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Organic Pollutants Removal from Olive Mill Wastewater using a new Ecosystem Treatment

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ABSTRACT

Olive mill wastewater is the main by-product derived from olive mills using the three-phase extraction process,displaying a serious environmental risk due to its notable content in organics and phenolics Olive oil production, an agro-industrial of vital economic particularly in Mediterranean countries, is unfortunately associated with the generation of large quantities of OMW (Olive Mill Wastewater) and solid wastes. The OMW is considered a major environmental problem, it is a powerful pollutant rejected in nature without any prior treatment. This research work aims to study the treatment of OMW by a new ecological and economic system, which consists of the use of the following components: gravel, sawdust, soil, activated carbon, bamboo, and the valorization of the solid residues. HPLC analysis showed that hydroxytyrosol is the most abundant biophenol. Many other biophenols were identified (Tyrosol, gallic acid, and eleonic acid). The comparison between before and after filtration by the new system showed an essential degradation of phenolic compounds after treatment and found a new compound resulting from their degradation.

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INTRODUCTION

Olive oil industry has an important economic, environmental and social impact in the Mediterranean countries, generating about 98% of the world olive-oil production. This activity is key in the agro-industrial sector of the Mediterranean basin since the olive oil mills are concentrated in this zone dedicating large surface area to the olive tree cultivation. In fact, 75% of the olive oil production comes from Mediterranean Member States of the European Union (Gullon et al, 2020).

The Moroccan olive heritage has an area of 1200,000 ha during the 2021-2022 campaign. Moreover, it counts 51 million working days per year. Morocco is one of the Mediterranean countries in olive production with an intense concentration at the level of Fez-Meknes and Marrakech-Safi (Figure 1). In this sense, the increase in olive production and the introduction of modern oil extraction techniques have placed the olive tree in a delicate position of potential polluters (Iboukhloulef, H, 2014).

The processing of fruits includes cleaning, debittering, and fermentation (Niazmand et al, 2020). In the debittering step, they are soaked in alkaline or NaCl solutions and are washed with water frequently. The whole process consumes a large quantity of water, which is entirely

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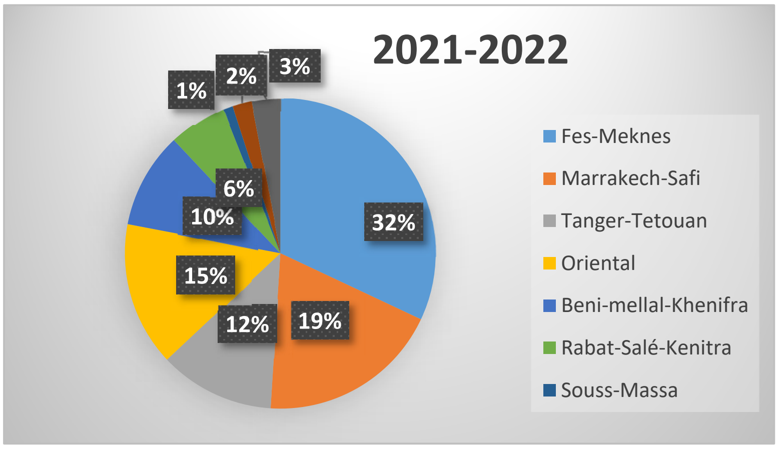


Fig. 1. Annual production of olive oils in Morocco in 2021-2022

converted to the waste stream (Gutiérrez-Rosales et al, 2003). Generally, olive industries use about 0.4–0.8 m^3 water per ton of green olives in the debittering stage. A previous study in Greece showed that olive treatment factories produce 3.9–7.5 m^3 wastewater per ton of green olives and 0.9–1.9 m^3 wastewater per ton of black olives (Parinos et al, 2007). The generated discharge contains different organic compounds, such as phenolic compounds, as well as inorganic compounds with environmental risks that require appropriate remediation methods (Agglis et al, 2002). Generally, these industrial wastewaters have high chemical oxygen demand (COD) values due to high organic load; for example, for olive mill wastewater this value is 48,500 mg/l (Inan et al, 2004), and for potato chips manufacturing wastewater it is 2800 mg/l (Kobya et al, 2006). Phenolic compounds are considered primacy pollutants, which are dangerous to organisms and may have adverse effects on human health. They are distributed in different concentrations in industrial wastewaters; for example, olive mill wastewater has a concentration of 2400 mg/l (Adhoum, N and Monser, L, 2004), while oil refinery wastewater has a concentration of 13 mg/l. The treatment of industrial effluents containing phenolic compounds encountered operating problems due to their poor biodegradability, high toxicity, and ecological status, such as the generation of toxic by-products (Abdelwahab et al, 2009).

The olive industries produce a massive quantity of OMW every year. Unfortunately, these are rejected without any pre-treatment. Thus, it presents high toxicity against the whole natural ecosystem, including microorganisms, plants, and animals.

In addition, the extraction of olive oil generates large quantities of solid residues (pomace) and liquid residues OMW, which are not treated and are often discharged into sewage systems, stored in evaporation ponds or spread directly on the ground. This has a negative impact on the environment, resulting in the clogging of soils, pollution of surface and groundwater, and the release of foul odors (Achak et al 2011). Discharging untreated OMW loaded with polyphenols poses a major ecological problem for olive oil producing countries (Zghari et al, 2018).

In this regard, researchers developed different treatment methods, but some were expensive, and others were inappropriate. Thus, more efforts should be made to save nature from this pollutant. Among the methods that have been developed and tested recently are the treatment of olive oil mill wastewater by advanced oxidation processes based on the Fenton-like system (H2O2/Cu) (Iboukhloulef, H, 2014) and chemical oxidation for the treatment of natural industrial wastewater from olive oil mills (Hodaifa et al, 2019) and treatment of OMW by adsorption on titanium oxide nanoparticles (Al Bsoul et al, 2019). However, these methods use chemicals that can have a negative impact on the environment and are relatively expensive.

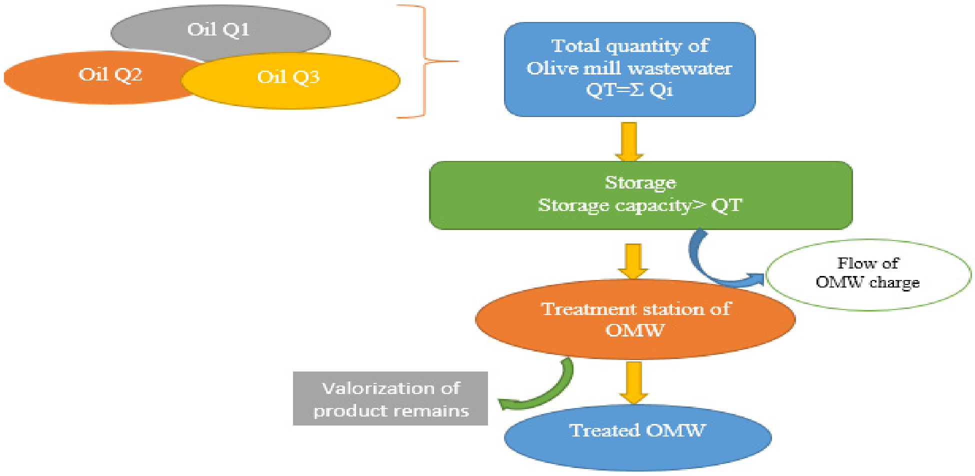


Fig. 2. Strategic Proposal for OMW treatment

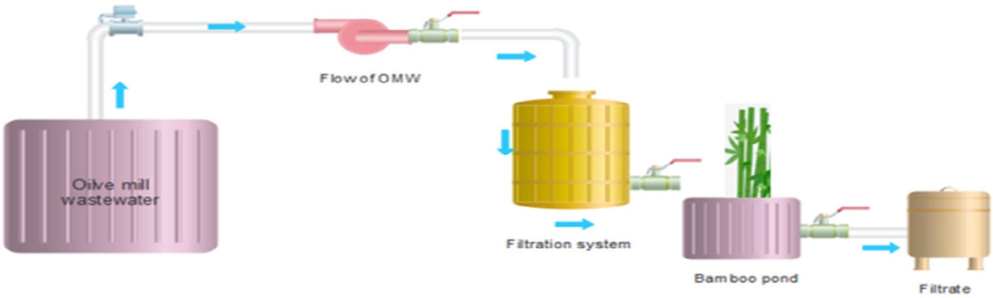


Fig. 3. A pilot system for the treatment of OMW

Adsorption of phenolic compounds is one of the most recently investigated methods for treating OMW (Solomakou,N and Goula, A.M, 2021) and (Achak et al, 2011). Nevertheless, most of these techniques have low treatment efficiency, may be ineffective or even generate more toxic by-products (Lee et al, 2019). The objective of the present study is the filtration of OMW by an innovative, ecological and economic system in order to determine the level of some phenolic compounds before and after the filtration.

MATERIALS AND METHODS

Olive oil is a product of great economic value, especially for Mediterranean countries. More than 800 million olive trees are cultivated worldwide, and the Mediterranean represents about 97% of the world's olive cultivation (Khdair, et al, 2019).

- The olives were processed in an industrial plant in the region of Beni-Mellal -Khenifra.
- The capacity of treatment of olives for 2h to 3h is 300 Kg, producing 50 L of extracted oil depleted in polyphenols and 15 L of OMW.
- The experimental pilot system (Figure 3) is composed of: a recipient of capacity 25 L and a diameter of 35 cm, filled with a thickness of 10 cm of pebbles at the bottom of the recipient, 6 cm of a mixture of (sand, coal, sawdust), and 10 cm of gravel (coarse and fine) at the top.
- The feeding of the system is done horizontally by OMW with a regular frequency of 2 times per week (one day of feeding and one day of rest)

Table 1. Characteristics of the pilot system

Volume	**2420 cm^3**
Base area	121 cm^2
Volume/ Surface area	20 cm^3/cm^2
Total area	1122 cm^2
Density	0,5 g/ cm^3

Table 2. Treatment technologies of Olive Mill Wastewater

Methodology	**Results**	**References**
Treatment of Olive Mill Wastewater and municipal wastewater mixture by pilot scale vertical flow constructed wetland	HPLC analysis of phenolic fractions in studied effluents highlights the removal of a variety of these compounds, especially toxic ones such as tyrosol and hydroxytyrosol after treatment by the PS-VFCW.	[El Ghadraoui et al, 2020]
Treatment of modern olive mill effluent by infiltration percolation on sand filter	The HPLC analysis shows that the major monomers of the studied OMW are hydroxytyrosol and tyrosol. The dilution of OMW by urban wastewater ensures an important elimination of the organic matter.	[Achak et al, 2009]
Performance and dynamic modeling of a continuously operated pomace olive packed bed for Olive Mill Wastewater treatment and phenol recovery	The analysis of the phenolic composition of this effluent by HPLC revealed the presence of a varied phenolic pool composed mainly of gallic acid, tyrosol, ferulic acid, caffeic acid, cinnamic acid, and coumaric acid	[Lissanddine et al, 2021]
Biodegradation of polyphenolic compounds from OMW during two-stage Anaerobic Co-digestion of agro-industrial mixtures	Main phenolic compounds namely Gallic acid, hydroxytyrosol, tyrosol, p-coumaric acid and oleuropein were identified in OMW samples. All five individual phenols in all OMW mixtures were degraded largely (80% degradation for the mixture in a ratio of 55 OMW: 40 CW: 5% LCM and 80 OMW: 20% CW and 100% degradation for 20 OMW: 80% LCM.	[Vavourak et al, 2020]
Chemical characteristics of two-phase OMW and Evaluation of their direct-soil application in Humid Mediterranean Regions	Hydroxytyrosol was the major phenolic compound identified in this two-phase OMW (148–8505 mg kg−1 DW). The other phenolic identified and quantified at lower concentrations were hydroxytyrosol glucoside, trysol glucoside, luteolin-7-O-glucoside, luteolin, and apigenin.	[Podgomik et al, 2022]
Phenolic decontamination of OMW using onion solid by-products homogenate	HPLC analyzes were carried out on samples representing OMW before and after the treatment. Results showed that the degradation of some phenols was carried out with the use of onion peroxidase, as well as with the use of the optimal conditions regarding the pH and the concentration of hydrogen peroxide. It was concluded that onion peroxidase could catalyze the degradation of total phenols and o-diphenols	[Reheema et al,2019]
Evaluation of the performance of a pilot-scale solar still for Olive Mill Wastewater treatment	Liquid chromatography – high resolution mass spectrometry analysis detected concentrations of tyrosol and 4- hydroxybenzoic acid in the collected distillates at concentrations up to 0.70 and 0.10 mg/L	[Mastoras et al 2022]

● The flow of the OMW is done by percolation through the substrate (soil). The percolated water is recovered using a Bamboo basin placed at the recipient's base. After filtering the OMW, it flows into the basin, The soil comes from a garden next to the olive factory; the gravel has a dimension of 4mm < d< 11mm. It was used to avoid the compensation of the soil at the bottom of the tank and reduce the clogging of the OMW.

Parameters analyzed

The phenolic compounds extraction technique of (Macheix et al, 1990) was used to 20 ml of OMW previously acidified by metaphosphoric acid (20%) to prevent the oxidation of phenolic compounds, 20 ml of 40% ammonium sulfate was added to increase the ionic strength of the medium. The sample then undergoes dilapidation three times 20 ml performed by three times 4.4 ml of ethyl acetate. The extract is evaporated at 35°C and the residue is recovered in 5 ml of pure methanol. The extraction of polyphenols is then performed by three times with 4.4 ml of ethyl acetate. For the determination of phenolic compounds, 50 µl of the phenolic extract is mixed with 1.35 ml of distilled water and 200 µl of Folin-Ciocalteu reagent. After 3 min, 400 µl of a 20% sodium carbonate solution is added to the mixture. The whole mixture is placed in a water bath for 20 min at 40°C. The absorbance is measured at 760 nm. The phenolic fractions of the OMW were identified by high-pressure liquid chromatography (HPLC) using a UV detector (280 nm) and a C18 column (4.6 x 250 mm), 20 µL of the extract was injected and eluted by increasing gradient of acetonitrile (mobile phases: H_2O +H_3PO4 (0.1%), acetonitrile/water (7/3), for 60 min with a flow rate of 0.8 ml/min (Yaakoubi et al, 2010). The analyses are realized at the CNRST in Rabat.

RESULTS AND DISCUSSION

OMWW are often disposed in evaporation ponds or various environmental receptors (Souilem et al, 2017) causing strong odor nuisance, soil contamination, plants growth inhibition, natural streams pollution as well as severe effects on the aquatic fauna and on the ecological status (Komnitsas et al,2016). OMWW is one of the most polluting effluents produced by the agro-food industries due to its high organic load and a wide range of contaminants, including organo-halogenated pollutants, fatty acids, phenolic compounds, and tannins (Karaouzas et al 2011) , (Ntougias et al, 2013). The high phenolic nature of OMWW and its organic contents make it highly resistant to biodegradation (Zirehpour et al 2012),(Zirehpour et al 2014).

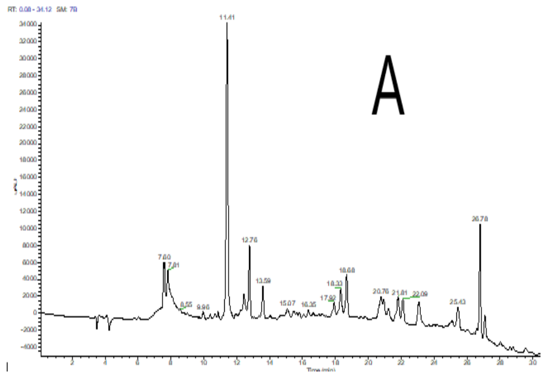
The phenolic extract of the crude OMW appeared very rich in phenolic compounds by HPLC at 280 nm (Figure 4). More than ten peaks were observed, four of which were majorities.

The results obtained after applying the proposed process are considered sufficient for the first step of OMW treatment.

The identification of phenolic compounds in the extracts of OMW was previously performed by HPLC-UV, comparing the relative retention times and UV spectra with those of the st&ard solution (De Marco et al, 2007). A representative chromatogram of a phenolic extract of OMW is reported in the following Figures (4, 5, 6, and 7). The HPLC profile showed several peaks corresponding to different biophenols, among which 4 compounds were identified: Hydroxytyrosol, Tyrosol, Gallic acid, and Eleonic acid.

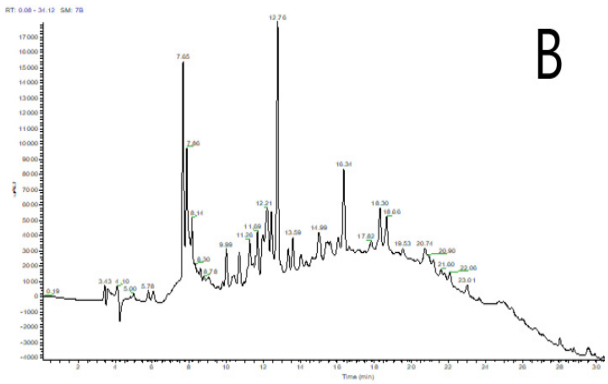
The results of the HPLC analysis of the phenolic compounds of the raw (Figure 4, A) and treated (Figures 5; B, 6; C, 7; D) OMW show that the polyphenols undergo degradation after filtration with the pilot system (Figure 2).

HPLC analysis of the phenolic extracts (Figures 5 and 6) shows a significant removal of OMW. The chromatograms also revealed the presence of traces of new compounds which do not correspond to the initial compounds and would be compounded resulting from their degradation.



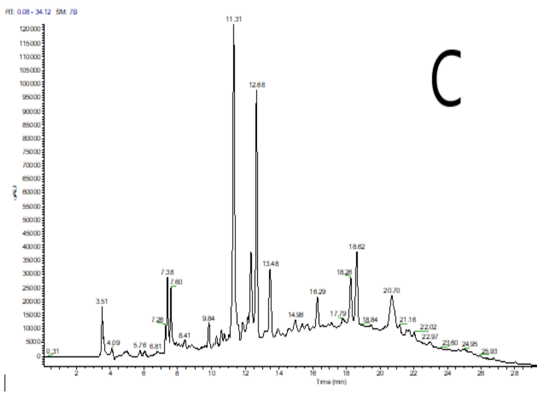
RT	Peak Area	Peak Height	Area %
3.41	10872.13	1187.37	0.99
3.62	18373.30	1352.48	1.67
4.09	17263.38	1417.08	1.57
4.54	16126.99	859.21	1.47
7.54	63007.78	6835.83	5.74
7.60	33907.10	6841.04	3.09
7.81	122738.49	5794.77	11.19
11.41	260761.52	35124.86	23.77
12.44	30680.42	2730.60	2.80
12.76	61501.26	8553.37	5.61
13.59	28963.92	3763.74	2.64
15.07	11226.45	966.63	1.02
17.92	18024.59	1720.06	1.64
18.33	34692.52	3325.14	3.16
18.68	45411.52	4968.45	4.14
20.76	35183.01	2730.58	3.21
20.93	23441.46	2433.48	2.14
21.21	17110.69	1426.59	1.56
21.81	37113.02	3002.15	3.38
22.09	29948.87	2947.83	2.73
23.07	37357.54	2886.26	3.40
25.09	14264.75	886.27	1.30
25.43	29460.31	2682.74	2.68
26.78	87684.89	13400.18	7.99
28.03	12109.20	774.00	1.10

Fig. 4. HPLC of raw phenolic extract (before treatment)



RT	Peak Area	Peak Height	Area %
3.61	12654.38	1040.17	1.42
4.10	21848.78	1882.75	2.46
4.54	23998.76	1241.93	2.70
4.82	13595.27	916.57	1.53
5.00	13971.00	945.22	1.57
7.66	136349.71	15705.77	15.32
7.86	93928.64	9891.52	10.56
8.14	64662.57	5063.80	7.27
8.60	12562.85	1569.18	1.41
9.02	12439.28	828.95	1.40
9.99	15944.01	2419.27	1.79
10.69	15130.17	2136.70	1.70
11.26	26725.33	2542.25	3.00
11.69	19473.25	2924.17	2.19
11.98	26231.36	2560.06	2.95
12.21	50658.95	4250.68	5.69
12.43	34013.19	3966.96	3.82
12.76	122239.64	16377.10	13.74
13.59	16556.01	2162.13	1.86
14.99	25193.29	1968.05	2.83
15.46	16440.81	1153.27	1.85
16.05	12739.55	1242.96	1.43

Fig. 5. HPLC of raw phenolic extract (after treatment)



RT	Peak Area	Peak Height	Area %
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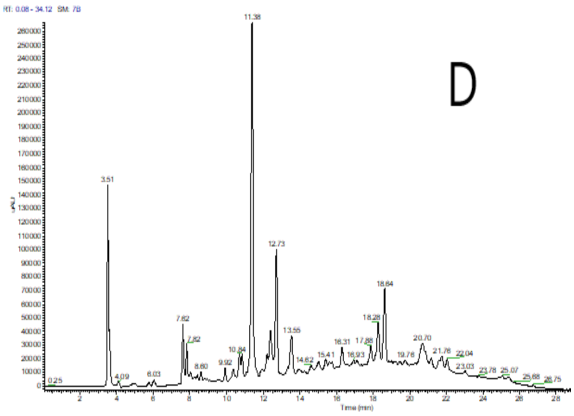
25.43	29460.31	2682.74	2.68
26.78	87684.89	13400.18	7.99
28.03	12109.20	774.00	1.10

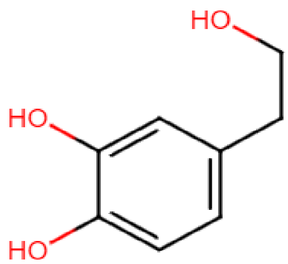
Fig. 6. HPLC of raw phenolic extract (after treatment)

This result is comparable to that found by Achak et al (Achak et al, 2011), who used the sand filter &, Yaakoubi A (Yaakoubi et al, 2010), who used treatment at neutral pH and aerobic conditions by the soil microflora before spreading, which showed degradation of phenolic compounds by the microbial activity of the sand that leads to the transformation of phenols into phenates with the formation of ions $C_6H_5O^-$ (Achak et al, 2009), These are well retained by soil cations, such as the oxides. These last ones are well retained by the cations of the soil, such as

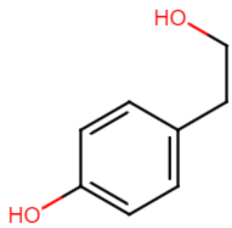


RT	Peak Area	Peak Height	Area %
3.51	746626.70	146722.30	7.29
7.62	288659.62	45456.55	2.82
7.82	165536.12	29921.71	1.62
10.71	165058.93	19723.89	1.61
11.38	2231419.46	264183.69	21.77
12.21	194999.19	20056.57	1.90
12.40	414768.73	36956.50	4.05
12.73	781165.02	96432.01	7.62
13.55	362174.76	32783.48	3.53
14.62	171858.98	10253.41	1.68
15.03	251118.42	12835.26	2.45
15.41	203115.52	14238.61	1.98
16.31	333782.97	22914.35	3.26
16.93	215966.82	13631.46	2.11
17.11	183550.67	13186.62	1.79
17.88	376771.22	23734.31	3.68
18.28	552307.35	40497.33	5.39
18.64	684772.48	65289.01	6.68
19.20	179741.91	11612.44	1.75
19.76	220921.77	12649.15	2.16
20.28	231454.53	9969.69	2.26
20.70	686973.62	24594.56	6.70
21.16	204139.47	13468.96	1.99
21.76	171425.35	14818.94	1.67
22.04	229708.95	12692.93	2.24

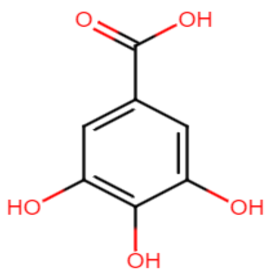
Fig. 7. HPLC of raw phenolic extract (after treatment)



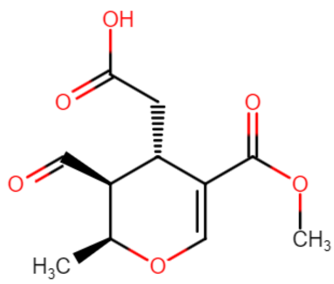
Hydroxytyrosol



Tyrosol



Gallic acid



Eleonic acid

Fig. 8. The compounds were identified with HPLC analysis

the oxides of aluminium and iron, the carbonates of calcium and the silicates.

After the treatment of the OMW, the phenol richness decreased, only a few weak peaks were detected. The new peaks at 280 nm observed by HPLC after filtration could correspond to new phenolic compounds resulting from the degradation of the starting phenolic compounds.

The analysis of HPLC chromatograms (Figures 4, 5, 6 and 7) shows that the major phenolic compounds present in the raw OMW are hydroxytyrosol (Tr = 7.81) and Tyrosol (Tr= 15.03; 15.07). This confirms the results (Achak et al, 2011), (Artajo et al, 2006), (Ying et al, 2009), and (Peralbo-Molina et al, 2012), who showed that an extract of raw OMW is composed of these two compounds.

CONCLUSION

The present study consists of developing an efficient and ecological solution to the problem of OMW. This solution was first developed at the laboratory scale to consider experimentation in a real site next to an olive crushing plant. This work was carried out to test the removal of phenolic compounds by a new ecological system that could be considered a new solution at the industrial scale of OMW treatment.

The results obtained after applying the proposed process exhibit a promising achievement, which would be considered as a first step in the treatment of OMW. The HPLC chromatogram analyses show that the major monomers of the studied OMW are Hydroxytyrosol, Tyrosol, and their degradation after applying the proposed system. This treatment ensured an important elimination of toxic phenolic compounds in the environment and the valorization of by-products that could be considered a potential source of natural products. The toxicity of the OMW can be minimized, and the environment therefore protected, by using this system as a pretreatment of Olive Mill Wastewater.

GRANT SUPPORT DETAILS

The present research did not receive any financial support.

CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/or submission, and redundancy has been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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