine and red wine. Adapted from Devesa-Rey, R., Vecino, X., Varela-Alende, J. L., Barral, M. T., Cruz, J. M., & Moldes, A. B. (2011). Valorization of winery waste vs. the costs of not recycling. Waste Management (New York, N.Y.), 31, 2327–2335. https://doi.org/10.1016/j.wasman.2011.06.001; Oliveira, M., & Duarte, E. (2016). Integrated approach to winery waste: Waste generation and data consolidation. Frontiers of Environmental Science & Engineering,10, 168–176. https://doi.org/10.1007/s11783-014-0693-6; Zacharof, M.-P. (2017). Grape winery waste as feedstock for bioconversions: Applying the biorefinery concept. Waste and Biomass Valorization, 8, 1011–1025. https://doi.org/10.1007/s12649-016-9674-2.

5. *Maturation-stabilization*. Wastewater comes from the washing of the tanks and is rich in tartrate solids, fining agents, polyphenols, polysaccharides, potassium, and sodium (Day et al., 2011; Giacobbo et al., 2013b; Oliveira et al., 2019).
6. *Tartaric stabilization*. The excess of potassium hydrogen tartrate is removed from the wine by subtractive or additive methods. One of the methods uses a membrane process, electrodialysis, which gives rise to two different flows, the electrodialysis-treated wine and the wastewater flow, mainly containing potassium hydrogen tartrate and calcium tartrate. Besides this wastewater flow, wastewater is generated during the washing of the tanks and cleaning of the membranes, pumps, and production room. At this stage, wine can also be lost (Bories et al., 2011; Day et al., 2011; Gonçalves, Fernandes, Cameira dos Santos, & de Pinho, 2003).
7. *Filtration*. The wine is filtered to improve its quality. Wastewater comes from the washing of the tanks, from the prewashing of the storage tanks, from the cleaning of filters, from the transportation pump, and from the washing of the production room as well as from the possible wine losses during its transfer (Vlyssides et al., 2005; Zacharof, 2017). At this stage, the wastewater is rich in suspended solids, filtration earths, alcohol, polyphenols, polysaccharides, potassium, and sodium (Day et al., 2011; Giacobbo et al., 2013b; Oliveira et al., 2019).
8. *Bottling*. The produced wine is sold either in bulk or as bottled, which is charged from tanks to transportation trucks or in the packaging unit. At this stage, wastewater comes from the washing of tanks, the washing of equipment, and the washing of the packaging room (Vlyssides et al., 2005). At this stage, the wastewater is rich in suspended solids, polyphenols, and sodium (Day et al., 2011).

This diversity of compounds that constitute winery wastewater, the spatiotemporal dynamics of the wastewater generation between and within wineries, and its potential for recovery and reuse pose real challenges to technologists (Ioannou et al., 2015; Mosse, Patti, Christen, & Cavagnaro, 2011). As was mentioned earlier, the quality and volume of wastewater, the end use for treated wastewater, the local environment and the implementing and operation costs are the main parameters to be considered in the winery wastewater management (Pirra, 2005; Braz, Pirra, Lucas, & Peres, 2010). The qualitative composition of winery wastewater is displayed in Table 11.1.

Table 11.1 Physicochemical composition of winery wastewater.

Parameter	Unit	Minimum	Maximum
Chemical oxygen demand	mg O_2 L^{-1}	320	49,105
Biochemical oxygen demand	mg O_2 L^{-1}	203	22,418
Total organic carbon	mg C L^{-1}	41.0	7363
Total solids	mg L^{-1}	748	18,332
Total suspended solids	mg L^{-1}	66.0	8600
Turbidity	NTU	251	782
Total nitrogen	mg L^{-1}	10.0	415
Total phosphorus	mg L^{-1}	2.10	280
Potassium	mg L^{-1}	5.00	2105
Conductivity	mS cm^{-1}	1.10	5.60
pH	—	2.50	12.9
Total phenolic compounds	mg L^{-1} GAE	0.51	3531
Total sugars	mg L^{-1} GE	100	8000

GAE, gallic acid equivalent; *GE*, glucose equivalent.
Source*: Adapted from Braz, R., Pirra, A., Lucas, M. S., & Peres, J.A. (2010). Combination of long term aerated storage and chemical coagulation/flocculation to winery wastewater treatment. Desalination, 263, 226–232. https://doi.org/10.1016/j.desal.2010.06.063; Ioannou, L. A., Puma, G. L., & Fatta-Kassinos, D. (2015). Treatment of winery wastewater by physicochemical, biological and advanced processes: A review. Journal of Hazardous Materials, 286, 343–368. https://doi.org/10.1016/j.jhazmat.2014.12.043; Pirra, A. J. D. (2005). Characterization and treatment of winery effluents from the Douro Wine Region (Caracterização e tratamento de efluentes vinícolas da Região Demarcada do Douro). University of Trás-os-Montes and Alto Douro. Vila Real, Portugal; Shilpi, S., Seshadri, B., Sarkar, B., Bolan, N., Lamb, D., & Naidu, R. (2018). Comparative values of various wastewater streams as a soil nutrient source. Chemosphere, 192, 272–281. https://doi.org/10.1016/j.chemosphere.2017.10.118; Welz, P.J., Holtman, G., Haldenwang, R., & le Roes-Hill, M. (2016). Characterisation of winery wastewater from continuous flow settling basins and waste stabilisation ponds over the course of 1 year: Implications for biological wastewater treatment and land application. Water Science and Technology: A Journal of the International Association on Water Pollution Research, 74, 2036–2050. https://doi.org/10.2166/wst.2016.226.*

11.3 Value-added biomolecules found in winery wastewaters

As was previously mentioned, winery wastewater contains several contaminants, including commercially important biomolecules that originate from grapes and wine-processing operations, such as phenolic compounds, which are known to have antioxidant properties (Bhise, Kaur, Gandhi, & Gupta, 2014; Giacobbo et al., 2017b).

Polyphenols and other phenolic compounds are secondary metabolites of plants (Cañadas, González-Miquel, González, Díaz, & Rodríguez, 2021). They comprise a wide variety of molecules and may contain only one phenolic ring, such as phenolic acids, or a polyphenolic structure with several hydroxyl groups on aromatic rings, forming a very diverse group (e.g., flavonoids, stilbenes, and lignans) containing several subgroups (Manach, Scalbert, Morand, Rémésy, & Jiménez, 2004). Phenolic compounds may also be associated with one another or with various carbohydrates and organic acids; that more than 4000 flavonoids and 8000 phenolic structures have been identified so far (Cheynier, 2005; Tsao, 2010). Thus this large variety of interactions possibilities between and within groups of molecules results in compounds with a wide molecular weight (MW) range, covering small solutes such as benzoic acid (MW = 122 Da) and proanthocyanidins (tannins) with a degree of polymerization of 80 (Souquet, Cheynier, Brossaud, & Moutounet, 1996), which corresponds to a MW on the order of 25,000 Da. Considering this wide variety of phenolic compounds, they are usually analyzed and quantified as total phenolic content, and gallic acid is conventionally used as a reference standard, so the results are expressed in $mg\,L^{-1}$ or $mg\,kg^{-1}$ of gallic acid equivalent as displayed in Table 11.1. The antioxidant activity of the extract/wastewater is sometimes also analyzed, and the result is usually expressed in $mg\,L^{-1}$ or $mg\,kg^{-1}$ of Trolox equivalent.

The phenolic compounds derived from wine production are usually divided in flavonoids and nonflavonoids, the former being the most important (Oliveira, Ferreira, De Freitas, & Silva, 2011). The flavonoid structure is composed of a C6-C3-C6 skeleton, in which two aromatic rings (A and B) are connected by a central pyran ring (C) (Jackson, 2008; Santos-Buelga & Feliciano, 2017), as illustrated in Fig. 11.2.

The most common flavonoids (Fig. 11.3) in wine are flavonols (kaempferol, quercetin, and myricetin), flavan-3-ols (catechins and proanthocyanidins or condensed tannins), and anthocyanins (Kammerer, Kammerer, Valet, & Carle, 2014). Small amounts of flavan-3,4-diols are also found (Jackson, 2008), while the nonflavonoids (Fig. 11.4) are mainly derivatives

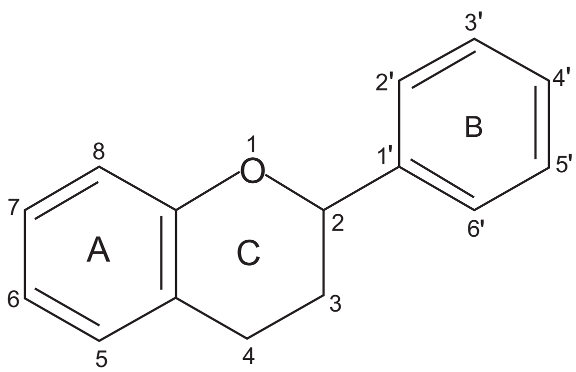

Figure 11.2 Basic flavonoid structure.

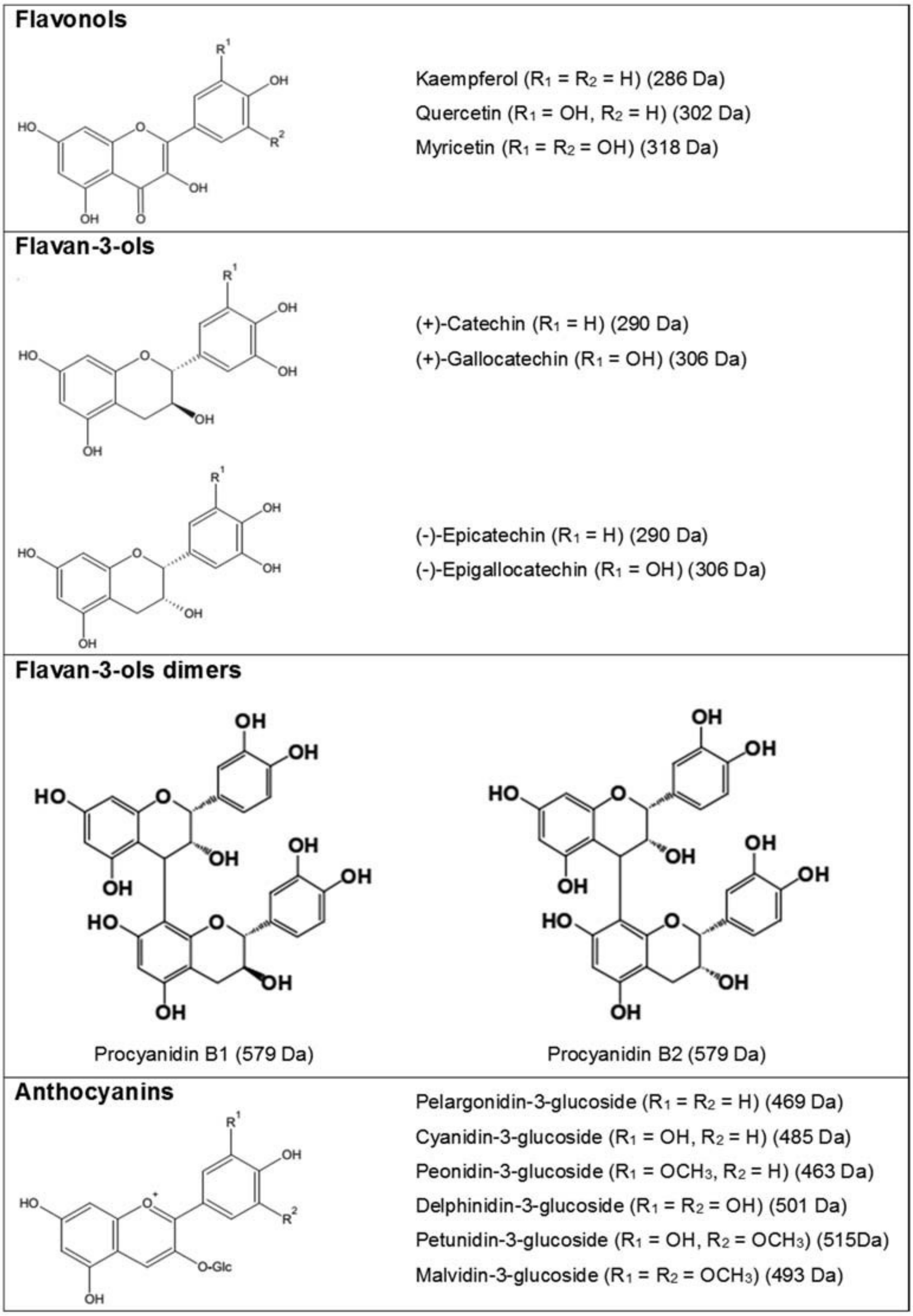

Figure 11.3 Most common flavonoids found in wine. Adapted from (Oliveira et al., 2011; Tsao, 2010). Molecular weight (MW) data from (Pubchem, 2021).

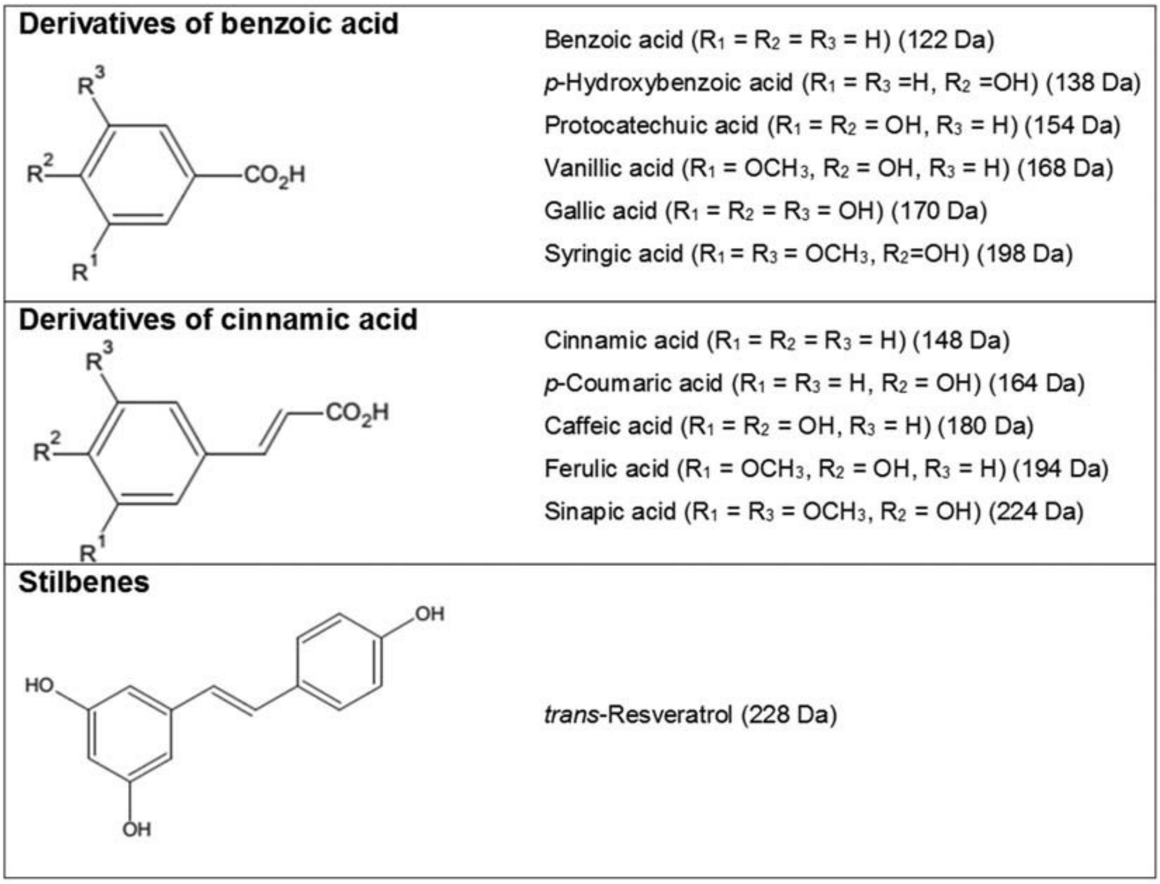

Figure 11.4 Most common non-flavonoids found in wine. Adapted from (Oliveira et al., 2011; Tsao, 2010). MW data from (Pubchem, 2021).

of benzoic and cinnamic acids, although stilbenes, as trans-resveratrol, are also present (Oliveira et al., 2011).

11.4 Winery wastewater treatment systems

In general, wastewater treatments are based on physical, physicochemical, biological, membrane filtration, and advanced oxidation processes (AOPs) (Colin, Bories, Sire, & Perrin, 2005; Ioannou et al., 2015; Mosse et al., 2011). They may be used in different combinations (and sequences) and are generally grouped as primary, secondary, and tertiary treatments.

11.4.1 Physical treatments

Almost all the wastewater treatment operations in wineries involve at least one physical step, predominantly to screen out or settle out the large solids, including grape seeds, stalks, and leaves, thus preventing other treatment machinery from getting clogged with solids, during the primary treatment. The applicability of various physical treatments, such as evaporation (natural or forced), evapoconcentration by fractional condensation, microfiltration (MF), ultrafiltration (UF), electrodialysis, and reverse osmosis (RO) for wastewater wineries have been studied (Durham, Bourbigot, & Pankratz, 2001; Giacobbo et al., 2013b; Jacob et al., 2010; Portilla Rivera, Saavedra Leos, Solis, & Domínguez, 2021; Rengaraj, Yeon, & Moon, 2001; Zhang, Ghyselbrecht, Meesschaert, Pinoy, & Van der Bruggen, 2011), as secondary and tertiary treatments.

In the Mediterranean regions, natural evaporation ponds have been used for a long time in wineries, owing to the low investment and maintenance costs. Although technically simple, this methodology has several drawbacks, including the emission of malodors and contamination of soil and groundwater. The evaporation ponds act as a reservoir of wastewater that is subjected to an evaporation effect, which may be natural or forced, leading to the concentration of suspended particulate organic matter. The forced evaporation system is composed of a buffer tank of small size and high surface alveolar panels (which increase the amount evaporated) with automated injection of a biocide cleaning solution (Clerc, 2004). The effluent from this process can be used in agriculture, applied through irrigation (Clerc, 2004; Masi, Conte, Martinuzzi, & Pucci, 2002). In areas with high land value, the footprint associated with this technology is a relevant issue (Mosse et al., 2011). Saraiva et al. (2020) reported an average water footprint of 2.6 L/FU,[1] which depends on the year under study.

The evapoconcentration to fractional condensation (ECCF; abbreviation from French) is a new biophysical process comprising two stages; in the first the fermentation of sugars occurs by forming ethanol (a biological process,) and the second stage consists in the separation of ethanol from the final effluent. This process can be used as a complete treatment or pretreatment. For complete treatment, which includes demineralization of purified water, Colin et al. (2005) reported an efficiency of COD removal of 99%–99.7%, and for pretreatment (involving only the separation of ethanol), 80% of COD removal was achieved. The final effluent can be reused (e.g., washing operations and industrial applications). The alcoholic

[1]FU: functional unit. The functional unit (FU) selected by Saraiva et al. (2020) was the 0.75 L bottle that is commonly used for wine.

product may be sold or used as fuel. Also, the residual product can be used as a fertilizer because of the content in organic compounds and inorganic compounds. Thus the ECCF appears as a new concept for the treatment of winery wastewater, opening the way for a new generation of wastewater treatment, with a view to sustainable development through the enhancement of the compounds produced and reuse of the final effluent (Colin et al., 2005; Fillaudeau, Bories, & Decloux, 2008).

Physical treatments are used for salt removal, an important step when high sodium ion concentration is present in the wastewater, and the treated water is reused or disposed onto land (Tillman & Surapaneni, 2002). As reviewed by Mosse et al. (2011), there are several technologies for salt removal, such as electrodialysis, ion exchange, and RO, which tend to be disadvantageous for use in most wineries, owing to the high energy consumption and maintenance costs, mainly for the smaller ones. Moreover, the desalination process produces highly concentrated brine, which requires disposal, and to our knowledge, these technologies are not yet being employed in winery wastewater treatment plants. Nevertheless, electrodialysis, ion exchange, and RO are already well-known technologies with large-scale application in a wide variety of industries for the desalination of wastewater for industrial reuse purposes. These methods, although essentially physical, are included in Sections 11.5 and 11.6.

11.4.2 Physicochemical treatments

Within the physicochemical methods there are some processes that are applicable to winery wastewater treatment, in particular chemical precipitation with the addition of chelating agents, sedimentation with the addition of flocculants, coagulation/flocculation, and electrocoagulation and AOPs.

As reviewed by Ioannou et al. (2015) there are several parameters that influence the removal efficiency of the treatment process. However, the electrocoagulation process was shown to be a suitable technology, achieving removal efficiencies very close to those of biological processes. On the other hand, the search for more sustainable treatment technologies showed that the use of the natural coagulant chitosan could be an alternative to chemical coagulants, achieving a COD removal of up to 73% (Ioannou et al., 2015; Rizzo, Bresciani, Martinuzzi, & Masi, 2020).

The AOPs are innovative technologies that have received increasing attention in the research and development of wastewater treatment in the last decades. They provide an alternative for wastewater treatment for the removal or degradation of toxic pollutants. This process can be used as pretreatment to convert recalcitrant pollutants into biodegradable compounds to be treated by a biological process or as posttreatment after a biological step to remove the recalcitrant contaminants. The efficacy of AOPs depends on the generation of reactive free radicals, the most important of which is the hydroxyl radical ($HO^{\bullet}$) used for the oxidation process (Wang & Xu, 2012). Radiation, photolysis and photocatalysis, sonolysis, electrochemical oxidation technologies, Fenton-based reactions, and ozone-based processes are the main types of AOPs that have been described for wastewater treatment in general (Ioannou et al., 2015; Sevillano, Chiappero, Gomez, Fiore, & Martínez, 2020).

Photocatalysis reactions are a subset of AOPs that rely on a catalyst and ultraviolet (UV) or visible radiation to cause oxidation. Commonly, the most widespread catalysts used are Fenton's reagent, titanium dioxide, or ozone; each of them gives different characteristics to the photocatalysis process (de Heredia, Torregrosa, Dominguez, & Partido, 2005; Gernjak, Krutzler, Malato, Caceres, & Bauer, 2001; Ioannou et al., 2015; Lucas, Dias, Bezerra, & Peres, 2008; Ormad, Mosteo, Ibarz, & Ovelleiro, 2006). Although associated with a low cost, the main disadvantage is that Fenton's reagent is a homogeneous catalyst, added as salts of iron, which may remain dissolved, causing additional water pollution. To overcome this issue, the heterogeneous photo-Fenton process emerged, characterized by the use of a semiconductor oxide in the presence of UV or visible radiation, capable of interacting with the Fenton's reagent. Lucas, Mosteo, Maldonado, Malato, and Peres (2009) suggested that the efficacy of the photo-Fenton reaction could be increased if ethanol were previously eliminated from winery wastewater by air stripping. Other authors also reported the use of AOPs with Fenton's reagent as pretreatment, making certain organic compounds more degradable by further biological treatment (Agustina, Ang, & Pareek, 2008; Mosteo, Ormad, Galé, Sarasa, & Ovelleiro, 2004).

The main advantages of photocatalysis with titanium dioxide are the availability of sunlight and the availability, stability, and low price of the catalyst (TiO_2). Moreover, TiO_2 is capable of oxidating of a wide range of organic compounds into harmless compounds such as CO_2 and H_2O (Chatterjee & Dasgupta, 2005).

Among the drawbacks associated with TiO_2 photocatalysis are the difficulty of separating final particles from the aqueous TiO_2 matrix and loss of radiation after recombination. To overcome these drawbacks, coating the surface of the reactor with TiO_2 particles and the use of oxygen excess or addition of inorganic H_2O_2 to prevent light loss have been proposed (Gimeno, Rivas, Beltrán, & Carbajo, 2007).

Photocatalytic ozonation (O_3/UV/TiO_2) is a powerful chemical oxidation method that involves two major pathways of degradation: ozonation (O_3) and direct photolysis. This method is considered superior to ozonation and photocatalysis (UV/TiO_2), owing to synergistic effects (Giri, Ozaki, Taniguchi, & Takanami, 2008), and is emerging as a promising oxidation method for recalcitrant organic contaminants, including pesticides, due to the large number of $HO^{\bullet}$ that are generated (Agustina et al., 2008; de Heredia et al., 2005; Farré et al., 2005; Gimeno et al., 2007; Li, Zhu, Chen, Zhang, & Chen, 2005). The advantages of this method arise mainly from the properties of ozone, a strong oxidizing agent that is not a source of pollution and whose degradation leads to a lower formation of toxic elements. The ozonation process reduces recalcitrant organic matter and enhances the biodegradability of organic compounds, since it allows the formation of smaller and less toxic molecules, which are more easily metabolized by microorganisms. The effectiveness of different ozone-based AOPs in winery wastewater treatment was investigated in a bubble column reactor (Lucas, Peres, & Li Puma, 2010). The O_3/UV/H_2O_2 treatment was shown to be the most efficient for total organic carbon and COD removal, especially if the system is operated at alkaline pH (pH 10), and the most economical process when compared to O_3 or O_3/UV treatments. However, according to Mosse et al. (2011) it is unlikely that ozone-based processes will be used in the winery industry at this stage, since they are rather expensive, require safety precautions (ventilation, maintenance, frequent monitoring), and are relatively complex.

Despite some drawbacks, wastewater treatment with ozone has been recommended both as pretreatment and as tertiary treatment. When used as a pretreatment, it promotes increased biodegradability of the effluent and permits the removal of toxic compounds and inhibitors. When used as tertiary treatment, it allows the removal of the remaining recalcitrant compounds (Beltran-Heredia, Torregrosa, Dominguez, & Garcia, 2000).

According to Ioannou et al. (2015), combined biological and advanced processes (pretreatment and posttreatment) present the most effective technologies applied for the treatment of winery wastewater with a COD removal efficiency of 98%–99.5%.

11.4.3 Natural biological treatments

Biological processes have proven to be efficient to the treatment of wastewaters with high organic loads (Bolzonella, Papa, Da Ros, Anga Muthukumar, & Rosso, 2019). The organic matter in winery wastewater is essentially soluble and quickly biodegradable. For this reason, biological treatment systems are particularly interesting options for this type of effluents (Bolzonella & Rosso, 2009; Torrijos, Moletta, & Delgenes, 2004). Nevertheless, the variable nature of wastewater composition and quantity should be faced, and the treatment plants must be able to handle fluctuations in influent composition and volumes. Concerning the wastewater composition, the toxicity of the wastewater may lead to a partial inhibition of biodegradability because some microorganisms are particularly sensitive to phenolic compounds and some intermediates of their degradation, pesticides, and chemical compounds (de Heredia et al., 2005; Stricker & Racault, 2005).

In a broad sense, biological treatments can be divided into aerobic and anaerobic processes. The first is based on oxygen to facilitate microbial-mediated breakdown of organic matter present in wastewaters; the second occurs in the absence of oxygen, relying on alternative metabolic pathways utilized by a consortium of different microorganisms (Mosse et al., 2011). Nevertheless, the combined use of anaerobic and aerobic treatments is referred as the best option to be used on winery wastewater treatment (Fernández et al., 2007), as Ioannou et al. (2015) advocate the combined use of biological treatment and AOPs.

The preference for anaerobic processes is associated with their proper performance and economy of operation (Rodrigues et al., 2006). When aerobic and anaerobic systems are compared, the aeration costs are proportional to the content of organic matter to be removed, which may lead to quite significant operating costs. In contrast, the anaerobic systems require no aeration, and in the case of use of the biogas that is generated, anaerobic digestion can present a positive energy balance. Also, the anaerobic systems have the advantage of lower production of sludge, owing to the slower growth of anaerobic microbes, have a slower kinetics, thus reflecting higher hydraulic retention times and larger volumes of reactors. They are more sensitive to pH variation and biomass transfer problems and have limitations with respect to degradation of some compounds. Moreover, the startup of anaerobic reactors is often considered to be unstable and dependent on several factors, including wastewater composition, available inoculum, reactor operating conditions, and reactor configuration (Alkarimiah, Mahat, Yuzir, Din, & Chelliapan, 2011; Duarte, Reis, & Martins, 2004; Kalyuzhnyi, Gladchenko, Sklyar, Kurakova, & Shcherbakov, 2000; Oliveira, Neves, & Duarte, 2007; Pérez-García, Romero-García, Rodríguez-Cano, & Sales-Márquez, 2005; Sevillano et al., 2020).

Often, after an anaerobic treatment, is advisable to apply an aerobic treatment as a thinning process that is used to remove organic matter, which is still in the wastewater. Anaerobic processes can also be used as a pretreatment, allowing the reduction of energy and sludge management costs (Rodrigues et al., 2006).

For economic reasons and for their simplicity, the aerobic systems are referred as the most appropriate choice for small wineries (Mosse et al., 2011). In this case, the wastewater generation is low, and expenses associated with aerobic treatment will not be as significant as with anaerobic digestion.

For winery wastewaters, another aspect that is not always optimized involves the removal of inorganic suspended solids, since they can affect the mechanical equipment (e.g., pumps, Venturi type aerators) by abrasion. In addition, as biological processes are not very effective for insoluble compounds; a preliminary treatment is always desirable to also remove the organic suspended solids (Rodrigues et al., 2006).

11.4.3.1 Anaerobic treatment systems

The anaerobic treatment systems show better adaptation to the winery wastewaters than aerobic systems, owing to the high COD/N/P ratio, that is these effluents have low nitrogen and phosphorus contents as compared to carbon. Moreover, by anaerobic digestion it is possible to minimize the energy costs through the recovery of biogas that is produced during the process (Artiga, Carballa, Garrido, & Méndez, 2007; Brito et al., 2007; Mace, Bolzonella, Cecchi, & Mata-Alvarez, 2004; Moletta, 2005). However, the anaerobic systems are often affected by the need to maintain the operating temperature (mesophilic or thermophilic), which is significantly higher than room temperature. Anaerobic reactors operating at low temperatures have been developed (Kalyuzhnyi et al., 2001, 2000). Of the anaerobic digestion technologies that are available for this type of effluents, emphasis is given to those listed in Table 11.2.

One of the most significant drawbacks of anaerobic digestion is the production of volatile fatty acids (VFAs) and other compounds, which are responsible for malodors in the vicinity of wineries (Bories, Sire, & Colin, 2005). To control the odor

Table 11.2 Removal efficiencies of various anaerobic treatment systems

System	Advantages	Drawbacks	COD Removal (%)	Polyphenol removal (%)	References
Upflow anaerobic filters			70 87–90	—	Moletta (2005) Fernández et al. (2007)
Upflow anaerobic sludge blanket	High sludge activity, low sludge production	Relatively high installation costs	90 60 – 70[3] 57[1], 68[2], 70[3]	— — 20[1], 39[2],40[3]	Kalyuzhnyi et al. (2000) Kalyuzhnyi et al. (2000) Kalyuzhnyi et al. (2001)
Upflow Sludge blanket filter			96 – 98	—	Molina, Ruiz-Filippi, García, Roca, and Lema (2007)
Continuous stirred tank digester			62 – 66	—	Mace et al. (2004)
Anaerobic fluidized bed reactor			81.5 – 92.5	—	Pérez-García et al. (2005)
Upflow anaerobic floating filter			47.89 – 75.5	—	Pérez-García et al. (2005)

[1]Temperature: 4°C.
[2]Temperature: 7°C.
[3]Temperature: 10°C.

emission, nitrate salts (e.g., calcium nitrate) can be added to the wastewater, thus preventing the formation of VFAs (Bories et al., 2007). However, this process requires large quantities of added nitrate salt, which is expensive and degrades the final quality of the wastewater. In addition, there is an increased risk of nitrogen runoff into streams and subsequent eutrophication, which represents a threat to the aquatic ecosystems (Burgin & Hamilton, 2007). Therefore the use of nitrate salts should be limited to emergency/backup situations, owing to both economic and environmental impacts (Mosse et al., 2011).

11.4.3.2 Aerobic treatment systems

The high efficiency and versatility that aerobic treatment processes provide allow them to often be the most selected option. The aerobic processes are generally preferred for degrading phenolic compounds because of the lower costs associated with this option and the possibility of complete mineralization of xenobiotic compounds (Ruiz-Ordaz et al., 2001). Several aerobic treatment systems have been developed, as summarized in the Table 11.3.

The lagooning system usually requires large surface areas, frequently in land with high value, and has also problems related to the generation of malodors due to deprived oxygen mass transfer (Agustina et al., 2008; de Heredia et al., 2005; Pirra, 2008). The aerated lagoons are similar to the previous system, but a mechanical stirrer is responsible for oxygenation. The large land areas that are required to implement stabilization ponds makes these systems more advantageous in regions where the cost and availability of land are not a constraint (Mosse et al., 2011).

This process is widely used in France for very small wineries. To reduce the tank volume and the treatment time, the performance of this process may be optimized by combining it with another treatment, such as decantation or thinning treatment with sand filtration, membrane filtration, filtration combined with constructed ponds, or physicochemical processes. This method has advantages such as small production of sludge and absence of sludge recirculation, reduced need for manual labor, and low cost of setting up and maintenance (Racault & Stricker, 2004).

Constructed wetlands (CWs) are classified as a biological treatment based on the principle of infiltration-percolation. These ponds behave like a biofilter in which bacteria located on the surface of carrier material (sand, gravel) degrade the organic matter that is present in the effluent. They can be settled as vertical filters (water is injected into the surface of the system, which promotes oxygen and prevents saturation) or as horizontal filters (the system is permanently saturated). These filters can be used separately or in combination (Kerner & Rochard, 2004). This process can provide considerable efficiency, low cost, low maintenance, and low energy consumption. Furthermore, it is well adapted to accept seasonal flows without adversely affecting functional aspects of the treatment system. According to Shepherd (1998) and Grismer, Carr, and Shepherd (2003), this system was effective in the treatment of winery wastewaters with 5 g COD L^{-1}, and COD loads up to 160 g COD m^{-2} d^{-1}. Removal efficiencies of 85%–97% for COD and 50% for total suspended solids (TSS) were achieved 9.4 days after the startup. Higher COD concentrations may be

Table 11.3 Advantages, drawbacks, and removal efficiencies of various aerobic treatment systems.

System	Advantages	Drawbacks	COD removal (%)	References
Aerated lagoons	Easy management; widespread	Energy intensive; do not always meet the requirements during vintage	91 99 87.5–97.8	Montalvo et al. (2010) Kerner and Rochard (2004) Masi et al. (2002)
Activated sludge	Easy management; high sludge activity; widespread	Relatively high installation costs; energy intensive; supplementation with N and P for microbial growth	87–90 93–95	Brucculeri et al. (2005) Fernández et al. (2007)
Sequencing batch reactor	Low capital costs; simple automation	Requires storage tanks for batch feeding	90–95	Brito et al. (2007); López-Palau et al. (2009); Pirra et al. (2004); Torrijos & Moletta (1997); Torrijos et al. (2004)
Packed-bed bioreactor	Low area requirement	Lab scale	91.1	Petruccioli, Duarte and Federici (2000)
Fluidized-bed bioreactor (FBB)	Low area requirement	Lab scale	88.7	Petruccioli et al. (2000)
Air bubble column bioreactor (ABB)	High efficiency; low area requirement	Lab scale	92.2	Petruccioli et al. (2000)
Jet-loop reactor (JLR)	High efficiency; lowered energy requirements	Limited number of application to date	94–98	Eusébio, Mateus, Baeta-Hall, Almeida-Vara, and Duarte (2005); Eusebio, Petruccioli, Lageiro, Federici, and Duarte (2004); Petruccioli, Cardoso Duarte, Eusebio, and Federici (2002)
Rotating biological contactor (RBC)	Easy to operate; small startup	Maintenance during treatment process	41 43	Coetzee et al. (2004) Malandra et al. (2003)
Membrane bioreactor (MBR)	High efficient; higher organic loading rate and F/M; small footprint; lower sludge production	High installation cost; high energy requirement; membrane fouling.	95 97	Bolzonella et al. (2010) Artiga et al. (2005)

(Continued)

Table 11.3 (Continued)

System	Advantages	Drawbacks	COD removal (%)	References
Fixed-bed biofilm reactor (FBBR)	Simple management; no bulking problems.	Limited number of application to date	91	Andreottola, Foladori, Nardelli, and Denicolo (2005)
Air microbubble bioreactor (AMBB)	High efficiency; lower energy requirements	Limited number of application to date	98	Oliveira et al. (2007, 2009)
Sequencing batch biofilm reactor (SBBR)	High organic loads; sludge recirculation not required; no bulking problems; simple management	High installation cost; requires large area	86–99	Andreottola et al. (2002)

applied if the recirculation of treated wastewater is performed (Kerner & Rochard, 2004). However, this system should be considered only when the wineries have large viable areas (Kerner & Rochard, 2004; Masi et al., 2002). Moreover, experiments simulating a wetland microcosm, in which three macrophyte wetland species (*Phragmites australis*, *Schoenoplectus validus*, and *Juncus ingens*) were tested, revealed the phytotoxicity of the treated wastewater for concentrations greater than 25%. Cress (*Lepidium sativum*) and onion (*Allium cepa*) were similarly sensitive to the treated wastewater (Arienzo, Christen, & Quayle, 2009b). Nevertheless, the same authors showed that this system, when combined with a previous sedimentation or aerobic process, could be used for small wineries located in rural areas, achieving a 72% COD removal rate (Arienzo, Christen, Quayle, & Di Stefano, 2009a).

Options such as lagooning and CWs may constitute interesting solutions for winery wastewater treatment if there has been the previous removal of suspended solids and if the local edaphoclimatic conditions are favorable (Rochard, 2017; Rodrigues et al., 2006).

Fernández et al. (2007) studied an activated sludge system model in which the COD removal efficiency ranged from 93% to 95%. The implementation of this system required the nutrient adjustment and the sludge production was about 0.3–0.6 g TSS g^{-1} COD (Jourjon, Racault, & Rochard, 2001; Racault, Cornet, & Vedrenne, 1998). Although this system was able to provide substantial removal yields, the sludge sedimentation was poor, and the long retention times were often problematic (Agustina et al., 2008; Artiga et al., 2007; Jourjon et al., 2001).

A long-term activated sludge system may provide a COD removal rate between 97% and 99% (Fumi, Parodi, Parodi, Silva, & Marchetti, 1995). In addition to a good removal efficiency, the system is simple (not very labor-intensive and dispensing with the need for skilled personnel), flexible and economical (with costs that were approximately one-half of those resulting from a conventional activated sludge system). This system can also operate by utilizing two reactors in series, the first one corresponding to the conventional treatment (F/M between 0.25 and 0.60 g COD g^{-1} VSS d^{-1}), with a hydraulic retention time of 3–5 days, and the second operating under extended aeration (F/M between 0.05 and 0.15 g COD g^{-1} VSS d^{-1}), with a hydraulic retention time of about 4–8 days. The overall treatment allows a COD removal efficiency of 96%–99% (Bolzonella et al., 2019; Jourjon et al., 2001; Racault et al., 1998; Rochard, Racault, & Canler, 2000).

According to Rodrigues et al. (2006), the sequencing batch reactor (SBR) is the most suitable technology for this type of industry. The system is characterized by a sequential operation, consisting of the periodic repetition of the operation cycle. Each stage of operation is under non-steady-state conditions, where the biomass retention within the system is performed by introducing a sedimentation phase under fully quiescent conditions, by combining different operations in a single tank. Trials conducted in a full-scale SBR showed the suitability of this system for winery wastewater treatment with a feed of 0.8 g COD L^{-1} d^{-1} and a ratio F/M of 0.25 g COD g^{-1} VSS d^{-1}, showing a COD removal efficiency of 93%–97% (Torrijos et al., 2004). Similar results were obtained by other authors (Wilderer, Irvine, & Goronszy, 2001). Further, Pirra, Arroja, and Capela (2004) showed that the SBR can operate with higher organic loads (5–18 g COD L^{-1} d^{-1}), achieving a COD removal of 95%, although some problems during the biomass sedimentation have been observed.

Winery wastewater often requires adjustment of the C/N/P ratio, as the carbon loads are usually higher. This high organic load also leads to oxygen transfer efficiency problems (López-Palau, Dosta, & Mata-Álvarez, 2009). Also, to optimize the sludge settling time, the formation of granules can be performed based on feast and famine periods (López-Palau et al., 2009). In fact, the high organic load promotes microbial growth and increases the biomass concentration, thus causing aeration problems in the reactor. Consequently, to achieve good performance, the aeration must be proportional to the COD load.

Another strategy to optimize the SBR cycle for total organic carbon and ammonia removal is based on dissolved oxygen (DO) control (Puig et al., 2006). The cycle consisted of three phases: reaction (under aerobic and anoxic conditions), settling, and discharge. During the aerobic phase, the set-point was DO 2.0 mg L^{-1}, with an On/Off control. Reactor optimization was performed on pH, DO, and oxygen uptake rate (OUR). This model allowed the ammonia valley to be detected during pH evolution and the end of nitrification, using the OUR plot. By identifying the bending points for the pH (ammonia valley) and the calculated OUR, it is possible to optimize the aerobic phase of the SBR cycle for organic matter and ammonia removal (Puig et al., 2006).

11.4.4 Membrane bioreactors

Membrane bioreactors (MBRs) represent an important technical option for wastewater treatment and reuse, being very compact and efficient systems for separation of suspended and colloidal matter (Delgado, Villarroel, González, & Morales, 2011; Valderrama et al., 2012). This technology is based on the combination of biological treatment, usually conventional activated sludge, with a membrane process of MF or UF. The membrane is a barrier that retains all particles, colloids, and microorganisms, providing complete disinfection of treated wastewater, enabling a high-quality effluent. The advantages of this system include the elimination of foaming and suspended solids in the effluent, the smaller footprint, the lower sludge production, and the improvement in treated wastewater quality (Artiga et al., 2005; Guglielmi, Andreottola, Foladori, & Ziglio, 2009). MBR treatment of winery wastewater has been shown to be highly effective, with COD removal rates higher than 97% (Artiga et al., 2005). According to Bolzonella et al. (2010), the MBR was able to handle hydraulic and organic loading peaks without changes in the reactor's performance. Even for an organic loading rate up to 2 g COD L^{-1} d^{-1}, the COD removal efficiency was over 95%, and nitrogen removal was also reported (Bolzonella et al., 2010). The growing interest in this system has led to its application in several full-scale wastewater treatment plants (Andreottola, Foladori, & Ziglio, 2009; Bolzonella et al., 2010; Delgado et al., 2011; Ferre, Trepin, Giménez, & Lluch, 2009). Also, a combined MBR–RO plant demonstrated the viability of integrating membrane technology with a bioreactor to enable significant water savings (Dolar et al., 2012; Vanossi & Durante, 2009).

11.4.5 Other bioreactors

There are some specific aerobic treatments systems that have been studied for winery wastewater; among them are the fixed bed biofilm reactor, the air microbubble bioreactor (AMBB) or the jet-loop reactor (JLR), using self-adapted microbial population, either free or immobilized (Eusebio et al., 2004; Eusébio et al., 2005; Oliveira, Queda, & Duarte, 2009; Petruccioli et al., 2000). In the JLR and AMBB (vertical reactors) the oxygen supply is performed by recirculation of the reactor effluent through a Venturi injector, which permits a good oxygen diffusion rate, overcoming the energetic costs associated with the aeration systems, with the advantage of requiring a small area. However, the high shear stress applied on the Venturi injector influences the composition of the microbial population (Eusébio et al., 2005; Oliveira & Duarte, 2011; Oliveira et al., 2009; Petruccioli et al., 2000), leading in some cases to settling sludge problems. These reactors achieve a COD removal efficiency up to 95%, for an applied organic load of 0.4–5.9 g COD L^{-1} d^{-1}. Vertical reactors appears to be among the most promising technologies, not only because of the economy of space but also because they are characterized by a good oxygen transfer capability and a high biological conversion rate (Duarte et al., 2004; Petruccioli et al., 2000; Xu, Zhou, Qu, Yang, & Liu, 2010).

Data from the literature point to several challenges related to winery wastewater treatment systems, namely, the wastewater's seasonality, volume and quality variations, and high oxygen transfer requirements as well as the presence of recalcitrant compounds. Criteria for the selection of the treatment technology include winery size, location, land and water availability, energy costs, and quality required in the treated wastewater. Moreover, the quantification and characterization of new fluxes from wine production need to be evaluated. Because of the variability and unique characteristics of these fluxes, the selection of the most adequate treatment system is of paramount importance. In fact, the most suitable method is to use processes that allow simultaneous treatment of the effluents and recovery of the bioproducts.

11.5 Membrane separation–based processes for biomolecules recovery from winery wastewater

Membrane separation processes such as MF, UF, nanofiltration (NF) and RO are unit operations in which a pressure gradient that is applied between the two sides of a permselective membrane promotes the physical separation of a feed stream into two others: retentate and permeate streams. The retentate stream comprises the components rejected by the membrane, while the permeate stream contains the components that get through the membrane. In these operations the separation mechanism in UF is governed mainly by steric hindrances, also known as sieving mechanism, in which the components that are smaller than the pores size or the MW cutoff (MWCO) of the membrane get through into the permeate stream and the larger ones remain in the retentate. Conventionally, MWCO determines the selectivity of a membrane and is related to the MW of a solute whose rejection is higher than 90% (Baker, 2012). However, other mechanisms, such as solute-solute and solute-membrane interactions, can also occur, interfering with the process. Table 11.4 shows the operating principles and applications of pressure-driven separation processes (de Pinho and Minhalma, 2019).

In fact, membrane separation processes, especially those driven by pressure gradient, have been proposed for the recovery of biomolecules from diverse wastewaters and extracts of

Table 11.4 Pressure-driven membrane processes: operating principles, and applications.

Separation process	Pore size or MWCO	Operating pressure (bar)	Range of application	Rejected material
Microfiltration	10–0.1 μm	0.1–1.0	Sterilization, Clarification	Particles, colloids, and bacteria
Ultrafiltration	350,000–1000 Da	0.5–8.0	Separation of macromolecular solutes	Proteins, polysaccharides, polyphenols, and other macromolecules
Nanofiltration	1000–200 Da	5–40	Separation of ions and small organic solutes	Glucose, fructose, amino acids, small organic solutes, and bivalent ions
Reverse osmosis	< 200 Da	20–100	Separation of ions and microsolutes	Ions and small organic solutes

Source: *Adapted from Cassano, A., Conidi, C., Ruby-Figueroa, R., & Castro-Muñoz, R. (2018). Nanofiltration and tight ultrafiltration membranes for the recovery of polyphenols from agro-food by-products. International Journal of Molecular Sciences, 19, 351. https://doi.org/10.3390/ijms19020351; de Pinho, M. N., Minhalma, M. (2019). Introduction in membrane technologies. In: C. M. Galanakis (Ed.), Separation of functional molecules in food by membrane technology (pp. 1–29). Chennai: Academic Press. https://doi.org/10.1016/B978-0-12–815056-6.00001-2; Habert, A. C., Borges, C. P., Nóbrega, R. (2006). Processos de separação por membranas. Editora E-papers, Rio de Janeiro.*

agroindustrial byproducts (Cassano, Conidi, & Ruby-Figueroa, 2014; Garcia-Castello, Cassano, Criscuoli, Conidi, & Drioli, 2010). Nevertheless, concerning the wine industry, the recovery of biomolecules by membrane technologies has been the focus mostly of studies involving solid byproducts, such as lees (Cassano et al., 2019; Giacobbo, Bernardes, & de Pinho, 2017a; Giacobbo, do Prado, Meneguzzi, Bernardes, & de Pinho, 2015) and grape pomace (Mora et al., 2019; Pereira et al., 2020), while studies addressing wastewater are still incipient. Despite that, winery wastewater (Table 11.1) can also be seen as a source for biomolecules recovery, and membrane technologies have emerged as a promising alternative for this purpose (Cassano, Conidi, Ruby-Figueroa, Castro-Muñoz, 2018). Therefore considering the precepts presented in studies with extracts from wine industry byproducts and also those with wastewater from other agroindustrial activities, it is possible to propose membrane-based processes that will be capable of recovering biomolecules from winery wastewater.

In a process for biomolecules recovery using a cascade of membrane technologies, MF or loose UF may be used to remove the components responsible for the turbidity of wastewater, such as suspended solids and colloids, resulting in a permeate stream containing phenolic compounds, organic acids, sugars, and minerals. Subsequently, the permeate of MF and loose UF can be concentrated by tight NF or RO, and the retentate can be used as food additives and pharmaceutical and cosmetic products, while the tight NF/RO permeate can be water for reuse (Fig. 11.5). Additionally, by adding a loose NF step between MF/loose UF and tight NF/RO, the MF/loose UF permeate could be fractionated, resulting in a concentrate that is rich in polyphenols and polysaccharides and a permeate containing monosaccharides (glucose, fructose, sucrose) and small

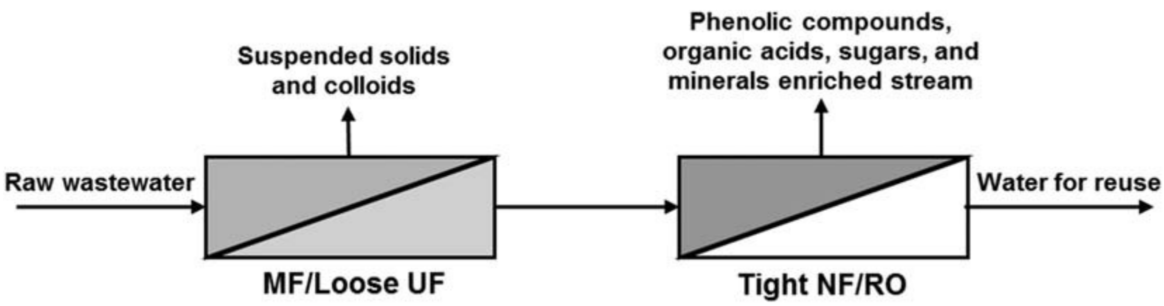

Figure 11.5 Conceptual framework of a cascade membrane process for the recovery of phenolic compounds from winery wastewater.

organic acids, which can then be subjected to the final concentration step via tight NF or RO to be used later as a food additive (Fig. 11.6). Moreover, the operations shown in Figs. 11.5 and 11.6 may be conducted in diafiltration mode, resulting in a greater recovery of target compounds in the MF/loose UF permeate stream and obtaining concentrate streams with a greater degree of purity in subsequent steps.

Alternatively, an extraction step can also be employed, aiming at the selective extraction of target compounds (e.g., phenolic compounds), and integrated with membrane technologies, which would act as a concentration step for the target compounds present in the extract (Fig. 11.7). In this sense, Cañadas et al. (2021) assessed chloride ammonium salts–based hydrophobic eutectic solvents as a greener alternative to organic solvents for the recovery of phenolic compounds from winery wastewater. By using trimethyloctylammonium chloride-DL-menthol, at a molar ratio of 1:2, and trimethyloctylammonium chloride-octanoic acid at a molar ratio of 1:1, the authors achieved recovery efficiencies of phenolic compounds up to 83.64% and 84.10%, respectively, from a winery wastewater. They performed the liquid-liquid extraction, at a solvent/wastewater ratio of 1, extraction time of 15 min under agitation at 500 rpm, and posterior centrifugation for 15 min at 3500 rpm, in which the phenolic compound–enriched fraction (less dense)

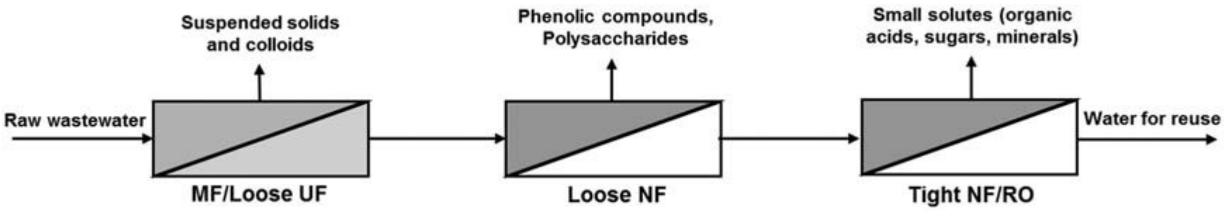

Figure 11.6 Conceptual framework of a cascade membrane process for the recovery and fractionation of biomolecules from winery wastewater.

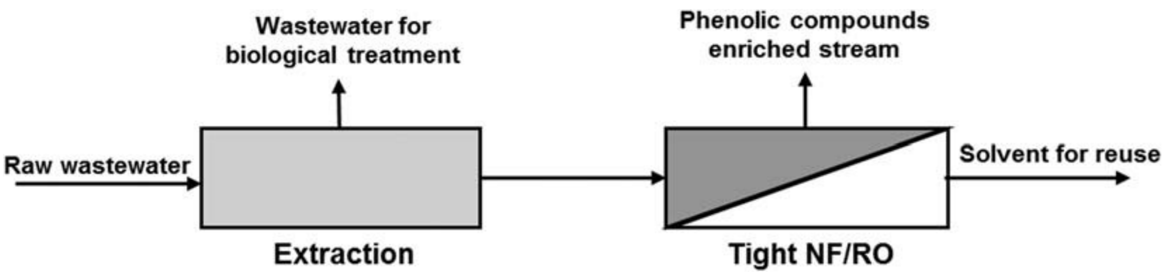

Figure 11.7 Conceptual framework of an integrated membrane process for the selective recovery of phenolic compounds from winery wastewater.

was recovered on the top and the water-rich fraction remained at the bottom.

Indeed, the recovery of biomolecules is considered a promising alternative for the valorization of byproducts of the wine industry, as in addition to providing an improvement in environmental performance, owing to the reduction of the pollutant load of wastewater, it also facilitates its treatment in downstream stages, mainly owing to the removal of phenolic compounds, which have been considered toxic to microorganisms present in biological treatments (Mosse et al., 2013). In this way, it is possible to obtain treated wastewater with a better quality, which allows its reuse in agriculture. Reports have pointed out that polyphenols have also shown phytotoxic effects on plants and soil microorganisms (Mosse, Patti, Christen, & Cavagnaro, 2010; Shilpi et al., 2018), being recommended the removal of these compounds prior to using the winery wastewater for irrigation purposes. Furthermore, membrane technologies also play an important role in terms of water and wastewater reuse, which will be discussed in the next section.

11.6 Wastewater reuse

Water is becoming increasingly scarce, mainly in regions that suffer droughts and have experienced increased levels of irrigation (European Commission, 2012). Actually, irrigation in the vineyard is a practice of increasing use in many regions. The Mediterranean region is identified as one of the most prominent hotspots in future climate change projections, and projected climate changes will have a direct impact on water resources and crop irrigation requirements (Costa et al., 2020; Diffenbaugh & Giorgi, 2012; Giorgi, 2006).

In 2006 the World Health Organization (WHO) has published guidelines for the safe use of wastewater in agriculture (WHO, 2006). However, these guidelines do not address the concerns of the wine industry, as the main focus is on heavy metals and human pathogens. Similarly, the European regulations are more directed to microbiological parameters, since their focus has been on the reuse of domestic wastewater (Brissaud, 2008). Microbiological parameters have great relevance from a public health point of view, but they must be considered only in treatment systems receiving domestic wastewater. In this sense, wineries that plan to reuse treated wastewater for irrigation must segregate flows and treat industrial and domestic wastewater separately, since the domestic effluent flow is about 100-fold to

1000-fold smaller. This avoids the need for the disinfection step in the total flow generated, reducing treatment costs.

The analytical parameters of the treated winery wastewater are usually in agreement with these guidelines. However, sodium and potassium compounds from sanitizing agents and measured as sodium adsorption ratio and potassium adsorption ratio are typically higher than the allowed parametric value (Laurenson, Bolan, Smith, & McCarthy, 2010; Oliveira et al., 2009). These substances are a concern when the treated wastewater is to be reused for irrigation, since conventional treatment processes do not significantly reduce salt concentrations (Hirzel, Steenwerth, Parikh, & Oberholster, 2017; Mosse, Patti, Smernik, Christen, & Cavagnaro, 2012). A study conducted in South Africa revealed that in regions with low rainfall, irrigation with winery wastewater would lead to the spread of cations, increasing soil salinity (Mulidzi, Clarke, & Myburgh, 2020). Studies on the long-term effects of the use of untreated winery wastewater in the irrigation process showed a negative impact on soil structural stability (Liang, Rengasamy, Smernik, & Mosley, 2021; Mosse et al., 2012, 2013). When the treated wastewater was applied, no significant differences in nitrogen and carbon cycling were detected in the short-term analysis (Mosse et al., 2012, 2013).

Considering the treatment processes that were identified in Section 11.4 and constraints highlighted previously, the most relevant processes applied for the reuse of treated wastewater will be outlined (Fig. 11.8).

Mitigation strategies should be adopted at the winery level. The reuse of the wash water to exhaustion, at which point it ceases to be effective as a bitartrate dissolving agent, could be very useful in preventing contamination. Also, it is advisable to replace disinfectants and cleaning agents by ozone (Cullen & Norton, 2012; Guillen, Kechinski, & Manfroi, 2010; Pascual, Llorca, & Canut, 2007). It is probable that the ozone treatment will allow decreases in both conductivity and COD, thus contributing to compliance with the legal limits for beneficial crop irrigation (Cullen & Norton, 2012; Lucas et al., 2009). Also, the reduction in

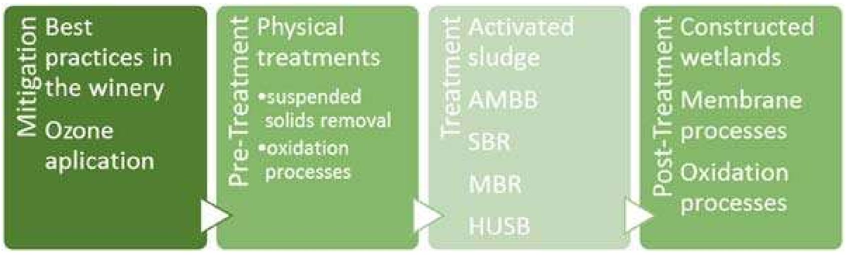

Figure 11.8 Treatment strategy for winery wastewater reuse, based on reviewed literature.

COD can be achieved by screening out solids larger than 0.5–1.0 mm with basket screens as a pretreatment and by reducing the contact period between solids and wastewater. The reuse of wastewater in irrigation is limited to a maximum concentration of organic matter of 500 mg L^{-1}, expressed as biochemical oxygen demand (BOD). The WHO guidelines further state that the application of wastewater with a BOD between 110 and 400 mg L^{-1} can be beneficial to cultures (WHO, 2006).

MBR technology was applied to wine wastewater and compared with conventional activated sludge systems, revealing similar COD removal rates. The MBR was very effective in removing suspended solids and microbiological contamination, producing treated wastewater that met the requirements for reuse in agricultural practices (Valderrama et al., 2012) and all kinds of industrial applications (Töre & Sesler, 2021). In addition, treated wastewater from SBR was used for irrigation purposes, and no negative impacts were detected (Mosse et al., 2012). Regarding COD removal, a treatment system consisting of hydrolytic upflow sludge blanket (HUSB) and CWs allowed reuse of the treated wastewater for irrigation as long as the influent did not exceed COD values higher than 2000 mg L^{-1} (Pascual et al., 2021). Although the treatment system treated industrial and domestic flow in combination, the study did not evaluate the removal of *Escherichia coli*. From the results it can be concluded that the combination of CWs and HUSB can be adapted to treat the high and variable organic load from the wine industry while producing water that is suitable for agricultural irrigation (Pascual et al., 2021). Given the relevance of the salt content in these wastewaters, it becomes urgent to evaluate the efficiency of wine wastewater treatment systems against these parameters. Studies conducted in CWs have shown the potential application of halophytes in removing the salt content from wine wastewater, namely, Na^{+} and K^{+} (Mader, Holtman, & Welz, 2022; Matinzadeh, Akhani, Abedi, & Palacio, 2019). However, the composition and concentration of contaminants should be known prior to selection of the halophytic plant species.

The potential ecological risk associated with the application of treated wastewater cannot be assessed by chemical characterization alone, as this does not allow an assessment of the possible combined effects of the different contaminants mixed together as well as an evaluation of their bioavailability. One of the methods that is used is phytotoxicity assessment through germination and growth of seedlings (*L. sativum*) to understand the ability of plants to compete and survive in their environment (APHA, 2005). Therefore to evaluate the potential phytotoxicity of wastewater after treatment, due to possible synergistic and deleterious effects of various water contaminants, bioassays should be envisaged, such as cress as a plant indicator (Fjällborg, Ahlberg, Nilsson, & Dave, 2005; Mekki, Dhouib, & Sayadi, 2007; Mosse et al., 2010; Muyen, Moore, & Wrigley, 2011; Oliveira et al., 2009; Stutte, Eraso, Anderson, & Hickey, 2006; van Gestel et al., 2001).

Studies conducted with the AMBB showed that the diluted treated wastewater was suitable for irrigation. These conclusions were based on physicochemical analyses and phytotoxicity tests (Oliveira et al., 2009).

As posttreatment, various methods such as ion exchange and RO can be used, which are effective in removing salt in winery wastewaters. The ion-exchange process is characterized by the exchange of ions between the solution to be treated and an immobilized resin; it is a widespread process in wastewater treatment systems for the removal of ions, including ammonium (Jorgensen & Weatherley, 2003), chromium (Rengaraj et al., 2001) and boron (Kabay et al., 2004). Ion-exchange resins can be generated synthetically or by using natural zeolites (clinoptolites), which are also effective in removing cationic contaminants (Pitcher, Slade, & Ward, 2004). This natural mineral shows high and moderate specificity for K^+ and Na^+, respectively, suggesting its applicability to the removal of these inorganic ions from winery wastewater.

RO is the most suitable technology for salt removal and water purification, being used in wastewater treatment for potable water production. Although RO is very effective, pretreatment by MF (Durham et al., 2001) or UF (van Hoof, Hashim, & Kordes, 1999) is necessary to prevent biofouling of the RO membrane and ensure that the RO system will operate at design capacity (Jacob et al., 2010). Another key issue in membrane treatment is the high energy demand for operation, although pretreatment stages have been shown to reduce the energy requirements (Pearce, 2007). Although RO is appropriated to wastewater treatment (Dolar et al., 2012; Ioannou et al., 2013; Jacob et al., 2010; Zhang et al., 2011), the economic and environmental costs should be taken into account (Mosse et al., 2011). Nevertheless, if a membrane system is used to recover biomolecules and other products, in addition to water (Section 11.5), this can be compensated for.

The selection of the appropriate treatment system that allows compliance with legal requirements for reuse and at the same time prevents negative impacts on the ecosystem is crucial. To valorize the value-added compounds of the wine

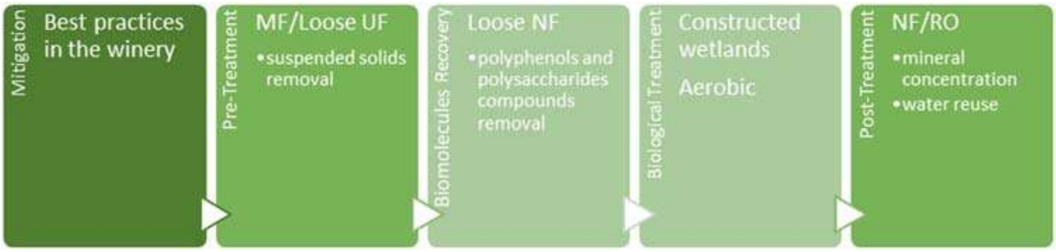

Figure 11.9 Conceptual approach for value-added compound recovery and wastewater reuse in a winery.

wastewater and to produce high-quality treated wastewater, we propose a sequence of separation and treatment processes (Fig. 11.9).

This management model will enable the recovery of value-added compounds and water saving, with obvious benefits for the environment and no apparent risk to public health.

11.7 Conclusions and future trends

The wine industry is one of the most important food and beverage industries in the world and also one with great polluting potential due to the high volume and load of wastewater that is generated. Therefore considering average values of wastewater generation, 2.2 L per liter of wine produced (Oliveira et al., 2019; Welz et al., 2016), the average wine production of the last decade, 270 MhL (OIV, 2021), and moderate values for the concentration of phenolic compounds in wastewater, 5 mg L^{-1} (see Table 11.1), it is estimated that the wine industry annually generates about 54 billion liters of wastewater containing about 297,000 kg of phenolic compounds. Currently, these wastewaters are seen as environmental liabilities that must be treated before being discharged into the environment when in fact they are important and inexpensive sources of raw materials such as phenolic compounds and water. Although this is a rough and simplistic calculation, it serves to give an idea of the amount of resources wasted annually in wastewater from the wine sector, since these phenolic compounds are not recovered and only a small fraction of this water is reused for irrigation.

In this regard, membrane technologies have been shown to be effective in the recovery, purification, and concentration of phenolic compounds and other biomolecules from wastewater and agroindustrial waste extracts, emerging as a promising alternative for the recovery and valorization of biomolecules from winery wastewater. Furthermore, membrane technologies can be integrated with other wastewater treatment methods, resulting in high-quality treated effluent for industrial or agricultural reuse, meeting the concepts of a circular economy.

It is important to highlight that actions of this magnitude, associated with those that already exist in the context of recovering value-added compounds from solid wastes, tend to bring a traditional winery closer to the concept of biorefinery, in which losses are minimized and resources are used to the full, providing achievements in economic, environmental, and social areas, as preconized in the Sustainable Development Goals of the United Nations for 2030.

List of acronyms

ABB air bubble column bioreactor
AMBB air microbubble bioreactor
AOP advanced oxidation process
BOD biochemical oxygen demand
COD chemical oxygen demand
CW constructed wetland
DO dissolved oxygen
ECCF evapoconcentration to fractional condensation (abbreviation from French, *evapoconcentration à condensation fractionnée*)
FAO Food and Agriculture Organization
FBB fluidized-bed bioreactor
FBBR fixed-bed biofilm reactor
F/M food-to-microorganism ratio
FU functional unit
GAE gallic acid equivalent
GE glucose equivalent
HUSB hydrolytic upflow sludge blanket
JLR jet-loop reactor
NF nanofiltration
MBR membrane bioreactor
MF microfiltration
MW molecular weight
MWCO molecular weight cutoff
OIV International Organization of Vine and Wine (abbreviation from French, *Organisation internationale de la vigne et du vin*)
OUR oxygen uptake rate
RBC rotating biological contactor
RO reverse osmosis
SBBR sequencing batch biofilm reactor
SBR sequencing batch reactor
TSS total suspended solids
UF ultrafiltration
VFA volatile fatty acid
WHO World Health Organization

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