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Review

Recovery of Value-Added Compounds from Winery Wastewater: A Review and Bibliometric Analysis

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Abstract: Value-Added Compounds (VACs) are molecules that have attracted great attention in the literature regarding their potential for integration into existing processes or products. In Winery Wastewater (WW), VACs are valuable because of their antioxidant characteristics. Furthermore, integrated systems for recovery of these molecules and treatment of WW can be a powerful strategy towards an environmentally efficient way of wine production. Therefore, a bibliometric analysis was conducted to establish the status and trace research trends on that topic. The analysis investigated the Web of Science database from 1953–2023, where the software VOSviewer® and R were used. Secondly, an extensive literature review was carried out on VACs recovery from WW by membrane process, which was identified as a promising strategy. As a result, the historical evolution of publications has a growing behavior while citations take time to grow. The most productive authors, journals, and countries were analyzed, with Spain being the leading country in publications on that topic. Lastly, keyword mapping revealed the relevance of "circular economy" and "biorefinery" as knowledge transfer concepts where both traditional and emergent technologies are connected towards the achievement of sustainable development goals. The recovery of phenolic compounds from wine production through membrane processes stands out as a promising technology. Different recovery approaches using membrane processing were assessed in the review. Studies could achieve up to 100% phenolic compound and polysaccharide rejections and also addressed the possibility of fractioning these compounds.

Keywords: winery wastewater; value-added compounds; phenolic compounds; membrane processes; circular economy

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1. Introduction

Wine has been produced since 7000 b.c. in the region where Georgia is located nowadays [1]. Since then, wine manufacturing has been industrialized and is now considered an important social-economic product included in the Mediterranean Diet. In fact, moderate wine intake may have health benefits such as helping diabetic type 2 control [2], anti-inflammatory effects in ulcerative patients, and gut and oral microbiome improvement [3], the antimicrobial effect [4], the anticarcinogenic effect [5], and increasing levels of HDL [6]. Some of these benefits may come from phenolic compounds, which are a class with many molecules that are secondary metabolic products of plants. These compounds have been associated with high antioxidant activity with positive health effects [7].

On the other hand, the wine industry is still associated with negative effects on natural ecosystems. Wine production is highly water intensive for plant growth while generating 2.20 L of wastewater and 200 kg of solid pomace per litter of wine produced (not considering pruning) [8]. The International Organization of Vine and Wine registered a world wine production of 525 MhL (171 MhL in Europe), which represented 856 million euros (393 million euros in Europe) as exportation income. This wine production generated about

1.1 GhL (0.11 Gm^3) of wastewater and 29 Gkg of solid waste worldwide, which will be treated (376 MhL of wastewater and 10 Gkg for Europe) in 2020.

The by-products of the wine industry have raised great attention for further valorization instead of wasting renewable resources in a circular economy strategy and as a positive contribution to environmental sustainability [9]. Indeed, these by-products are rich in phenolic compounds, which are composed of more than 8000 molecules that can be subdivided into many other subcategories such as phenolic acids (e.g., gallic acid, vanillic acid, ferulic acid), flavonoids (e.g., cyanidin, malvidin, catechin, quercetin), tannins, which are phenolic polymer compounds capable of precipitating proteins (e.g., tannic acid), and stilbenes (e.g., resveratrol) within others. Phenolic compounds can be recovered through several solid-liquid extraction recovery strategies from the stalk [10], grape marc [11], and lees [12]. Wine lees can also be valorized as a protein source [13], while polysaccharides can also be recovered from grape marc [14]. After the first step of valorization, biomass can be used to further recover energy, for example, through anaerobic digestion. In this case, sludge remains at the end, which can be stabilized by composting in an integrated biorefinery approach [9]. The liquid phase can also be valorized for phenolic compound recovery using membrane processes [15] or adsorption processes [16]. After phenolic compounds are recovered from wastewater, reclaimed water for irrigation may be obtained. Water recirculation would also contribute to freshwater availability and, thus, to sustainable development goal 6 (one of the 17 SDGs from the United Nations). That way, process integration and optimization play an important role in the development of solutions for fractioning by-products and generating new products, while solid and liquid waste are valorized instead of wasted.

Wine industry waste valorization has been reviewed for specific themes such as the production of bioproducts with vine pruning waste [17], vine canes as phenolic compound sources [18], grape pomace polymeric pigment extraction [19], grape marc utilization for value-added products [11], winery wastewater used for irrigation [20], waste to energy in the wine industry [21], and renewable energies utilization in the wine industry [22]. As far as the authors know, there is only one meta-analysis on that topic. In that study, the author was capable of analyzing quantitative data from several articles about using grape pomace as an additive for food products to assess the most promising alternatives [23]. There is no bibliometric analysis on that scenario, and that represents a lack in the literature about quantitative analyses of authors, journals, publications, and citations in the countries, qualitative analyses of the evolution of trend topics, and the use of keywords.

The objective of this research is to contribute to covering this gap in the literature by providing a two-step approach. The first aim is to broadly analyze how waste and effluents from the winemaking process have been managed/valued/wasted through bibliometric analysis tools and to understand whether circular economy approaches have been used in practice. This part may act as a guide for authors to quickly identify the most productive authors, journals, and countries that can facilitate knowledge exchange and increase social value through partnership formation. Secondly, a case study is presented to demonstrate how wine wastewater can be valorized through membrane processes. This step reviewed several articles that optimize value-added compounds recovered from wine industry wastewater that aim to catalyze economic value growth using the most promising approaches. Finally, future trends were established based on all the literature information reviewed.

2. Methodology

The methodology of the bibliometric analysis was based on the strategy depicted in Figure 1. Briefly, the Web of Science database was chosen once it had been broadly accepted in the scientific community as a tool for acquiring articles from several different areas of knowledge [24]. After that, a research equation was established to request the maximum number of papers that refer to wastes (in general) in the wine-making process, from vine management to wine bottling. The research equation then was TS = ((vine* OR wine*) AND (waste*) NOT (vinegar*)) which means that a search was completed for articles that both contain vine(s), vineyard(s), wine(s) or winery(ies), and waste(s) or wastewater(s) but not vinegar(s) written in the title, abstract, or keywords. The data timespan was 1953–2023 and was collected on 11 December 2022 and stored in excel and text format for later analysis using Excel®, VOSviewer®, and Bibliometrix® package in the R language. Finally, inside this restricted database, the bibliometric analysis was conducted to respond to the following research questions (RQ):

- RQ1: How has productivity and research quality evolved in the field and which research area leads publication?
- RQ2: Who are the most productive journals, authors, institutions, countries, and financial agents?
- RQ3: What are wine production wastes and how they are being managed?
- RQ4: What are future trends in the field?

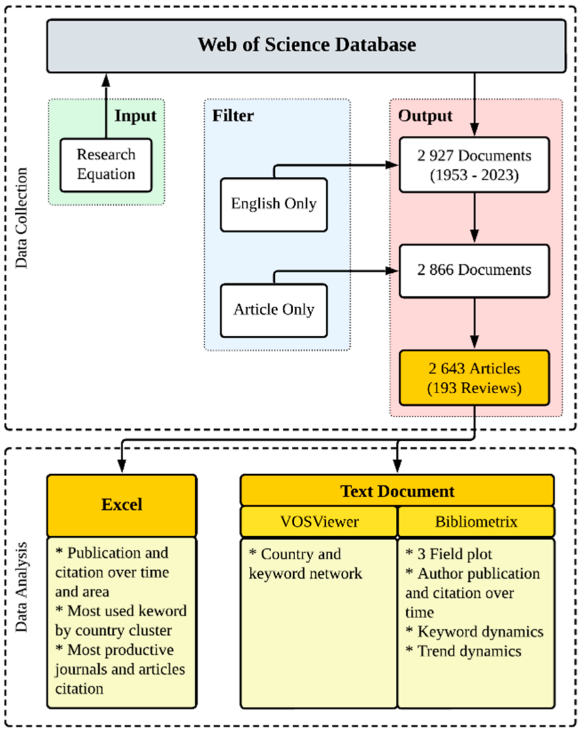


Figure 1. Diagram of bibliometric analysis strategy of this work.

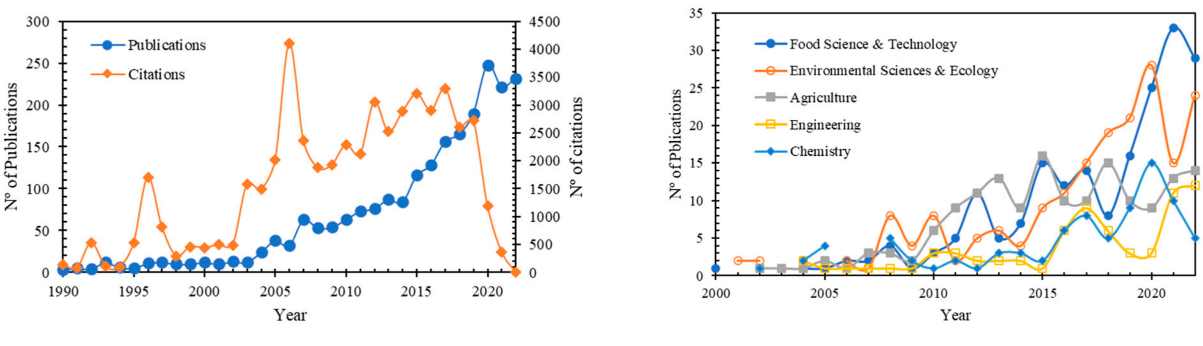
After that, the membrane technology was chosen as a case study to perform an extensive literature review within the articles collected for the general bibliometric analysis. This was accomplished considering the membrane processes as one of the most promising for the recovery of phenolic compounds from winery wastewater [25–34]. This part aimed to assess the status, actual capacity, advantages and disadvantages, and future trends for the recovery of phenolic compounds from wine industry wastewater.

3. Bibliometric Analysis

3.1. Historical Notes and Productivity Evolution

The first article published in the winery waste area was entitled "Disposal of California Winery Wastes", in 1953, highlighting the lack of an alternative to manage 200 gallons of vinasse from the previous wine production campaign [35]. Furthermore, this article suggests strategies for treating winery waste involving integrated technologies that combine neutralization, anaerobic digestion, and biological oxidation, which demonstrated a reduction in the biological oxygen demand (BOD) of vinasse sediments of around 70%.

Furthermore, scientific contributions were small until the 1990s, as can be seen in Figure 2a, despite the entire movement for sustainability and industrial ecology initiated in the late 1960s. In fact, with the increase in internet accessibility in the 1990s, the number of publications grew until the 2000s. From then on, the world agenda for sustainability was already consolidated after the Rio Earth Summit (1992) and the Kyoto Protocol (1998), and thus publications, and citations in this area have followed a growing trend until 2022. A slight increase in the yearly growth rate of publications comparing 2010–2015 with 2015–2020 can also be observed, and probably the Paris Agreement (2015) played an important role in that result. On the other hand, citations revealed a persistent downward pattern in the last 4 years, which can probably be explained by the time delay that most published articles need to have to accumulate a satisfactory number of citations, as observed again in Section 3.2.



(**a**) (**b**)

Figure 2. (**a**) Publications and citations over time; (**b**) publications over time by area.

The yearly growth pattern of the most productive areas is shown in Figure 2b. Food Science & Technology is nowadays the most active one, and this can be associated with raising interest in the health benefits of value-added compounds present in wine by-products [36]. For instance, solid waste in winemaking is a polyphenol-rich component and can represent up to 20% w/w of grape (dry basis) [8]. The combination of these two factors increased the number of publications in this area with the development of new food products such as cookies [37], functional muffins [38], fortified yoghurt [39], and flour [40]. However, there is still a lack of literature considering the risks associated with the daily intake of these products, health safety, and legislation. Grape pomace was identified as a potential ingredient for the human diet while highlighting the importance of arsenic level control on these products, as they observed a high level of this heavy metal in their samples [41].

3.2. Contribution by Authors, Countries, and Journals

In Figure 3, the co-authorship network is shown considering the 57 contributing countries. Among these countries, the top ten most productive (Publications/Citations) were: Spain (618/16987), Italy (318/9735), Portugal (196/4507), China (174/4365), France (156/4293), USA (155/5165), Brazil (142/4379), Greece (118/3997), India (105/3103), and Australia (94/2886). The Cluster Analysis demonstrates how countries collaborate internationally, allocating countries that collaborate closely and in the same cluster, while link strength is related to the intensity of collaboration. There are 4 clusters in Figure 3 (marked with different colors: Spain, Italy, Greece, and the USA), where a great knowledge exchange can be observed. The most productive continents are Europe, the Americas, and Asia, while Spain stands out among the countries with the most significant contribution to this topic.

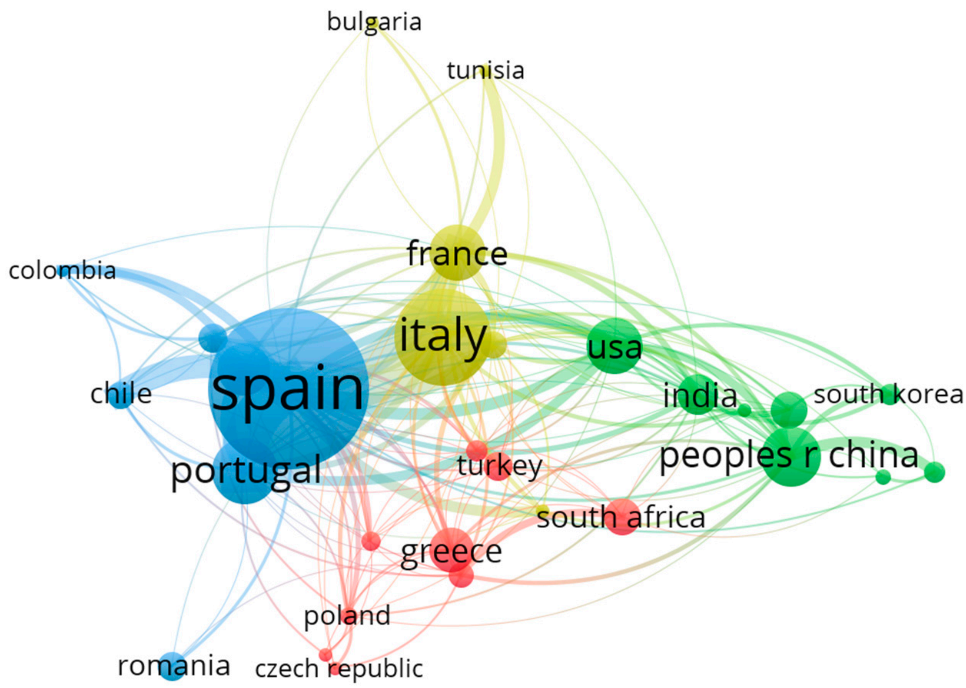


Figure 3. Countries co-authorship network.

Cluster 3 (Spain) is the most productive, as can be seen through the keywords in Table 1. It is a sustainable development-oriented joint that has developed work within new strategies for the valorization of wine industry waste. Especially focused on the recovery of bioactive compounds, the most relevant article in this cluster exploits a new pathway for resveratrol biosynthesis with a *saccharomyces cerevisiae* strain, using biomass generated by vine maintenance and wine processing [42]. Furthermore, in that research was estimated through the developed strategy and mass balances that it would be possible to produce approximately 8 kg of resveratrol per hectare of vineyard. Other studies toward sustainable development have been conducted in that cluster, for instance, the bioproduction of succinic acid using vine and grape biomass [43], the use of vine pruning to optimize organoleptic properties of wine [44], biobased packaging using grape canes [45], and green energy production [46,47].

The USA has played an important role in connecting the development of western and eastern knowledge. As can be seen in Figure 3, the USA is between Asia and European/American clusters, which indicates that, in addition to cooperating a lot with the eastern world, the USA also has direct connections with Europe and American countries on that topic. Cluster 2 (USA) is characterized by geographically distant countries and very specific topics within the parts, as the co-occurrence of keywords was revealed to be very low. The USA demonstrated a higher interest in governance topics, over the last 5 years, such as economic and environmental impact estimation of winemaking in the USA [48] and techno-economic analysis [49]. On the other hand, China is focused on industrial ecology to optimize the process with a zero-discharge policy. Therefore, studies have been carried out on industrial metabolism analysis and a waste-to-energy biorefinery approach [50–53].

Figure 4 is a three-field plot where the most productive authors—countries—keywords are plotted. Most productive authors are affiliated with Spain, which is followed by China, the USA, Portugal, and Italy. On the other hand, countries are interested in a very wide range of topics, with a higher tendency for most used keywords such as “polyphenols”, “grape pomace”, phenolic compounds”, “anaerobic digestion”, “wine”, “biochar”, and “antioxidant activity”.

Table 1. Keyword occurrence within clusters map.

Keyword	Cluster			
	1 (Greece)	2 (USA)	3 (Spain)	4 (Italy)
activated carbon	0	0	10	0
active packaging	0	2	0	0
adsorption	0	0	12	0
anaerobic digestion	6	2	31	0
anthocyanins	0	0	10	8
antioxidant	5	0	31	19
bioactive compounds	7	0	12	0
biochar	5	2	9	7
biomass	0	2	10	0
biorefinery	8	0	12	13
by-products	0	0	9	9
chemical oxygen demand	8	0	0	0
circular economy	0	2	18	14
compost	5	0	0	0
constructed wetland	5	0	0	0
cynodon spp.	0	2	0	0
environmental contamination	0	2	0	0
fermentation	0	3	0	0
grape marc	0	0	12	6
grape pomace	15	0	21	16
irrigation	0	0	0	6
phenolic compounds	0	0	36	0
polyphenols	11	0	29	21
resveratrol	5	0	0	0
sewage sludge	0	0	11	0
sustainability	5	0	13	9
valorization	5	0	0	0
vine shoots	0	0	9	0
vitis vinifera	0	0	8	0
wastewater	0	0	9	0
wastewater treatment	0	3	0	0
wine	0	3	0	0
winery wastewater	0	0	27	9

Note: The intensity of the colours indicates the higher number of occurrences of the keyword in that cluster.

The productivity in terms of publications and citations over time of the most prominent authors in the literature is represented in Figure 5. This plot reveals that many authors began to develop work on that topic by 2004, although one of them started earlier, in 1997. The most productive author (Dominguez JM) has extensively studied strategies for the valorization of vine trimming shoots, while other topics have been addressed, such as the production of ferulic acid esterases [54], biogas [55], vanillin, vanillyl alcohol, and vanillic acid [56], and biosurfactants [57]. The second and third most productive authors (Bustamente MA and Moral R, respectively) worked with biodegradation and sludge management strategies [58,59]. The first to publish on that topic currently works with the production of biogas [60], bio-hydrogen [61], and eco-friendly ways of valorizing winery wastewater [62]. The newest author to address that topic (Solera R.) has been working on anaerobic co-digestion strategies [63], performing techno-economic analysis [64], and modeling bioreactors [65].

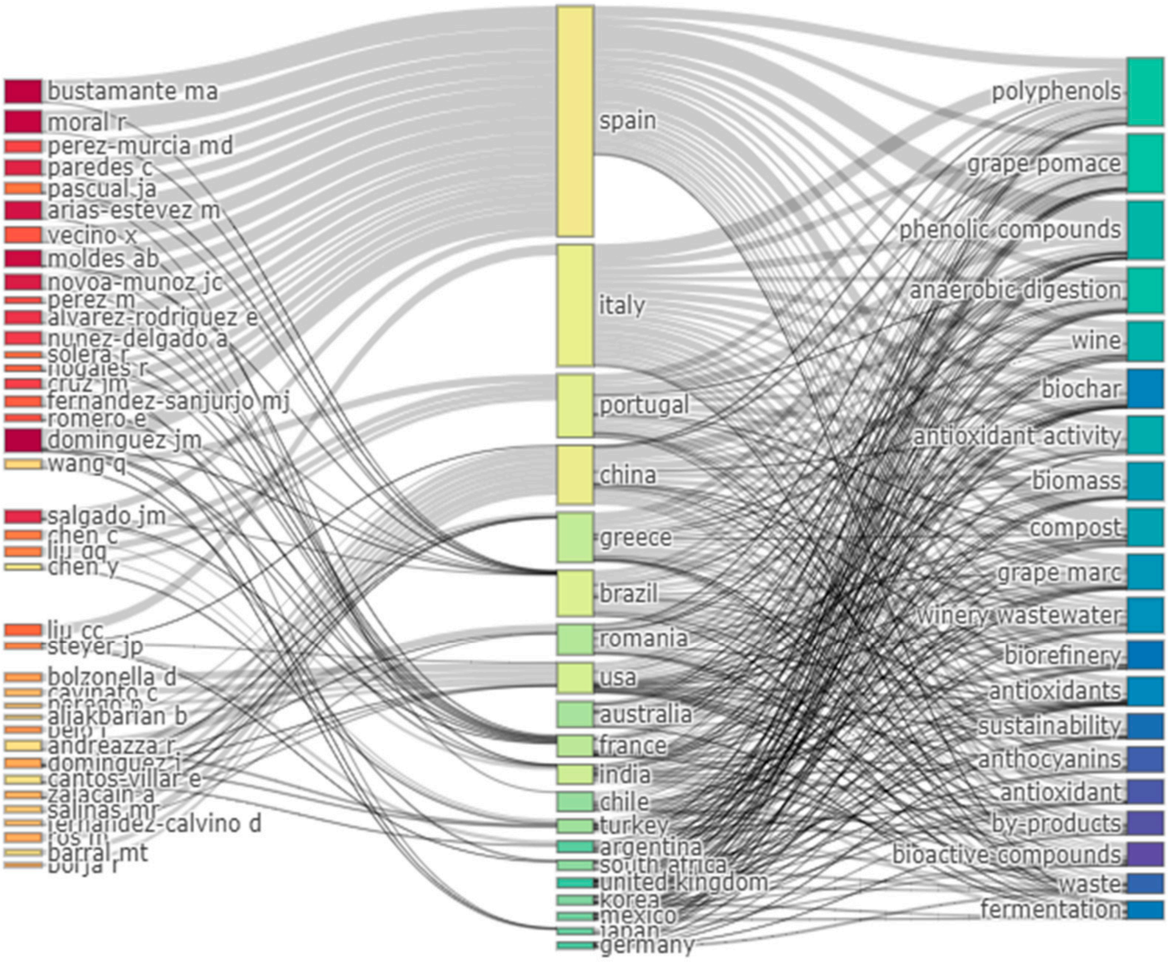


Figure 4. Authors on the left side, countries in the middle, and keywords on the right side in a 3-field plot (the minimum considered publication per author was 1 and the 40 most impactful authors were listed).

The most productive journals in terms of publications are concatenated in Table 2, along with their productivity metrics as impact factor and h-index. The impact factor is a measure of the average number of citations received by articles in each journal, while the h-index is a metric that considers both the number of articles published by an author and the number of citations those articles have received. Table 2 shows that the most cited articles in the most productive journals are at least 3 years old, which means that citations might take some time to rise. This may explain the behavior of citations in Figure 2a. The most productive journal was Water Science and Technology, with 120 publications, and the most cited article in this journal was a concatenation of available technologies in anaerobic digestion. The author analyzed several technologies, such as free cells or flocs, anaerobic granules, or biofilms on fixed support or mobile support [66]. The journal with the highest impact factor was Bioresource Technology, and the most cited article evaluated the differences in hydrothermal carbonization and torrefaction of grape pomace. In this study, [67] found that hydrothermal carbonization produced coal with greater energy densification and greater combustible reactivity than the method of roasting, which showed a product with more aromatic compounds. It was also verified that the residual water from hydrothermal carbonization had antioxidant activity due to the presence of phenolic compounds.

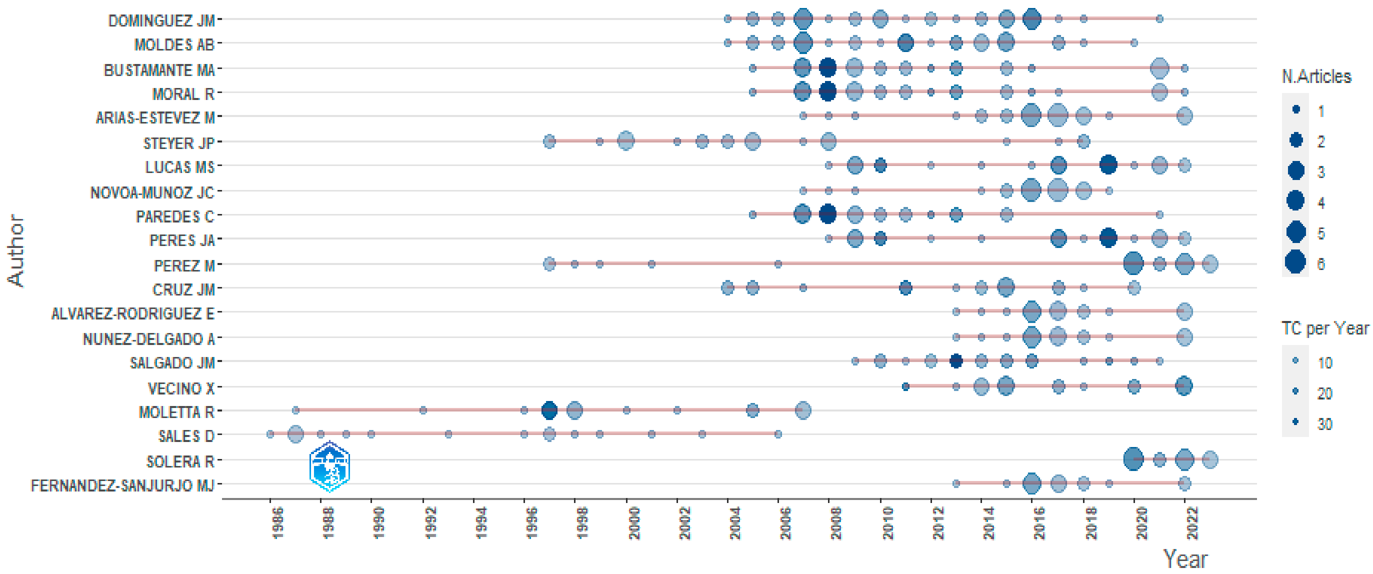


Figure 5. Authors' publication and citations over time.

The most cited article among the top 10 most productive journals did not even coincide with the journal with the highest impact factor or with the most productive. Phenolic compounds were extracted from grape pomace in the Greek wine industry with different solvents, contact times, solvent-solid ratios, and extraction pH [68]. In that study, it was experimentally demonstrated that the ethanol:water mixture in the 1:1 ratio was the most efficient in terms of the amount of phenolic compounds extracted, but the pure ethanol had the greatest antioxidant activity, which inhibited 93.3% of oxidation compared with 62.5% of the ethanol:water 1:1 mixture.

A novel way of interpreting "waste" in the wine industry was suggested [69]. In that study, it was stated that not recycling waste would become expressively expensive and that recycling should be completed to reduce costs. Furthermore, it was pointed out that certain opportunities had already been studied. Nowadays, the valorization of waste in the wine industry is an inevitability coupled with a great market opportunity.

Table 2. Most productive journals in terms of publications with their metrics and the most cited article.

Journal (Publication/Citation)	Impact Factor	*h*-Index	Title of the Most Cited Article	Citation	Ref.
Water science and technology (120/2564)	2.43	145	Winery and distillery wastewater treatment by anaerobic digestion	125	[66]
Bioresource technology (64/2813)	11.889	317	Hydrothermal carbonization and torrefaction of grape pomace: A comparative evaluation	181	[67]
Journal of cleaner production (50/2213)	11.072	232	Recovery of organic wastes in the Spanish wine industry. Technical, economic and environmental analyses of the composting process	170	[70]

Table 2. *Cont.*

Journal (Publication/Citation)	Impact Factor	*h*-Index	Title of the Most Cited Article	Citation	Ref.
Journal of agricultural and food chemistry (42/1504)	5.895	310	New flavanol derivatives from grape (Vitis vinifera) byproducts. Antioxidant aminoethylthio-flavan-3-ol conjugates from a polymeric waste fraction used as a source of flavanols	122	[71]
Science of the total environment (42/923)	10.753	275	Constructed wetlands for winery wastewater treatment: A comparative Life Cycle Assessment	49	[72]
Food chemistry (37/1576)	9.231	281	On the extraction and antioxidant activity of phenolic compounds from winery wastes	251	[68]
Waste management (35/2230)	8.816	182	Valorization of winery waste vs. the costs of not recycling	218	[69]
Industrial crops and products (33/1275)	6.449	141	Grape cane waste as a source of trans-resveratrol and trans-viniferin: High-value phytochemicals with medicinal and anti-phytopathogenic applications	102	[73]
Molecules (32/375)	4.927	171	Bio-Based Compounds from Grape Seeds: A Biorefinery Approach	60	[74]
Journal of environmental management (31/541)	8.91	196	Evolution of organic matter during the mesophilic composting of lignocellulosic winery wastes	99	[75]

3.3. Keywords Analysis and Future Trends

The analysis of keyword networks allows not only the identification of the most important keywords in the field but also their correlation within their proximity and clusters. In Figure 6, it is possible to identify four clusters that are very well defined. The "anaerobic digestion" cluster is intimately related to "sewage sludge", "wastewater treatment", and "methane" production because of their proximity. Although this cluster connects with polyphenols, terms such as "wine lees", "wine", and "grape marc" are more related to the topic of phenolic compounds. Therefore, the wastewater topic in the industry area is associated with anaerobic digestion, kinetics, adsorption, sludge composting, and waste reuse for irrigation, with great concern for chemical oxygen demand quality parameters. However, the constructed wetland has been an emerging topic for winery wastewater management, and it has been demonstrated to integrate electricity production and phenol removal [76] and the production of water for irrigation reuse [77].

Solid residues are mainly associated with biochar, pyrolysis, and composting, which are strategies for the management of grape marc, pomace, shoots, and wine lees after further valorization through the extraction of value-added compounds, for example. On the right side of Figure 6, there is the polyphenols cluster highly associated with grape, grape pomace, phenolic compounds, antioxidant, antioxidant activity, anthocyanins, extraction, and optimization techniques. It is noticeable the distance between this emergent cluster and the well-established wine industry's traditional waste and waste treatment strategies. Finally, the "circular economy" and "biorefinery" keyword clusters emerge as knowledge transfer agents. The circular economy is an emerging concept on that topic that represents the transition from a linear economy to a renewable and sustainable approach to wine production. This implies smarter use of bioresources to minimize waste generation and recirculate as many by-products as possible to minimize net-carbon emissions [78]. Biorefinery, on the other hand, is a specific topic for the development of innovative solutions to further process waste to produce new products with aggregated value (e.g., food, animal food, biofertilizer, biopesticide, biocosmetics, biopolymers, biofuels) [79]. The aim of biorefinery inserted in a circular economy-based society is to make the world independent of fossil fuels by making use of renewable bioresources only. Both concepts connect traditional wine industry waste management techniques with new trends in the market for the recovery of added value compounds in a biorefinery approach towards a circular economy through innovation, making use of life cycle assessment tools to achieve sustainable development goals.

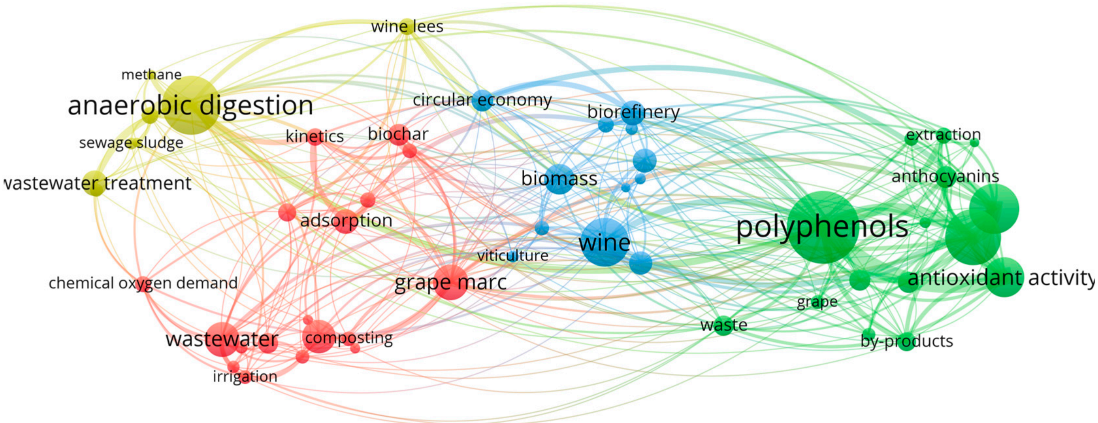


Figure 6. Keywords network.

Keyword thematic evolution is also an important tool to assess trends over time and for informed decision-making in research. There is a traditional core in winery waste treatment over time, which is shown in Figure 7. This core is represented by "anaerobic digestion", which has always been studied and recently integrated into anaerobic co-digestion, as discussed before in this work. Composting is another intergenerational applied technology. Nowadays, there is still great interest in studying these technologies that are described in other works, such as winery wastewater treatment [80] and integrated anaerobic treatment and composting in a biorefinery approach [81].

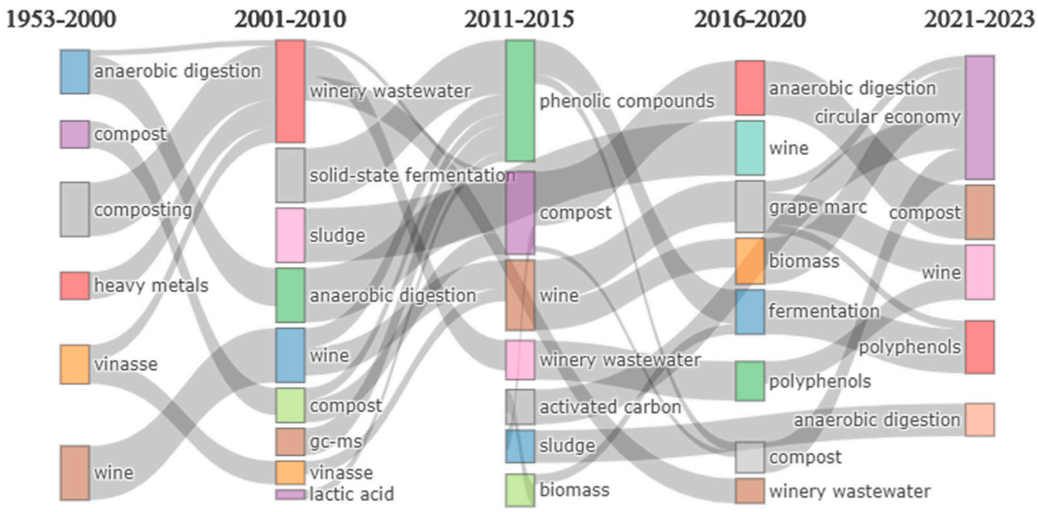


Figure 7. Keywords dynamics plot over 4-time slices.

The studies related to the valorization of wine waste into lactic acid production emerge in the 2000s [82–86] and the phenolic compounds in 2010 [87–93]. Indeed, it has been demonstrated over time that wine industry waste can be fractionated into a diverse range of compounds, such as succinic acid [94], xanthan [95], polyhydroxyalkanoates [96], biohydrogen and poly- β -hydroxybutyrate [97], bioethanol [98], and methane [99]. However, there is still a lack of information on pilot or demonstration scale fractionation units associated with a clear and established assessment of a wide range of strategies. Economic analysis and life cycle assessment are scarce, which would be relevant to facilitate decision-making for private and public productive institutions.

Keyword usage within time is shown in Figure 8. Thus, it is possible to identify historical trends for further comprehension of future perspectives in that area. For instance, lactic acid was a 5-year trend, and the keyword was used 50 times, but in 2012 it became obsolete. Since the 2000s, many keywords have also become obsolete as new trends emerge. This fact is probably due to the saturation of studies on the subject, the technology has been developed enough for the application, or new technologies have emerged. In the early 2000s, research topics were based on oxidation treatments, adsorption, anaerobic digestion, and vinasse and sludge management. Later, with the increased interest in feedstock fractioning and by-product valorization strategies toward sustainable development, the interest in solid and liquid waste returned. Nowadays, circular economy leads the trending topics, and future steps are probably based on innovation to identify new opportunities, systematic economic analysis of existing strategies for by-product valorization, comparative analysis within existing technologies in terms of environmental impact, process optimization, pilot demonstration, and the development of plants on an industrial scale.

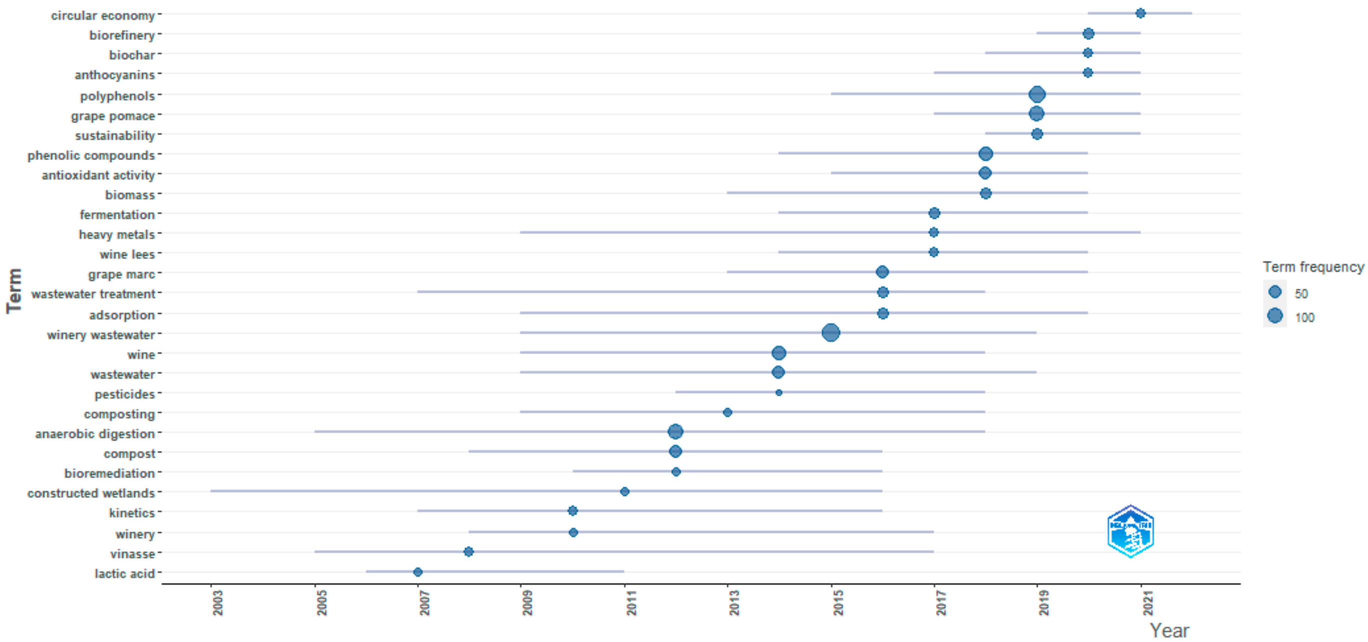


Figure 8. Trends plot described as keywords frequency over time.

4. Case Study—Phenolic Compounds Recovery from Wine Production Waste through Membrane Processes

4.1. Composition of Waste from Wine Production

The wine effluent is generated whenever it is necessary to use fresh water to wash some equipment in a winery. In this way, large volumes of effluent are generated at various stages of the process, including when washing the grapes and equipment and in tanks after racking. Thus, this effluent is characterized by large variability in its physical and chemical properties, given the different stages in which the effluent is generated, the intrinsic variability of the compounds present in the grape, the order of operations units, and the use of products to clean the equipment [100].

Typically, the winery effluent contains a large organic load, intense color, turbidity, and suspended solids. These factors associated with its seasonality can impair the treatment based on the traditional route of aerobic processes [101]. A comprehensive review was carried out with a total of 43 articles to establish a range of values for the main physical and chemical properties that characterize the wine effluent, as described in Table 3 [102]. These literary values were also confronted with the legal limits imposed by the Portuguese legislation, DL 119/19, of August 21, which concerns the production of water for reuse from wastewater depending on its final use. Data in Table 3 shows that the winery effluent may be characterized by high values of chemical oxygen demand (COD) and biochemical oxygen demand (BOD_5), associated with phenolic compounds and sugars. Moreover, there is low availability of nutrients such as nitrogen and phosphorus, which can also pose a challenge to the traditional treatment route of effluents. Finally, the pH can be well above the range of the winemaking environment (2.5–4.5) due to the use of cleaning agents based on sodium hydroxide.

Table 3. Range of values for some properties of winery effluents in the literature (adapted from [102] and [9]).

Property	Units	Interval			Legal Limit DL 119/19		
Chemical Oxygen Demand (COD)	mg/L	320	-	49,105		-	
Biochemical Oxygen Demand (BOD_5)	mg/L	203	-	22,418	<10	-	40
Total Organic Carbon (TOC)	mg/L	41	-	7363		-	
pH	-	2.5	-	12.9		-	
Electrical conductivity (EC)	mS/cm	1.1	-	5.6		-	
Total Solids (TS)	mg/L	748	-	18,332	<10	-	60
Total Volatile Solids (TVS)	mg/L	661	-	12,385		-	
Suspended Solids (SS)	mg/L	66	-	8600		-	
Total Phosphorus (PT)	mg/L	2.1	-	280	5	-	
Total Nitrogen (TN)	mg/L	10	-	415	15	-	
Total Phenolic Compounds (TPh)	mg/L	0.51	-	1450		-	
Turbidity	NTU		-		<5	-	
E. coli	cfu/100 mL		-		<10	-	10,000
Intestinal parasite eggs	N°/L		-		<1	-	
P	mg/L	3.30	-	188.3			
Fe	mg/L	1	-	77		-	
Mg	mg/L	1.96	-	1170		-	
Ca	mg/L	12	-	2203		-	
Mn	mg/L	200	-	1740		-	
As	mg/L	0.05	-	3260		-	
Zn	mg/L	12	-	1400		-	
Ni	mg/L	500	-	650		-	

Considering the load of pollutants in the effluent, there is a need for treatment before the water returns to the ecosystem. The approach to treatment may be directed toward the recovery of value-added compounds and the reuse of water. Phenolic compounds are the class of greatest interest for valorizing this type of effluent, although there is some interest in the literature regarding polysaccharides [103,104]. This class of compounds can be recovered through membrane processes (Section 3.3), adsorption [105], liquid-liquid extraction (L/L) [106], and integrated processes [45]. Finally, water recovery can be subsequently integrated into the recovery processes of phenolic compounds from the perspective of effluent fractionation.

4.2. Recovery of Phenolic Compounds by Membrane Processes

Literature studies have demonstrated a good performance of the membrane processes for the recovery of phenolic compounds. Within this objective, along with the framing of the wine effluent, 9 articles were selected to establish the frontiers of the work carried out in this area in recent years, as shown in Table 4. However, besides the conventional mode of operation with membranes in closed or semicontinuous systems, it is worth highlighting the alternative systems.

A liquid membrane extractor was developed to analyze the membrane in emulsion and microemulsion forms [107]. In this study, it was demonstrated that the membrane in its microemulsion form managed to extract 64% of the phenolic compounds initially present, while the emulsion extracted only 46%. The design of new membranes aimed at removing phenols is also an area of interest. An integrated process with membranes and adsorption was developed by synthesizing a cellulose acetate membrane impregnated with alumina to maximize the rejection of phenols [108]. A membrane with a 120 Da molecular weight cut-off limit (MWCO) was impregnated with 20% (w/w) of resin in the form of nanospheres with a diameter of about 62 nm, in which 91% of the phenols were rejected. In this study, it was demonstrated the strong dependence of rejection on the pH of the feed was demonstrated. In fact, pH 4 to 6 and 9 led to a rejection ranging from 3 to 16 and 91%, respectively.

There is also the strategy of encapsulating the phenolic compounds before filtration. [34] and [109] studied the effect of a load of a surfactant to make micelles with phenols at various concentrations, pH, concentration, and feed matrix on the rejection of phenols in ultrafiltration processes. It was demonstrated that lower rejections for phenolic compounds are obtained with a standard solution of gallic acid (R = 60%) when compared with real effluent (R = 78%), which demonstrates the influence of matrix complexity on the rejection of this type of compound [34]. For the standard solution, the permeability of gallic acid is facilitated because it has a low molecular mass and there are no other compounds to be simultaneously retained. On the contrary, in real effluents, there is a diversity of compounds, which accelerates and intensifies the concentration polarization that triggers a shielding effect, increasing the rejection of phenols.

The strong effect of pH in processes with membranes has also been demonstrated; when varying the pH from 3 to 9, the rejection of phenols varied from 95 to 15% [109]. The influence of pH, especially in systems with low MWCO membranes, is due to the magnitude of electrostatic interactions (much greater) between molecules and membranes concerning the influence of pressure [110]. Thus, when changing the pH, the charge of the medium is altered: at low pH, the medium is positively charged, and the phenolic compounds tend to have a greater non-protonated fraction (neutral). When the pH of the medium increases, some protonated groups of phenols lose their positive charge and become negatively charged. Consequently, this variation in the charge of the molecules alters their entire interaction with the medium, which, in the case of membranes, is also affected by the pH due to the change in their surface charge.

From Table 4, it is possible to identify some key variables in conventional membrane processes. Even though membrane processes are not only applied to winery effluents but are also used for the concentration of phenolic compounds after the extraction of winery solid waste. The pre-treatment of the sample to be filtered has been accomplished in terms of passages through cloth filters [111], microfiltration [32], centrifugation [27], or a combination of these [30]. Although previous studies also made use of sedimentation [103]. The choice between these methods depends on the objective. In the case of using cloth filters or microfiltration, the suspended solids of the raw sample will be lost, while in centrifugation (or sedimentation) they can be valorized, which makes this process more interesting from the point of view of the fractionation of the effluent.

Table 4. Studies published in the considered database study membrane filtration of wine effluents or extracts derived from wine by-products. Details of sample preparation, data regarding the membrane, operating conditions, rejections obtained for phenolic compounds and sugars, and identified phenolic compounds were recorded.

Operation	Membrane *	Conditions of Operation **	Rejection ***	Identified Compounds	Ref.
Centrifugation/wine lees/10 min at 4000 RPM/UF and NC/crossflow	UF—Flat; CA; 35; - NF1—Flat; CA; -; 4.63 NF2—Flat; CA; -; 4.42 NF3—Flat; CA; -; 8.34 NF4—Flat; PA; 0.20–0.40; 3.75	UF—0.5 a 2.5; 25; 0.55 NF—5 a 20; 25; 0.8	UF—40; 90 NF1—72; 99 NF2—70; 97 NF3—65; 98 NF4—95; 96	(−)-Epicatechin, (−)-Epigallocatechin gallate, (+)-Catechin, Caffeic acid, Chlorogenic acid, Coutaric acid, Fertaric acid, Gallic acid, Protocatechuic acid, rans-caftaric acid, Syringic acid, Vanillic acid, Cyanidin-3-*O*-glucoside, Cis-Caftaric, Malvidin-3-*O*-glucoside, Peonidin-3-*O*-glucoside, Petunidin-3-*O*-glucoside and Quercetin aglycone	[27]
Bagasse extract/Nylon cloth/microfiltered/NF/cross flow	NF1—Flat; PA TFC; 0.15 a 0.30; - NF2—Flat PA TFC; 0.30 a 0.50; - NF3—Flat PA TFC; 0.60 a 0.80; -	NF—25; 30; 245.5	NF1—97; 100 NF2—93; 80 NF3—91; 72	Malvidin 3-(acetyl)-glucoside, Malvidin 3-(coumaroyl)-glucoside and Malvidin-3-*O*-glucoside	[30]
Bagasse extract/3mm Nylon cloth/NF/cross flow	NF1—Flat; CA; -; 4.63 NF2—Flat; CA; -; 4.42 NF3—Flat; CA; -; 8.34 NF4—Flat; PA; 0.20–0.40; 3.75	NF—20; 25; 0.8	NF1—79; 86 NF2—80; 100 NF3—73; 20 NF4—98; 73		[111]
Wine lees extract/microfiltered/UF and NF/dead-end filtration	UF—Flat; Fluor composite; 1; 22.3 NF1—Flat; PA aromatic; 0.15; 13.4 NF2—Flat; PA cross-linked; 0.15 a 0.30; 4.5	UF—10; 25; - NF—24; 25; -	UF—10; 19 NF1—56; 54 NF2—40; 40	Gallic Acid, Syringic Acid and (+)-Catechin	[32]
Lees extract/Nylon cloth/NF/dead-end filtration	NF1—Flat; PES; 0.3 a 0.4; 0.73 NF2—Flat; PES; 1;3 NF3—Flat; PA; 1; 7.19	NF—20; 25; -	NF1—37; 63 NF2—36; 56 NF3—25; 55	Resveratrol	[31]
Wine lees/microfiltered/UF and NF/crossflow	UF—Flat; Fluor composite; 1; 8.8 NF—Flat; PPP TFC; 0.20 a 0.30; 8.7	UF—5; 25; 2.5 NF—5; 25; 2.5	UF—50; 81 NF—90; 100		[112]
Bagasse extract/microfiltered/UF or NF/crossflow or dead-end filtration	UF1—Flat; PVDF; 150; >285 UF2—Flat; PES; 150; >285 NF1—Flat; PES; 1 a 1.2; >200 NF2—Flat; PES; 0.5 a 0.6; >40 NF3—Flat; PA; 0.2 a 0.4; - NF4—Flat; PA; 0.2 a 0.4; -	UF *dead-end*—5; 35; - NF *dead-end*—8; 35; - NF *cross-flow*—4; 35;	Dead-end UF1—16; - UF2—42; - NF1—70; - NF2—57; - NF3—22; - NF4—40; - Crossflow NF1—79; - NF2—92; - NF4—75; -	cyanidin 3-*O*-(6″-acetyl)-glucoside, cyanidin 3-*O*-(6″-p-coumaryl)-glucoside, cyanidin 3-*O*-glycoside, delphinidin 3-*O*-(6″-acetyl))-glycoside, delphinidin 3-*O*-glycoside, malvidin 3-*O*-(6″-acetyl)-glycoside, malvidin 3-*O*-(6″-cis-p-coumaroyl)-glycoside, malvidin 3-*O*-(6″-trans-p-coumaroyl)-glucoside, malvidin 3-*O*-glycoside, peonidin 3-*O*-(6″-p-coumaryl)-glucoside, peonidin 3-*O*-glycoside, peonidin3-*O*-(6″-acetyl)-glucoside, petunidin 3-*O*-(6″-acetyl)-glucoside, petunidin 3-*O*-(6″-p-coumaryl)-glucoside and petunidin 3-*O*-glycoside	[113]

Table 4. *Cont.*

Operation	Membrane *	Conditions of Operation **	Rejection ***	Identified Compounds	Ref.
Bagasse extract/microfiltered/NF/crossflow.	NF1—Flat; Fluor composite; 1; 245 NF2—Flat; PA; 1; 184 NF3—Flat; PS; 0.5 a 1; - NF4—Flat; PA; 0.3 a 0.6; - NF5—Flat; PA; 0.15 a 0.3; - NF6—Flat; PA; 0.2 a 0.4; - NF7—Flat; PA; 0.15 a 0.3; - NF8—Flat; PA; 0.125 a 0.3; - NF9—Flat; PA; 0.125 a 0.2; -	NF—10 a 30; 20 a 40; -	NF1—46.0; 8.1 NF2—43.1; 24.1 NF3—64.6; 47.0 NF4—74.7; 32.7 NF5—82.1; 64.2 NF6—95.1; 69.2 NF7—97.8; 99.6 NF8—100.0; 99.5 NF9—100.0; 99.1	Catechin, quercetin and taxifolin	[114]
Wine lees/centrifuged/15 min at 8000 RPM/UF and NF/crossflow	UF—Tubular; PVDF; -; 11.8 NF—Tubular; PA TFC; 0.20; 7.3	UF e NF—2; 25; -	UF—28; - NF—79; -	Citric Acid, DL-Lactic Acid, DL-Malic Acid, Ferulic Acid, L-Tartaric Acid, Tartaric Acid, Epicatechin and Quercetin	[26]

* (Geometry; Material; *MWCO*/kDa; L_p/L/m^2 h bar). ** (PTM/bar; T/°C; Feed inlet/L/min). *** (TPH/%; Carbohydrate/%).

The polymeric materials of the membranes used were limited to cellulose acetate (CA), polyamide (PA), polyvinylidene fluoride (PVDF), polyimide (PI), and polysulfone (PS). According to [115], CA membranes are hydrophilic and cheap but have limitations regarding pH and temperature and can be degraded by microorganisms. On the other hand, the PS base returns a membrane with resistance to pH and temperature. PVDF membranes are less commonly used but highly recommended for operation with non-polar solvents and oxidative environments. Finally, PI has greater use in gas separation given its high thermal, chemical, and mechanical resistance [116].

There is still no consensus in the literature about the optimal arrangement of the recovery process of phenolic compounds from winery effluents. Thus, processes in series and parallel were found with several recirculation options. However, the type of membrane geometry used was essentially flat.

The rejection of phenolic compounds and sugars from the selected studies was in a range of 10–50% and 19–90% for UF and 22–100% and 8.1–100% for NF. It is noteworthy that these ranges are based on 6 UF and 34 NF studies, and the only conclusion that can be considered is the fact that nanofiltration achieves higher rejection values for the two types of compounds analyzed. Moreover, membrane processes are not only based on sieving mechanisms, and more complex types of pre-treatments and interactions such as electrostatics shall be taken into consideration. However, it was possible to identify the studies that stood out in the highest removals of phenolic compounds, as is the case with 100% [117] that made the comparative study of 9 NF membranes with different materials and MWCO in a range of 125–1000 Da for various operating conditions. Sugars are more easily rejected because they have a higher molecular mass, so in the analysis of these compounds, it is important to define whether the fractionation objective between phenols and sugars is relevant or not for the application. If the fractionation is relevant, the studies by [27] and [111] demonstrated that it is possible to have very different rejections between phenolic compounds and sugars in both UF and NF, with 40%/90% and 73%/20%, respectively.

In general, the literature already comprises a lot of studies on the composition of the membranes, MWCO, and operating pressure. In this sense, there is still a need for studies that unify all the information developed in the last five years. Furthermore, there is a lack of research that focuses on winery wastewater, makes use of tubular membranes, and determines the influence of pH in the ultrafiltration processes themselves and their effluent pre-treatment units.

5. Conclusions Remarks

In this study, it was possible to assess the current state of the art regarding waste valorization in the wine industry. It was noticeable that there is great potential in winery wastes (solids and liquids) that are yet not used in an integrated process. The leading strategy to manage the wine industry waste is the traditional anaerobic digestion that treats the waste instead of valorizing the by-product rich in bioresources (bioactive compounds and biomass). There is a higher interest in the theme in Europe, but there are great players worldwide, such as Australia, China, the United States, and Brazil. Although there are several attempts to integrate the valorization process in the wine industry using extraction, adsorption, and membrane processes, there is still a lack of pilot studies and demonstrations. The chosen technology shall be scalable, and further scalability studies using actual industry should be completed.

The membrane process for phenolic compound recovery from winery waste was deeply assessed. It is preferred technology to directly valorize winery wastewater or to integrate it with the extraction process. It can be fractionated by groups (proteins, carbohydrates, and phenolic compounds), while the operational cost must be assessed. Furthermore, the scale-up of membrane processes is not complicated, which is a critical step within other emergent technologies.

To summarize, the concern with environmental issues has catalyzed research toward sustainable development goals. Energy transition and a sustainable circular bio-based economy shall be achieved. It was demonstrated that in the wine industry, the keywords "circular economy" and "biorefinery" are knowledge transfer agents capable of accelerating the integration of emerging technologies with the traditional winemaking process.

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