sustainability

MDPI

Article

Technoeconomic Analysis of the Recovery of Phenols from Olive Mill Wastewater through Membrane Filtration and Resin Adsorption/Desorption

Dimitris P. Zagklis [1,*], Costas S. Papageorgiou [1] and Christakis A. Paraskeva [1,2]

[1] Laboratory of Transport Phenomena and Physicochemical Hydrodynamics (LTPPH), Department of Chemical Engineering, University of Patras, GR26504 Patras, Greece; papag011@umn.edu (C.S.P.); takisp@chemeng.upatras.gr (C.A.P.)
[2] Institute of Chemical Engineering Sciences, FORTH/ICE-HT, GR26504 Patras, Greece
* Correspondence: dimitriszag@chemeng.upatras.gr

Abstract: Olive mill wastewater is an important agro-industrial waste with no established treatment method. The authors have developed a phenol separation method that could potentially cover the treatment cost of the waste. The purpose of this study was to identify any economic hotspots in the process, the operational cost and examine the margin of profit for such a process. The equipment cost was scaled for different treatment capacities and then used to estimate the fixed capital investment and the yearly operational cost. The highest purchased equipment cost was identified for the membrane filtration system, while the cost for resin replacement was identified as the highest operational cost. The lifespan of the resin used in the adsorption step was identified as an economic hot spot for the process, with the phenols separation cost ranging from 0.84 to 13.6 €/g of phenols for a resin lifespan of 5–100 adsorption/desorption cycles. The lifespan of the resin proved to be the single most important aspect that determines the phenols separation cost. The price range that was calculated for the product of the process is very promising because of the typical value of antioxidants and the low concentration of phenols that are needed for food supplements and cosmetics.

Keywords: phenols; membrane filtration; resin adsorption; olive mill wastewater; agro-industrial wastes

check for updates

Citation: Zagklis, D.P.; Papageorgiou, C.S.; Paraskeva, C.A. Technoeconomic Analysis of the Recovery of Phenols from Olive Mill Wastewater through Membrane Filtration and Resin Adsorption/Desorption. *Sustainability* **2021**, *13*, 2376. https://doi.org/10.3390/su13042376

Academic Editor: Sean Clark

Received: 30 January 2021
Accepted: 18 February 2021
Published: 23 February 2021

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https://creativecommons.org/licenses/by/4.0/).

1. Introduction

Agro-industrial activity is a major source of waste production. These wastes can be in liquid or solid form and contain important phytochemical substances with high added value. An example of such phytochemicals are phenolic compounds characterized by their high antioxidant activity [1]. These compounds are present in most plant materials, with different functions, such as a defense against herbivores or as a response to environmental stress [2], and as a result, in many agro-industrial wastes. An important agro-industrial waste rich in phenols is olive mill wastewater (OMW).

OMW is prominently produced around the Mediterranean Sea, where most of the major olive oil producing countries are located [3]. It is produced through the three-phase olive oil extraction method with the addition of large quantities of water prior to centrifugation. During the last decade, an effort has been made to convert most of the three-phase olive mills that produce olive oil, olive kernels and OMW, to a two-phase process that only produces olive oil and alperujo, a paste-like material that contains both the olive kernels and the minced olives after oil extraction. The two-phase extraction process is considered more environmentally friendly because of the smaller volume of the waste produced, but still, there are problems to be faced [4], such as the air pollution during the removal of moisture before the extraction of kernel oil with hexane. In Greece, after the conversion of the three-phase olive mills to two-phase, the kernel oil producing factories had to treat enormous amounts of alperujo. Because of the small span of time in which

olive oil is produced, these quantities were concentrated in a three to four month period. As a result, most kernel oil factories stored the alperujo prior to its treatment, typically for several weeks, leading to the production of odors [5]. These odors were then spread, alongside other particles and water vapor when the alperujo was dried in the kernel oil production process, deteriorating the air quality several kilometers around the facilities [6].

OMW phenols have high antioxidant potential and are investigated as additives for several different consumable products [7,8] and cosmetic applications [9,10]. The great interest in OMW phenols presents an opportunity for high-added-value products that can cover the treatment cost of the waste and present a significant margin for profit.

Throughout the years, several treatment methods have been proposed for the treatment of OMW. Because of the high concentrations of phenol present in olive mill effluents, the biological treatment of OMW can be severely impeded [11]. This is usually tackled by dilution of the waste through the addition of water or its co-digestion with another waste. Examples of such processes are the co-digestion of OMW with liquid poultry manure [12], abattoir wastewater [13], cattle manure and slurry [14], and different food wastes [15,16].

Advanced oxidation processes have also been tested for the treatment of OMW. This type of methods is typically targeted at reducing the phenolic content of the waste and enabling post-treatment with other methods (usually biological). These methods can be Fenton oxidation [17,18], photocatalytic methods [19,20], and electrocoagulation [21,22].

Physicochemical methods are amongst the most used treatment methods because they are not inhibited by the presence of phenols and are typically cheaper than advanced oxidation processes. Membrane filtration has been widely tested, but certain limitations regarding rapid membrane fouling have been reported when raw OMW is used as feed [23]. Apart from the reduction of the organic load, membrane processes have been used for the recovery of the phenolic content of the OMW [24,25]. Adsorption has also been used for the selective separation of OMW phenols typical with resins as adsorbents [26,27], but other materials have been used as well, like activated carbon [28].

The number of publications related to OMW has increased rapidly during the last thirty years, with most of the published papers originating from the main olive oil producing countries (Figure 1). The most rapid increase in published papers was recorded from 2000 to 2010, while during 2020 alone, more than 100 papers were published, indicating the ongoing nature of the OMW treatment problem. The country of origin of most of these publications was Spain, closely followed by Italy and then Greece. The order of countries of origin of OMW-related publications appears like their ranking as olive oil production countries, indicating that the serious environmental problems that are caused by OMW act as motivation for the scientific communities of these regions for scientific research.

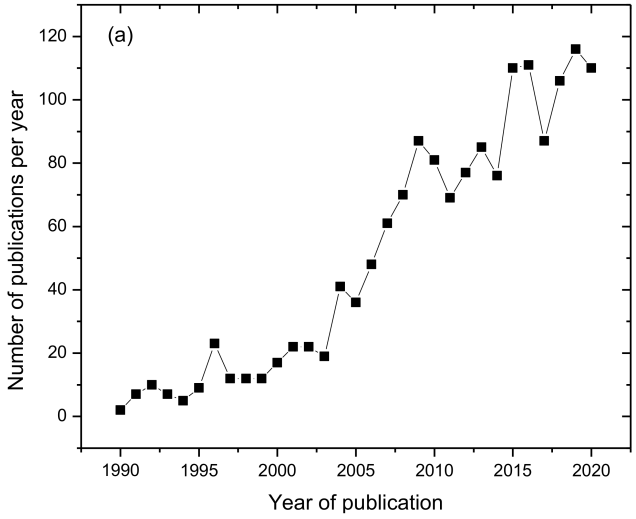


Figure 1. *Cont.*

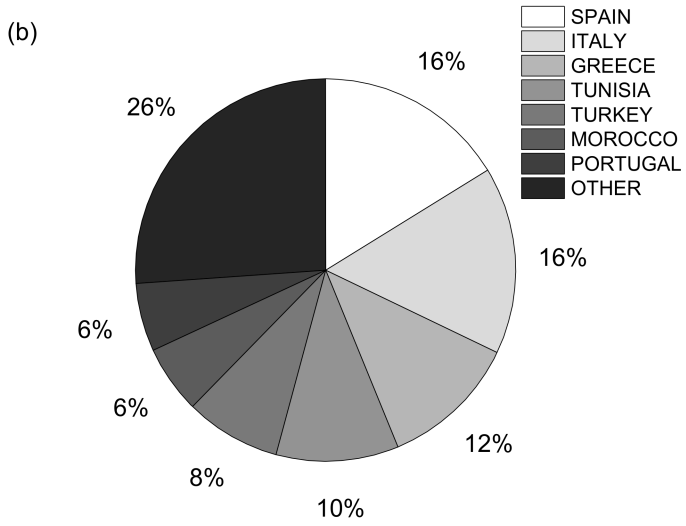


Figure 1. (**a**) Number of publications on the topic of olive mill wastewater per year and (**b**) distribution of these publications according to countries of origin [29].

The large number of publications and the different treatment methods examined for OMW indicate that no definitive solution has been found for this problem. This can be attributed to the technical difficulties presented during the treatment of OMW (high organic and solid content, phenols, etc.), and the cost of its treatment. In this scope, the authors have developed a separation method for the recovery of phenolic compounds from agro-industrial wastes using membrane technology and resin adsorption/desorption that can potentially cover the treatment cost. The same method can be applied to both solid and liquid waste by adding an extraction step for solid matrixes. The results of the implementation of the process to olive mill wastewater [30], grape marc [31], and olive leaves [32] have been published by the authors, followed by a preliminary design of a treatment plant implementing the method [33]. A preliminary technoeconomic analysis of the membrane filtration part of the process has also been reported by the authors [34], for an initial concept of the separation technique. The scope of this work is the technoeconomic analysis of the process, including the resin step, and identification of economic hotspots. The sustainable management of olive oil industry effluents is a matter of utmost importance for the producing countries, but a treatment method that is not economically viable is unlikely to be implemented by the stakeholders. The authors believe that this work can motivate the implementation of OMW treatment methods that allow the extraction of value from a waste stream and alleviate the environmental burden of olive oil production processes.

2. Materials and Methods

The proposed technical solution for the recovery of phenolic compounds has been extensively discussed by the authors in previously published work [33,35]. Briefly, it can be described as a series of steps, first with the pretreatment of the OMW for the removal of the suspended solids, followed by membrane filtration (in line ultrafiltration, nanofiltration, and reverse osmosis), for the separation of the dissolved compounds according to their molecular weight, followed by the selective adsorption and desorption of resins for the separation of low molecular weight phenols from the low molecular weight carbohydrates (Figure 2). Finally, after the desorption of phenols with the use of ethanol, the ethanolic solution undergoes vacuum evaporation for the recovery of ethanol and the production of the final product of the process, a concentrated solution of low molecular weight phenols. Experimental results have shown that the final product can reach 380 g/L of phenols in gallic acid equivalents, with 85 g/L being hydroxytyrosol [35].

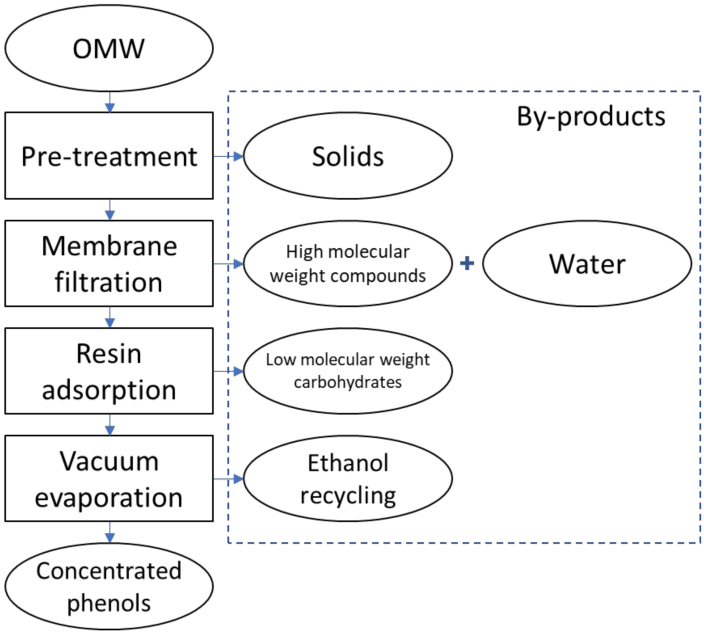


Figure 2. Proposed technical solution for the recovery of phenolic compounds from OMW.

The basis of the technoeconomic analysis in this study was the scaling of the equipment cost, followed by the calculation of the total operational cost on a yearly basis. The equipment cost for the scale of 36 m^3/d was collected from market data with average values being used. The number of offers collected per item was: One offer for the storage tanks, agitation system, vertical separator, membrane system, resin housing, and evaporator, three offers for the decanter, five offers for the flotator. For reasons discussed in Section 3.2, the cost of the purchased equipment was proven to have a small influence on the overall cost of phenols separation. This was validated through a sensitivity analysis at the scale of 36 m^3/d feed, with a 30% variation of the purchased equipment cost leading to only a 1% change in the phenols separation cost. Regarding the consumables, three offers were collected for the UF and NF membrane modules, two offers for the RO modules, and one offer for ethanol. As discussed in Section 3.2, resin cost was the most important cost identified through the analysis presented herein. Four offers were collected for the resin that had a standard deviation of 17%. Through a sensitivity analysis at the scale of 36 m^3/d feed, this resin cost deviation results in a 14% standard deviation of the final phenols separation cost.

The size of needed equipment was based on data published by the authors in previous works [33,35]. Ten percent equipment downtime for maintenance or repairs was taken into account for the calculations. Even though OMW is a seasonally produced waste, the proposed treatment method has been proven capable of the separation of phenols from all the liquid and solid (after solvent extraction of the phenolic content) agro-industrial wastes tested by the authors. This would allow the treatment plant to adjust to the locally produced agro-industrial wastes and operate throughout the year.

The purchased equipment cost was then scaled using the six-tenths-factor rule [34,36]. This rule is based on the premise that higher capacity equipment costs less per unit of capacity, a fact also known as the economy of scale. After the calculation of the purchased equipment costs at different scales, the calculation of the fixed capital investment and the total operational cost was possible, according to the equations presented in Table 1 [36].

Table 1. Equations used for the economic evaluation of the process.

Fixed Capital Investment [FCI]	Purchased equipment [PE]		Cost [a] × (Capacity [b]/Capacity [a])$^{0.6}$
	Direct costs	Piping	0.65 × PE
		Instrumentation and controls	0.18 × PE
		Electrical equipment and materials	0.2 × PE
		Buildings	0.45 × PE
		Yard improvements	0.15 × PE
		Service facilities	0.55 × PE
		Land	0.06 × PE
	Indirect costs	Engineering and supervision	0.3 × PE
		Construction expenses	(PE + Direct costs) × 0.1
		Contractor's fee	(PE + Direct costs) × 0.04
		Contingency	(PE + Direct costs) × 0.08
	Start-up expense		(PE + FCI) × 0.08
	Working capital		0.1 × FCI
	Total capital investment		FCI + start-up expense + working capital
Total Operational Cost [€/y] [TOC]	Direct production costs	Consumables and solvents	Mass balance of the process
		Operating labor [OL]	5000 × capacity in m^3/d
		Operating supervision	0.15 × OL
		Utilities	0.15 × OL
		Maintenance and repairs [MR]	0.07 × FCI
		Operating supplies	0.15 × MR
		Laboratory charges	0.15 × OL
		Royalties and patents	0.03 × TOC
	Fixed charges		0.1 × (PE + Buildings) + 0.02 × FCI
	Plant overhead		0.6 × OL
	Administrative expenses		0.25 × OL
	Distribution and marketing expenses		0.11 × TOC
	Research and development		0.035 × TOC
	Financing		0.05 × FCI
	Contingencies		0.03 × TOC

[a] Values collected from market data; [b] Calculated values.

The fixed capital investment included the cost of the purchased equipment, supplementary materials needed for the installation of the equipment (piping, electrical connections, etc.), and the building that would house the treatment plant with all its facilities. It also included indirect costs for the building of the facilities. The total operational cost can be divided again to direct production costs that include the consumables, labor, and other charges directly connected with the production process, and other costs like the depreciation of the equipment and building (included in fixed charges), expenses for the marketing of the product, the loan rate for the initial investment, etc.

Apart from the main product of the process, several other byproducts are produced with a potential for profit. These byproducts include the solids separated from the initial waste during pretreatment, the high molecular weight fraction of phenols separated in the nanofiltration concentrate, and the lower molecular weight carbohydrates separated during the resin process. All these products can be implemented for the enrichment of animal feed with antioxidants (separated solids), the production of natural herbicides (high-molecular-weight phenols), and possibly as food additives (low molecular weight carbohydrates with low concentrations of phenols). Because of the complexity of the exploitation of this type of byproducts, the process will be examined under the premise that the separated phenol will be the only source of income for the treatment plant.

3. Results

3.1. Purchased Equipment Cost and Fixed Capital Investment

As mentioned in Section 2, the collected data for the equipment refer to a process with a capacity of 36 m^3/d. This capacity may seem a bit arbitrary, but it was chosen to size the equipment to capacities that would be easier to find in the market. Moreover, this capacity is a good estimate for the production capacity of a typical olive mill. Three independent production lines were chosen for the design to offer some flexibility to the treating capacity of the plant.

The equipment necessary, alongside the data collected from suppliers, is presented in Table 2. The equipment includes all the necessary storage tanks for holding the wastewater between the treatment steps, three pretreatment steps prior to membrane filtration (flotator, decanter, and vertical separator) to prevent membrane fouling as much as possible, the three-step membrane system (ultrafiltration, nanofiltration, and reverse osmosis), the resin process and the evaporator.

Table 2. Main equipment used for the separation of OMW phenols and prices collected from suppliers.

Equipment	Capacity	Unit	Amount	Price in €/Piece
SS Tank	3	m^3	1	2133
	2.5	m^3	1	1912
	2	m^3	2	1673
	1	m^3	13	1103
	0.5	m^3	3	728
Agitation	3	m^3	1	1100
Flotator	0.5	m^3/h	3	450
Decanter	0.5	m^3/h	3	4800
V-separator	0.5	m^3/h	3	6000
Membrane system	1.5	m^3/h	1	100,000
Resin housing	65	kg	3	650
Evaporator	0.035	m^3/h	3	5500

As it was to be expected, the membrane filtration system is the most expensive part of the total equipment cost. This can be attributed to the high level of automation and monitoring required for the operation and maintenance of all the membrane steps needed in the process (UF, NF, RO). The pretreatment equipment also has a significant contribution (19%), followed by the large number of tanks required between the different steps of the process (13%). Through the rule of six-tenths for the economy of scale and the rest of the equations presented in Table 1, the purchased equipment cost and the fixed capital investment as a function of process scale are presented in Figure 3.

3.2. Total Operational Cost

Through the mass balance of the process [33], the most important consumables were identified and are presented in Table 3, with their expected yearly cost. For the membrane modules, a three-year lifespan was estimated, and for ethanol, 10% of its volume is considered not recoverable during the evaporation step and replenished with fresh solvent.

Table 3. Main consumables for the separation of OMW phenols and prices collected from suppliers.

Consumable	Amount	Unit	Price	Cost [€/y]
UF modules	1.9	m^2/y	71.4 €/m^2	137
NF modules	7.5	m^2/y	13.6 €/m^2	103
RO modules	10.1	m^2/y	9.4 €/m^2	94
Resin	7118	kg/y	319 €/kg	2,270,483
Ethanol	57.67	m^3/y	600 €/m^3	34,591

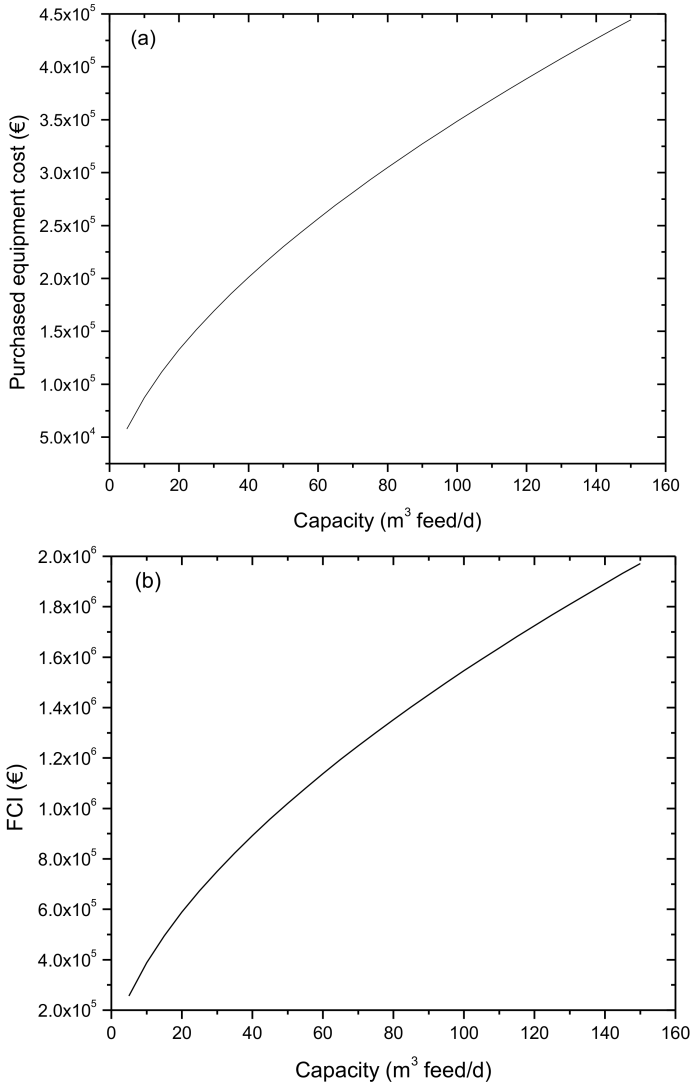


Figure 3. (**a**) Purchased equipment cost and (**b**) fixed capital investment, as a function of treatment plant capacity for the separation of phenols from OMW.

As it can be clearly observed from the yearly cost of resin replacement for the adsorption process, the cost is astronomical compared to the rest of the process. It is even larger than the total equipment cost. The cost presented in Table 3 corresponds to a resin lifespan of 50 adsorption/desorption cycles. From the mass balance of the process, with 50 cycles resin lifespan, approximately 0.6 kg of resin replacement per m^3 of OMW treated was calculated, with 11,826 m^3 of OMW being treated per year, leading to the resin cost presented in Table 3.

The effect of resin cost on the total operational cost is also illustrated in Figure 4, where consumables and solvents represent 54% of the total operational cost as it was calculated with the equations presented in Table 1.

The fact that the total operational cost depends so strongly on one consumable, like the resin needed for phenols adsorption that scales linearly with the amount of waste treated, indicates that the production cost per gram of phenols is not expected to drop at higher treatment plant capacities. This can be observed in Figure 5. It makes more sense to examine the effect of resin lifespan on the production cost of the separated phenols. Higher resin lifespans lead to less resin needed to be purchased per year and directly reduces the production cost, as can be observed in Figure 6.

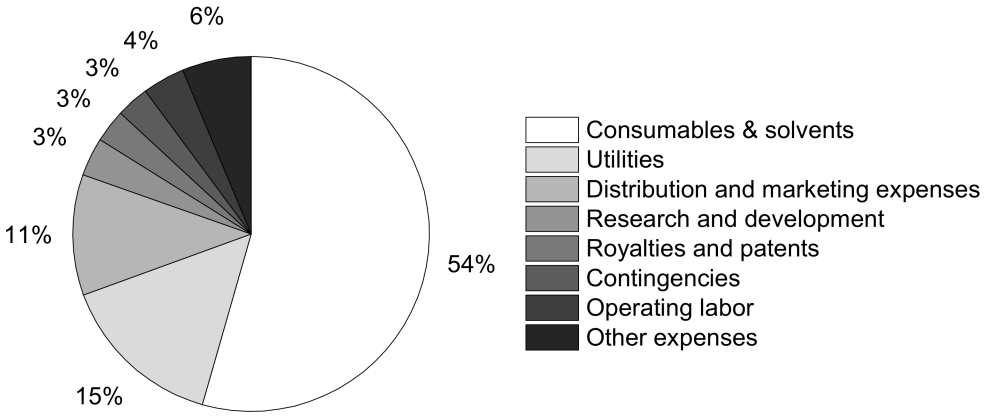


Figure 4. Operational cost categories calculated for 36 m^3/d feed capacity.

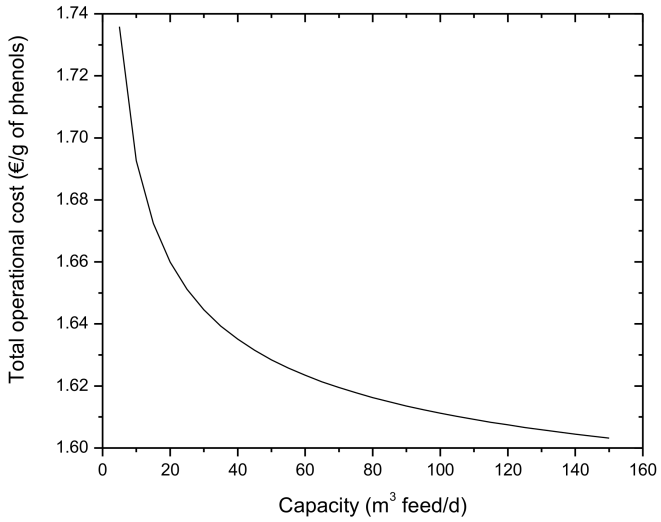


Figure 5. Total operational cost per g of product as a function of treatment plant capacity for the separation of phenols from OMW, calculated with a resin lifespan of 50 cycles.

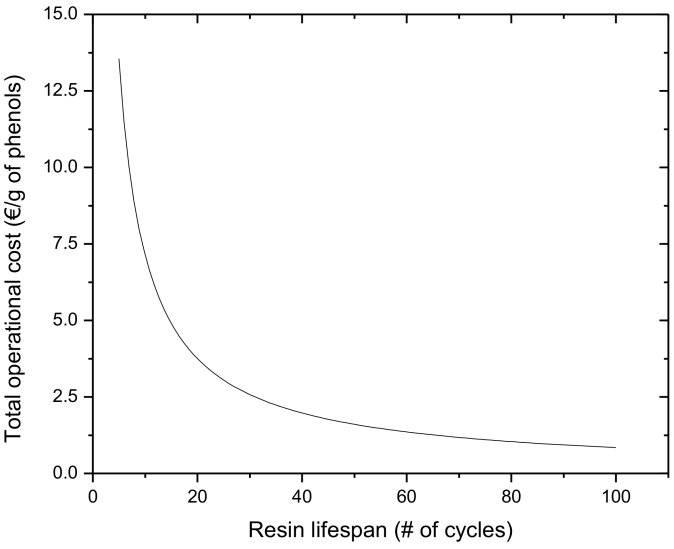


Figure 6. Effect of resin lifespan on the total operational cost for the separation of phenols from OMW, calculated for 36 m^3/d treatment plant capacity.

4. Discussion

From the analysis presented in this work, it is obvious that the resin lifespan is the single most important factor that will dictate the production cost for the separated phenols through the proposed process. During lab-scale experiments [30], the authors identified that after five resin cycles, no signs of separation efficiency drop were observed for the resin adsorption/desorption process. Unfortunately, in most of the scientific publications examined, the aspect of resin lifespan has not been explored. Moreover, the very specific nature of the solution used in the adsorption/desorption step of this process (only soluble lower molecular weight compounds contained in OMW) made finding relevant data for the expected resin lifespan proved rather difficult. Nevertheless, some information for processes as similar as possible to the one examined in this work will be mentioned. Sid Kalal et al. [37] used an amberlite XAD-4 resin functionalized with alizarin red-s for the adsorption of Rh(III), which exhibited less than 5% adsorption capacity change after 10 adsorption/desorption cycles. Pan et al. [38] examined the adsorption of 4-nitrophenol on a hyper-cross-linked polymeric resin from the same material as amberlite XAD-4. The authors of this work compared XAD-4 with the novel one they produced, with the novel resin having higher adsorption capacity than XAD-4 due to its cross-linked nature but similar desorption kinetics. The authors tested the cross-linked resin for 25 adsorption/desorption cycles with no significant changes. An estimate of 20–50 cycles resin lifespan would lead to a production cost of 3.7 to 1.6 €/g of phenols separated with the process. In order to put this number into perspective, the price of five extra virgin olive oils marketed as high phenolic content products that were found in the market ranged from 42 to 100 €/L. By subtracting a typical price of extra virgin olive oil (13.6 €/L) and using available data from the producers for the phenolic content of their products, a mean value of 76 €/g of phenols reported were added to the price of the olive oil. This may not be directly comparable to the price of the phenols separated from OMW but is indicative of the great interest in this type of antioxidants. Another possible application of the phenolic extract produced through the proposed process would be as a food supplement. Currently, there are products in the market with phenolic extracts, mostly from olive leaves, with very little information available regarding the extraction processes used for their production. The authors examined four supplements marketed as olive leaf extracts that reported their oleuropein content, with their price per g of oleuropein ranging from 1 to 17 €/g. One supplement marketed as olive polyphenols reported its content in hydroxytyrosol, with a price of 52 €/g.

In the work of Frascari et al. [39], for OMW phenol separation through resin adsorption after microfiltration, a separation cost of 1.7–13.5 per kg of phenols was calculated, but with an assumed resin lifespan of 500 cycles, with the authors stating that this number should be confirmed through further research. Even with 500 cycles resin lifespan, resin cost constituted 48% of the operational cost calculated. The authors of this work believe that 500 cycles lifespan is overoptimistic for a process that involves complex mixtures of phenols with other compounds contained in OMW. By assuming 500 cycles resin lifespan in the present study, the contribution of resin cost to the total operational cost drops from 54% to 22%, while the cost of phenols separation drops from 1.6 to 0.4 €/g. This price is still one to two orders of magnitude greater than the one calculated by Frascari et al., a difference that may be attributed to the more complex separation method presented herein that targets lower molecular weight and higher added-value phenols.

5. Conclusions

The process of phenols separation from olive oil production derived byproducts (and other agro-industrial byproducts rich in phenolic compounds) developed by the authors was examined from a technoeconomic point of view. The final product of this process is a phenolic extract with concentrations of phenols reaching 380 g/L. The main production costs were identified as direct costs that are not reduced at higher treatment plant capacities but are directly proportional to the amount of waste treated. Through the methodology

presented herein, a production cost of 3.7 to 1.6 €/g of phenols separated was estimated, strongly depending on the lifespan of the adsorption resin, highlighting the importance of resin lifespan identification and optimization for the viability of the process. Economic viability is one of the most important aspects examined by stakeholders before adopting a novel process.

This kind of product can be of interest to several rapidly growing industry sectors, like cosmetics, pharmaceuticals, and nutraceuticals production industries. Typically, phenols are present at low concentrations in this type of products (10 mg/L OMW phenols in sunscreens [40] and up to 3% grape extracts in cosmetics [41]), a fact that puts into perspective the actual cost for the amount of phenols needed for this type of products. In this perspective, the production cost identified in this study seems to allow the exploitation of the products of the proposed process by this type of industry. This can be a strong incentive for the treatment of OMW, diverting such a difficulty to treat waste from environmental receptors. The extraction of value and the alleviation of the environmental problems caused by OMW can lead to more sustainable olive oil production, an important agro-industrial product of Mediterranean countries.

Author Contributions: Conceptualization, C.A.P. and D.P.Z.; methodology, D.P.Z.; investigation, C.S.P.; writing—original draft preparation, D.P.Z.; writing—review and editing, C.S.P.; visualization, D.P.Z.; supervision, C.A.P.; funding acquisition, C.A.P. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by the project "INVALOR: Research Infrastructure for Waste Valorization and Sustainable Management" (MIS 5002495) which is implemented under the Action "Reinforcement of the Research and Innovation Infrastructure", funded by the Operational Programme "Competitiveness, Entrepreneurship and Innovation" (NSRF 2014-2020) and co-financed by Greece and the European Union (European Regional Development Fund).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data is contained within the article, in Section 3 Results.

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Aissa, I.; Kharrat, N.; Aloui, F.; Sellami, M.; Bouaziz, M.; Gargouri, Y. Valorization of antioxidants extracted from olive mill wastewater. *Biotechnol. Appl. Biochem.* **2017**, *64*, 579–589. [CrossRef]
2. Kumar, S.; Abedin, M.M.; Singh, A.K.; Das, S. Role of Phenolic Compounds in Plant-Defensive Mechanisms. In *Plant Phenolics in Sustainable Agriculture*; Lone, R., Shuab, R., Kamili, A.N., Eds.; Springer Singapore: Singapore, 2020; Volume 1, pp. 517–532. ISBN 978-981-15-4890-1.
3. Banias, G.; Achillas, C.; Vlachokostas, C.; Moussiopoulos, N.; Stefanou, M. Environmental impacts in the life cycle of olive oil: A literature review. *J. Sci. Food Agric.* **2017**, *97*, 1686–1697. [CrossRef] [PubMed]
4. Espadas-Aldana, G.; Vialle, C.; Belaud, J.-P.; Vaca-Garcia, C.; Sablayrolles, C. Analysis and trends for Life Cycle Assessment of olive oil production. *Sustain. Prod. Consum.* **2019**, *19*, 216–230. [CrossRef]
5. Brenes, M.; Romero, C.; García, A.; Hidalgo, F.J.; Ruiz-Méndez, M.V. Phenolic Compounds in Olive Oils Intended for Refining: Formation of 4-Ethylphenol during Olive Paste Storage. *J. Agric. Food Chem.* **2004**, *52*, 8177–8181. [CrossRef] [PubMed]
6. Hernández, D.; Quinteros-Lama, H.; Tenreiro, C.; Gabriel, D. Assessing Concentration Changes of Odorant Compounds in the Thermal-Mechanical Drying Phase of Sediment-Like Wastes from Olive Oil Extraction. *Appl. Sci.* **2019**, *9*, 519. [CrossRef]
7. Galanakis, C.M. Phenols recovered from olive mill wastewater as additives in meat products. *Trends Food Sci. Technol.* **2018**, *79*, 98–105. [CrossRef]
8. Ciriminna, R.; Meneguzzo, F.; Delisi, R.; Pagliaro, M. Olive biophenols as new antioxidant additives in food and beverage. *ChemistrySelect* **2017**, *2*, 1360–1365. [CrossRef]
9. Galanakis, C.M.; Tsatalas, P.; Galanakis, I.M. Implementation of phenols recovered from olive mill wastewater as UV booster in cosmetics. *Ind. Crop. Prod.* **2018**, *111*, 30–37. [CrossRef]
10. Lavecchia, R.; Zuorro, A. Evaluation of olive pomace as a source of phenolic antioxidants for the production of functional cosmetics. *Int. J. Appl. Eng. Res.* **2015**, *14*, 34405–34409.
11. Gunay, A.; Karadag, D. Recent developments in the anaerobic digestion of olive mill effluents. *Process. Biochem.* **2015**, *50*, 1893–1903. [CrossRef]

12. Khoufi, S.; Louhichi, A.; Sayadi, S. Optimization of anaerobic co-digestion of olive mill wastewater and liquid poultry manure in batch condition and semi-continuous jet-loop reactor. *Bioresour. Technol.* **2015**, *182*, 67–74. [CrossRef] [PubMed]
13. Bouallagui, H.; Rachdi, B.; Gannoun, H.; Hamdi, M. Mesophilic and thermophilic anaerobic co-digestion of abattoir wastewater and fruit and vegetable waste in anaerobic sequencing batch reactors. *Biodegradation* **2009**, *20*, 401–409. [CrossRef]
14. Carlini, M.; Castellucci, S.; Moneti, M. Anaerobic co-digestion of olive-mill solid waste with cattle manure and cattle slurry: Analysis of bio-methane potential. *Energy Procedia* **2015**, *81*, 354–367. [CrossRef]
15. El Gnaoui, Y.; Sounni, F.; Bakraoui, M.; Karouach, F.; Benlemlih, M.; Barz, M.; El Bari, H. Anaerobic co-digestion assessment of olive mill wastewater and food waste: Effect of mixture ratio on methane production and process stability. *J. Environ. Chem. Eng.* **2020**. [CrossRef]
16. Maragkaki, A.E.; Vasileiadis, I.; Fountoulakis, M.; Kyriakou, A.; Lasaridi, K.; Manios, T. Improving biogas production from anaerobic co-digestion of sewage sludge with a thermal dried mixture of food waste, cheese whey and olive mill wastewater. *Waste Manag.* **2018**, *71*, 644–651. [CrossRef] [PubMed]
17. Alver, A.; Baştürk, E.; Kılıç, A.; Karataş, M. Use of advance oxidation process to improve the biodegradability of olive oil mill effluents. *Process. Saf. Environ. Prot.* **2015**, *98*, 319–324. [CrossRef]
18. Amor, C.; Lucas, M.S.; García, J.; Dominguez, J.R.; De Heredia, J.B.; Peres, J.A. Combined treatment of olive mill wastewater by Fenton's reagent and anaerobic biological process. *J. Environ. Sci. Heal. Part A* **2015**, *50*, 161–168. [CrossRef]
19. Nogueira, V.; Lopes, I.; Freitas, A.C.; Rocha-Santos, T.A.P.; Gonçalves, F.; Duarte, A.C.; Pereira, R. Biological treatment with fungi of olive mill wastewater pre-treated by photocatalytic oxidation with nanomaterials. *Ecotoxicol. Environ. Saf.* **2015**, *115*, 234–242. [CrossRef]
20. Cuomo, F.; Venditti, F.; Cinelli, G.; Ceglie, A.; Lopez, F. Olive mill wastewater (OMW) phenol compounds degradation by means of a visible light activated titanium dioxide-based photocatalyst. *Z. Phys. Chem.* **2016**, *230*, 1269–1280. [CrossRef]
21. Salameh, W.K.M.B.; Ahmad, H.; Al-Shannag, M. Treatment of olive mill wastewater by electrocoagulation processes and water resources management. *Int. J. Environ. Ecol. Eng.* **2015**, *9*, 296–300.
22. Esfandyari, Y.; Mahdavi, Y.; Seyedsalehi, M.; Hoseini, M.; Safari, G.H.; Ghozikali, M.G.; Kamani, H.; Jaafari, J. Degradation and biodegradability improvement of the olive mill wastewater by peroxi-electrocoagulation/electrooxidation-electroflotation process with bipolar aluminum electrodes. *Environ. Sci. Pollut. Res.* **2015**, *22*, 6288–6297. [CrossRef]
23. Pulido, J.M.O. A review on the use of membrane technology and fouling control for olive mill wastewater treatment. *Sci. Total Environ.* **2016**, *563*, 664–675. [CrossRef]
24. Conidi, C.; Mazzei, R.; Cassano, A.; Giorno, L. Integrated membrane system for the production of phytotherapics from olive mill wastewaters. *J. Membr. Sci.* **2014**, *454*, 322–329. [CrossRef]
25. Gebreyohannes, A.Y.; Mazzei, R.; Giorno, L. Trends and current practices of olive mill wastewater treatment: Application of integrated membrane process and its future perspective. *Sep. Purif. Technol.* **2016**, *162*, 45–60. [CrossRef]
26. Frascari, D.; Bacca, A.E.M.; Zama, F.; Bertin, L.; Fava, F.; Pinelli, D. Olive mill wastewater valorisation through phenolic compounds adsorption in a continuous flow column. *Chem. Eng. J.* **2016**, *283*, 293–303. [CrossRef]
27. Pinelli, D.; Bacca, A.E.M.; Kaushik, A.; Basu, S.; Nocentini, M.; Bertin, L.; Frascari, D. Batch and continuous flow adsorption of phenolic compounds from olive mill wastewater: A comparison between nonionic and ion exchange resins. *Int. J. Chem. Eng.* **2016**, *2016*. [CrossRef]
28. Aliakbarian, B.; Casazza, A.A.; Perego, P. Kinetic and isotherm modelling of the adsorption of phenolic compounds from olive mill wastewater onto activated carbon. *Food Technol. Biotechnol.* **2015**, *53*, 207–214. [CrossRef] [PubMed]
29. Web of Science. Available online: https://www.webofknowledge.com (accessed on 14 December 2020).
30. Zagklis, D.P. Separation, Isolation and Enrichment of Phenolic Compounds from Agricultural Byproducts with Physicochemical Methods. Ph.D. Thesis, University of Patras, Patras, Greece, 2015.
31. Zagklis, D.P.; Paraskeva, C.A. Purification of grape marc phenolic compounds through solvent extraction, membrane filtration and resin adsorption/desorption. *Sep. Purif. Technol.* **2015**, *156*. [CrossRef]
32. Zagklis, D.P.; Paraskeva, C.A. Isolation of organic compounds with high added values from agro-industrial solid wastes. *J. Environ. Manag.* **2018**, *216*. [CrossRef]
33. Zagklis, D.P.; Paraskeva, C.A. Preliminary design of a phenols purification plant. *J. Chem. Technol. Biotechnol.* **2020**. [CrossRef]
34. Arvaniti, E.C.; Zagklis, D.P.; Papadakis, V.G.; Paraskeva, C.A. High-added value materials production from OMW: A technical and economical optimization. *Int. J. Chem. Eng.* **2012**. [CrossRef]
35. Zagklis, D.P.; Vavouraki, A.I.; Kornaros, M.E.; Paraskeva, C.A. Purification of olive mill wastewater phenols through membrane filtration and resin adsorption/desorption. *J. Hazard. Mater.* **2015**, *285*. [CrossRef] [PubMed]
36. Peters, M.S.; Timmerhaus, K.D.; West, R.E. *Plant Design and Economics for Chemical Engineers*, 5th ed.; McGraw-Hill: New York, NY, USA, 2003; Volume 66.
37. Sid Kalal, H.; Panahi, H.A.; Hoveidi, H.; Taghiof, M.; Menderjani, M.T. Synthesis and application of Amberlite xad-4 functionalized with alizarin red-s for preconcentration and adsorption of rhodium (III). *Iran. J. Environ. Health Sci. Eng.* **2012**, *9*, 7. [CrossRef] [PubMed]
38. Pan, B.; Du, W.; Zhang, W.; Zhang, X.; Zhang, Q.; Pan, B.; Lv, L.; Zhang, Q.; Chen, J. Improved Adsorption of 4-Nitrophenol onto a Novel Hyper-Cross-Linked Polymer. *Environ. Sci. Technol.* **2007**, *41*, 5057–5062. [CrossRef] [PubMed]

39. Frascari, D.; Bacca, A.E.M.; Wardenaar, T.; Oertlé, E.; Pinelli, D. Continuous flow adsorption of phenolic compounds from olive mill wastewater with resin XAD16N: Life cycle assessment, cost–benefit analysis and process optimization. *J. Chem. Technol. Biotechnol.* **2019**, *94*, 1968–1981. [CrossRef]
40. Galanakis, C.M.; Tsatalas, P.; Galanakis, I.M. Phenols from olive mill wastewater and other natural antioxidants as UV filters in sunscreens. *Environ. Technol. Innov.* **2018**, *9*, 160–168. [CrossRef]
41. Fiume, M.M.; Bergfeld, W.F.; Belsito, D.V.; Hill, R.A.; Klaassen, C.D.; Liebler, D.C.; Marks, J.G.; Shank, R.C.; Slaga, T.J.; Snyder, P.W.; et al. Safety Assessment of *Vitis vinifera* (Grape)-Derived Ingredients as Used in Cosmetics. *Int. J. Toxicol.* **2014**, *33*, 48S–83S. [CrossRef]