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Acquired Scientific Research on the Treatment or Valorization of Olive Oil Waste

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Abstract—Olive growing is an area that continues to improve. In 2019, the ministry of agriculture has recorded the production of 2 million tonnes, an increase of 28% compared to the year 2018. However, this industrial sector generates each year millions of tons of tow kind of wastes: liquid pollutants called olive oil wastewater (OWW), and solid waste named pomace. The liquid pollutant, for most olive crushing plants, is discharged directly into watercourses without prior treatment; which creates serious ecological problems. While the pomace is used, for some industries such as fuel, soap...

This work summarizes almost scientific research achievements on the treatment and / or recovery of olive waste during this last decade including those published via GoogleScholar, PubMed, Sciencedirect, Scopus, and web of science and those made in the LIEME laboratory of the FSDM of the university Sidi Mohammed Ben Abdallah (Morocco).

This study shows that OWW can be treated by several physicochemical or biological methods or coupling between the two methods; namely decantation, coagulation flocculation, electrocoagulation, adsorption, percolation, filtration and biodegradation. Recovery of this waste can be achieved by fertilization of crops, co-composting, germination of seeds, extraction of antioxidants and certain bioactive molecules and by production of biogas. Concerning pomace, few studies reported their use as co-fuel in cement kilns, or as a fertilizer by direct application or after composting. The choice of technique is related to the degree of pollution of this waste and the purpose of treatment or recovery.

Index Terms— Olive wastewater (OWW); pomace, recovery, fertilization, seed germination, antioxidants, biogas, bioactive molecules

1 INTRODUCTION:

The production of olive oil is mainly concentrated in the countries around the Mediterranean: Spain, Italy, Greece, Turkey, Syria, Tunisia and Morocco. World production of table olives is around 2,511,500 tonnes [1]

In Morocco the olive industry has made remarkable progress thanks to the "green plan" adopted by the ministry of agriculture (**fig. 1**). In 2018, total production was around 2,000,000 tons of olive[2]. Thus, the annual growth average rate of olive oil production in Morocco from 2000 to 2018 was 11.93% [1]

If we consider that the rate will remain the same in the next 20 years, we will have an olive production which will reach 2,798,250 T. Moreover, it is estimated that the crushing of 100 kg of olives by the triphasic olive-growing units produces from 50 to 120 litres of OWW, and from 33 to 65 kilograms of olive pomace[3]. So, in 2040, these units will dump more than 2.2.106T of OWW and more than 13.106 T of olive pomace in natural environments.

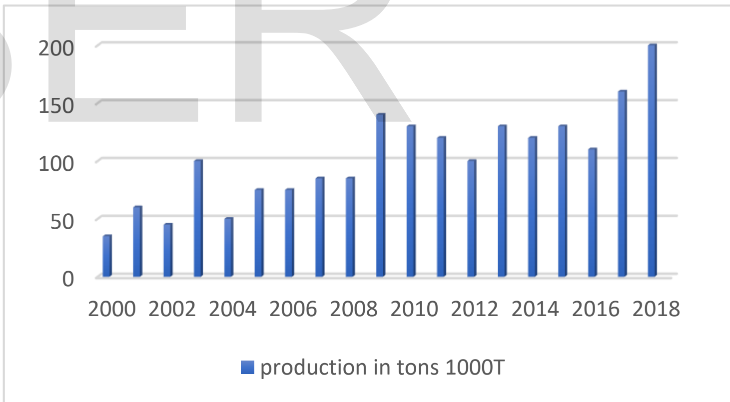

Fig. 1 Evolution of annual national olive production between 2000 and 2018[1]

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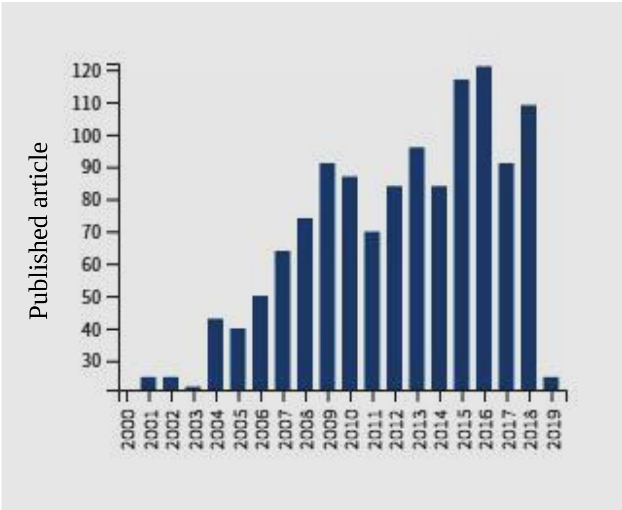

Fig. 2 Number of publications in indexed journals containing the phrase " olive mill wastewater " between 2000 and 2019

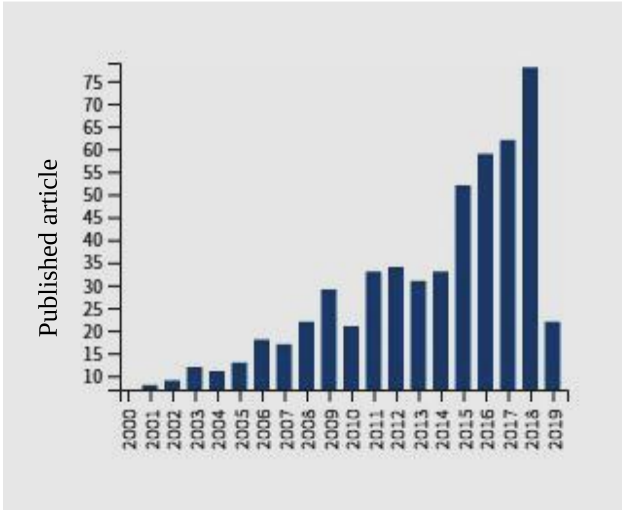

Fig. 3 Number of publications in Indexed journals containing the phrase " olive pomace " between 2000 and 2019

Despite its contribution of 5% to the national agricultural GDP[4], the olive industry has various environmental impacts in terms of water eutrophication confirmed by the proliferation of certain diatomaceous algae such as *Nitzschia palea* and *Gomphonema parvulum*[5], and soil degradation[6]. These impacts vary according to the practices and techniques used in the crushing of olives.

On the other hand, olive oil wastes (solid and liquid) are rich of organic matter and nutrients (NPK)[7] and bioactive molecules (polyphenols)[8]–[10]. The recovery of these residues by the extraction and / or biotransformation of their organic materials is a relevant opportunity, as it can combine waste treatment with the production of value-added products, considering industrial ecology concepts such as "cradle to cradle (C2C)" and the circular economy. By using the analysis tools of the " WebOfSciences " platform, it can be seen that there is a progressive growth of published articles containing the phrases " olive mill wastewater " and " olive pomace " along the period between 2000 and 2019 (**fig.2**. and **fig.3**. successively), as well as the areas of research interested in the subject of the recovery of olive oil waste over the same period (**fig.4**. and **fig.5** successively)

This study shows that olive oil waste water (OWW) can be treated by several physicochemical, biological methods or by coupling between the two methods. Among these methods are decantation[11], coagulation-flocculation[12]–[15], electrocoagulation[7], [16], [17], adsorption[18]–[20], percolation[21], filtration[22], and biodegradation[23]–[26]. this liquid waste can be used as a fertilizer for crops[17], [27]–[29], and in co-composting processes[17], [29], [30], seed germination[31], extraction of antioxidants and certain bioactive molecules[9], [32]–[35], as well as in the production of biogas[24], [36], [37]. While for pomace, few studies have reported their use as co-fuel cement kilns[38], a source of molecules with biological activities[39]–[41]. The choice of technique is related to the degree of pollution of this waste and the purpose of treatment or recovery.

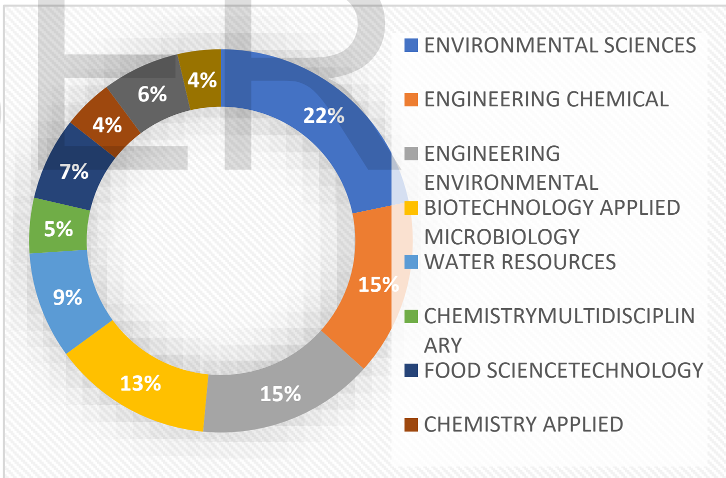

Fig. 4 Percentage of scientific areas affected by olive waste water during the period 2000 - 2019

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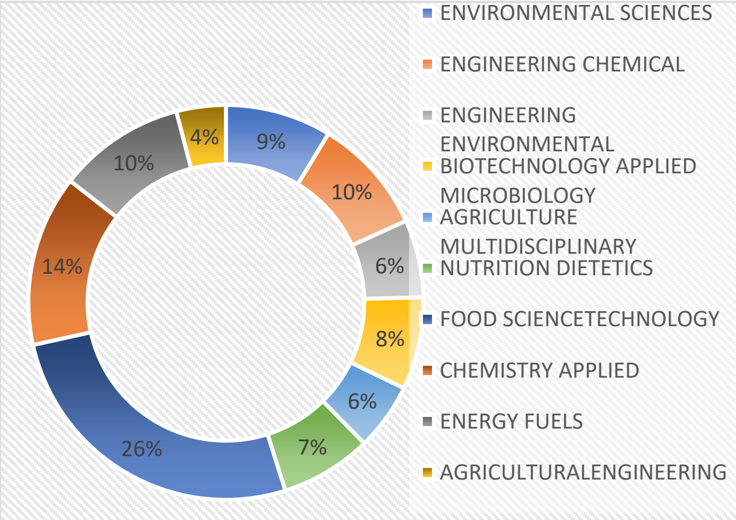

Fig. 5 Percentage of scientific Areas Affected in olive pomace during the period 2000 - 2019

2 ENVIRONMENTAL IMPACT OF OLIVE OIL WASTE:

The discharge of effluents from olives mills is a major problem, especially in the Mediterranean basin countries. It is liquid waste (vegetable water) that causes serious environmental damage. The lack of suitable treatment methods leads oil mill owners to release these waters in the wild without any control or discharge these toxic substances into the sewer system that can contaminate natural environments (soil and surface water).

2. 1 Water pollution:

The (OMWW) are not very degradable because of their high organic load[17]. These olive oil wastes are most often released into natural receptors, streams, without any prior treatment and strongly affect the quality of these surface waters. The very high organic matter load prevents these waters from self-purification and the pollution can spread over very long distances and for a period that can last up to five months[31].

The OMWW discharged directly into streams can cause eutrophication problems. This was confirmed by excessive proliferation of *Nitzschia* palea and *Gomphonema* parvulum. The highest proliferation of these species witnessed during the winter[5].

Discarding olive wastewater in the sea may cause physiological disturbances to aquatic species by triggering oxidative stress. As it has been proved by a work done on mussels *Mytilus galloprovincialis*[42]. In this work, it was shown that exposure of these species to a dose of 0.1 or 0.01% of olive wastewater for 5 days showed significant alterations of stress indices in their tissues. Specifically, a decrease in neutral red retention (NRR) assay duration, inhibition of acetylcholinesterase (AChE) activity, and a significant increase in micronucleus (MN) frequency and DNA damage was detected in haemolymphs / haemocytes and gills, compared to tissue values of control molds.

2. 2 Soil pollution:

The process of extracting olive oil produces large quantities of waste that can be reused in land-based agriculture. The OMWW are part of this waste. With acidic pH, high salinity and high levels of polyphenols, olive waste waters are potentially dangerous for the environment.

Toxicological effects of waterweed on *Eisenia fetida* worms have been demonstrated using biomarkers of neurotoxicity (AChE), oxidative stress (LPO, CAT), and genotoxicity (comet assay). Raw olive wastewater has caused high dose of earthworm mortality and measurable biochemical and cellular effects at lower doses. The main cause of these effects is probably the high level of polyphenols in these wastes specifically those come from two-phase mills[37].

Irrespective of the negative effect of OMWW on soil as being stated previously, some other works confirm that irrigating maize fields with OMWW for a long time has added nutrients such as carbon, nitrogen, phosphorus and potassium to the soil[43], [44]. Moreover, in some works it has been stated that after two months of spreading this kind of water on fields no activation or inhibition of soil microflora activity was observed. This has been explained by reorganization or degradation of the OMWW[45].

The differences observed in the results of the verified works are may be due to the method of the treatment and the application of OMWW, the physicochemical characteristics of the soil as well as the type of crop.

3 PROGRESS OF THE WORKS ON THE TREATMENT OF OLIVE WASTEWATER:

In order to avoid the pollution caused by olive oil waste, researches have focused on the development of numerous treatment processes based on physical, physicochemical and biological processes.

In this synthesis, we tried to compare the efficiency of these different techniques according to the rate of abatement of COD, total polyphenols (PT) and turbidity. And on the neutralizing power of the pH of OMWW (Table I).

3. 1 Physicochemical methods:

Although the physicochemical processes are not the most suitable for purifying residual water of high organic load, but they are applied especially for the rejections of the agri-food industries including olive mill wastewater (OMWW). These techniques that we have mentioned, are often integrated with other techniques[17], [18], [31]. Various processes have been tested, including decantation, flocculation coagulation, electrocoagulation, adsorption, percolation, and filtration.

3. 1. 1 Decantation:

It is a natural process of mechanical separation which is done under the action of gravity, of several immiscible phases of which at least one is liquid. The decantation of 100 liters of OWW can release up to 19.7 liters of oil with a yield of 19.7% which proves that this water is still rich in oily residues[46]. This method generates significant quantities of solid pasta and presents the problem of release of undesirable odors[11].

3. 1. 2 Coagulation-flocculation:

Coagulation-flocculation is one of the most widely used methods for removing suspended and colloidal material from effluents. In this technique the effluents are treated by adding a coagulant and a flocculant. In this sense, many coagulants have been used namely aluminum sulphate[47]; lime[12], iron sulphate[15], [47], calcium hydroxide[47], chitosan[14] or commercial flocculants such as **FLocudex CS-51**. These treatments remain a partial solution, producing a large quantity of sludge which must be provided with means of treatment and recovery. They are more considered as pretreatment solutions to reduce the organic load of effluents[13].

3. 1. 3 Electrocoagulation:

This technique removes metals, solids and colloidal particles, as well as soluble inorganic pollutants from margins by applying electrical current between pairs of electrodes that can be aluminum[17], [21], or iron and stainless steel[48]. This elimination is done by liberation of the species of highly charged polymeric metal hydroxides. These species neutralize the electrostatic charges of the suspended solid portion and the oil droplets to facilitate agglomeration or coagulation and the resulting separation of the aqueous phase. The treatment leads to the precipitation of certain metals and salts and the formation of a supernatant[17].

The effectiveness of electrocoagulation treatment effluents with high organic load depends on the choice of the type of electrode, the distance between them, the imposed current density and the pH of the medium. Thus, a 98% and 80% reduction of COD and PT was recorded successively using aluminum electrodes[7], [15]. While the use of iron cells allowed the abatement of 92.6% of COD, 76.2%[49]. It turned out that this technique is promising because it is efficient and less demanding (12 to 14 KWh / d).

3. 1. 4 Adsorption:

Among the most used adsorbents are activated carbon[22] and active clay bentonite[20]. However, the use of activated carbon is not only a relatively expensive material, but after saturation, the regeneration cost for its reuse is high. Recently, an adsorbent at the base of olive pomace has been suggested with a cost lower than50 $ / ton against $ 4500 / ton for activated carbon[50]. As well as the use of banana peel as an adsorbent to dephenilize the OMWW, causing a reduction of 60% to 88% of polyphenols [21].

3. 1. 5 Filtration and ultrafiltration:

It is a method of separating the suspended solid portion of watermills using gravel and sand-based filters[21] or clay and eucalyptus sawdust[51]. The technique has an abatement rate of up to 90% and 92% COD and PT successively (Table I). If the waters are very charged, we can proceed to ultrafiltration which depends on applying a hydrostatic force of a few bars. This filtration which goes through a membrane makes it possible to sort different solutes by molecular sieving. The size of the molecules retained by ultrafiltration ranges from 0.002 µ to 0.1 µ according to the chosen membrane with an abatement rate of 74% and 31.5% of PT and COD successively[22]. Regardless of this, the above-mentioned technique still has some undesirable results such as the acidification of OWW.

3. 1. 6 Advanced oxidation:

The advanced oxidation allows a very significant reduction of the polluting load of the olive wastewater. It is a process that relies on the formation of very reactive chemical entities that will break down the most recalcitrant molecules into biologically degradable molecules or mineral compounds. These compounds are known for their rapidity in decomposing organic and inorganic compounds and in their elimination of OWW toxicity[52]. Advanced oxidation processes have been widely studied for the treatment of waste from agri-food industries including ozonation[53], photo-fenton oxidation[54], [55], photocatalysis[56], electrochemical oxidation and wet oxidation[57]. The literature suggests that these methods are promising. However, it should be noted that their application on an industrial scale is very limited because of their high cost of investigation[23], [58].

3. 2 Bioremediation of olive mill wastewater:

Several treatment techniques based on the biodegradation

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and decomposition of organic vegetable constituents by micro-organisms (bacteria and fungi) have been examined to reduce margins toxicity[81], [82]. These techniques are considered the most healthy, effective and less expensive methods[58]. There are two main categories of biological treatment: aerobic treatment and anaerobic treatment:

3. 2. 1 Aerobic treatment:

Aerobic treatment relies on oxygen to facilitate microbial degradation of organic matter in OWW. By involving bacterial species such as *Bacillus pumilus*[81], *Pseudomonas putida*[71], and *Ralstonia sp*[83]. These species are characterized by a high capacity of withstanding the acidic pH of margins and their high concentration of polyphenols which must not exceed 800 mg / ml for optimal yield[83]. Other studies have used fungal species such as *Yarrowia lipolytica*[82]. This species is able to reduce COD up to 75% by producing citric acid, lipids and proteins that can be used as animal feed or commodity[84]. Another species is known for its resistance to the high content of OWW in phenolic compounds which is Lentinula edodes. This alga can reduce 75% of polyphenols[85]

3. 2. 2 Anaerobic treatment

The anaerobic treatment is carried out in the absence of oxygen by a series of anaerobic microorganisms, mainly bacteria. This treatment has been widely applied to limit the toxicity of agro-food discharges[58]. According to the results, the effectiveness of the biological treatment was high, equal to 90% COD and 80% elimination of the total phenolic compounds[23]. It has the advantage of reducing energy requirements, sludge production and also allows the recovery of energy in the form of biogas (methane)[75], which can be used for energy production[24]. The major problem encountered during the anaerobic treatment of effluents with a high organic load, in particular phenolic compounds, is the inhibition of the process of digestion of bacteria, the release of unpleasant odors, as well as the production of a significant quantity of spent sludge at the end of anaerobic digestion[11]

4 PROGRESS OF WORKS ON THE VALUATION OF OLIVE MILL WASTE WATER:

In this chapter we will underline some applications of the OWW which are summarized in table II

4. 1. Spreading on the fields:

The richness of OWW in fertilizer has prompted some researchers to test their direct application to the land (Sorghum cultivation)[86]. The results showed an improvement in the organic matter, total nitrogen and mineral content in the margins-treated soils, in proportion to the increase in applied doses. In addition, the microbiological activities of soils have been greatly enhanced compared to control soils. Besides, this study shows that the use of raw OWW blocks the seed germination of Sorghum bicolor[86]. Other studies have shown that the effect of spreading depends on the texture and the chemical nature of the soil[45]. Today, organic waste spreading is one of the most successful techniques for ensuring stable organic matter in agricultural soils and

TABLE I. METHODS AND TECHNIQUES FOR TREATING OLIVE MILL WASTE WATER

PROCESSES	TECHNIQUES	EFFICIENCY	REMARKS	REFERENCES
PHYSICAL	Distillation	92% reduction in COD and 95% in phenolics	Strong acidity of the distillate product	[51], [59], [60]
	Natural evaporation	Ineffective	Sludge production, undesirable odors	[11], [61]
	Forced evaporation	Ineffective	Sludge production, energy consumption, unwanted odors	[11]
	Ultra-nanofiltration, reverse osmosis, electrodialysis	90% reduction in COD, low energy consumption	Membrane piling and sealing, very high investment and operating costs	[22], [62]–[64] ; [65] ; [66] ; [11]
PHYSICOCHEMICAL	Coagulation-flocculation	Ineffective	Production of large quantities of sludge	[12] ; [14] ; [15] ; [13], [67]–[69]
	Advanced oxidation, ozonation, photo-fenton oxidation, photocatalysis, electrochemical oxidation, wet oxidation	High COD depletion rate (80%) and 99.8% of phenolic compounds	High cost of investigation	[70] ; [71] ; [25] ; [72] ; [48], [73]
	Electrocoagulation	High COD (98%) and phenolic compounds (80%) abatement rate depending on test conditions and type of electrodes	Processing rate can reach 9.6L / d with consumption of 12 to 14 KWh / m3	[7] ; [16] ; [15], [17], [48], [74]
	Adsorption	67-95% reduction of phenolic compounds	Generally the regeneration cost of activated charcoal is high	[21] ;[18] ;[19]; [20]
BIOLOGICAL	Anaerobic treatment	Energy production (biogas), reduction of waste / effluent toxicity	Clearance of unpleasant odors, production of a significant amount of spent sludge, inhibition of the process of digestion of bacteria for effluents with high organic loads, especially phenolic compounds	[23], [26] ; [24], [75]–[77]
	Aerobic treatment	Significant reduction in COD	Sludge generation, excessive oxygen consumption	[78] ; [25] ;[73], [79] [80]

nutrients for plant growth[27], [28], but it must be applied precautionarily because the excessive spreading results in destruction of the receiving environment or destruction of the soil microflora[87].

4. 2. Co-composting:

The composting process is defined as a process similar to that of the natural humification of organic residues into humic substances in soils[30]. To speed up the composting process, organic compost (OWW, wastewater) is added to the compost to provide additional moisture, nutrients and microorganisms. The use of OWW in the humidification of compost contributed to the aerobic biodegradation of all substrates, as indicated by the measurement of physicochemical parameters as a function of time until its maturation after 12 weeks[17]. The use of OWW in co-composting after solar drying gave a compost rich in nutrient 3.5% N, 1% P and 6.5% K with a low phenol load 2.9g / Kg. The use of this product for the cultivation of pepper has approved the fertility, which has been found to be similar with commercial NPK fertilizers[29]

4. 3. Seed germination:

The olive mill wastewater (OMWW) can be used to stimulate the germination of wheat, lettuce and bean seeds after dilution (50%, 25% and 12.5%) or after treatment with lime at different dilutions. With a germination rate of up to 100%[88]. The best germination result of Campbell 33 tomato seeds was obtained after dilution to 25% or after treatment of OWW by electrocoagulation at different dilution[7]

4. 4. The extraction of certain molecules with biological activities:

Margins are rich in phenolic compounds[8]. These compounds have biological activities, namely the antioxidant activity which can prevent the rancidity of fatty acids and the oxidation of proteins during food storage[35], the anti-inflammatory and cosmetic activity[33]. Margins have been shown to inhibit the proliferation of certain bacteria such as Staphylococcus aureus and Escherichia coli at an inhibitory concentration of 0.125 mg / ml and a bactericidal effect of OWW begins at 0.5 mg / ml[34]. In another work, activities associated with the potential preventive properties of a phenolic compound extracted from OWW (A009) were studied on three in vitro models of prostate cancer cells (PCa). A009 was able to inhibit proliferation, adhesion, migration and invasion of PCa cells[32].

4. 5. The production of energy:

Anaerobic digestion has also been the subject of numerous studies aimed at energy recovery from OWW to limit their pollution. In this context, many researchers have shown that these discharges can be considered as an important source of energy[24], [37], [89].

TABLE II: OLIVE MILL WASTE WATER RECOVERY TECHNIQUES

TECHNIQUES	EFFICIENCIES	REMARKS	REFERENCES
SPREADING	Contributions of stable organic matter to agricultural soils and nutrients to plants	Excessive spreading causes destruction of soil microflora	[87] ; [27] ;[28]
ENERGY PRODUCTION	Biogas production	_	[36] ;[89] ;[24] ;[37]
EXTRACTION OF PHENOLIC COMPOUNDS	Valorization as food additives and pharmaceutical thanks to their antioxidant power	_	[34] ;[33] [9], [10]; [35] ; [32]
COMPOSTING	Production of an organic amendment	_	[30] ;[17] ; [29], [90]–[93]

5 PROGRESS OF WORK ON THE TREATMENT AND RECOVERY OF THE OLIVE POMACE:

The characterization of the pomace has shown that it is mainly composed of phenolic compounds, carbohydrates, organic acids and mineral nutrients distributed in various ways depending on the process used and agronomic practices[8].
Despite the richness of the olive cake in organic and mineral materials, the work that has been done on this material remains limited. Among these works we quote:
The possibility of lightening the concrete by introducing crushed olive pomace[94]
The extraction of oleanolic and maslinic acids (the main triterpene acids identified in olive pomace oil obtained from "Alpeorujo" stored)[95]. Polyphenols and terpenes known for their antioxidant activity[39], [40] and a source of lipases[41].
In an other work, it has been reported that olive pomace can be used as a partial substitute for fish oil in fish feed, thus improving its cardioprotective properties[96].
Olive pomace represents a promising alternative as a secondary fuel resource in cement kilns because the presence of its ashes in the crude mixtures did not affect the mineralogical composition of the clinker nor the physicomechanical properties of the final cement produced[38]

6 CONCLUSION:

This literature review has allowed us to conclude that there are many processes that have been developed for the treatment of these effluents. These methods are based on physical, chemical and biological processes. Although there is not yet a perfect solution to eliminate water pollution, some processes seem to be more effective.

However, the choice of treatment or recovery channels depends on the decision-makers, the cost of investigation, the added value of the product that results from the treatment, the flow of effluents to be treated, the objective of the treatment and environmental impact of the sector.

Among these methods, we have found that electrocoagulation (98% of COD and 80% of phenolic compounds abatement rate) and advanced oxidation (80% of COD depletion rate and 99.8% of phenolic compounds abatement rate), are the most effective for the treatment of OWW. However, the high cost of these techniques makes their industrialization difficult to achieve. A renewable energy source can be used to lower treatment costs. Several techniques can be integrated for more effective treatment.

About the pomace, it has been suggested to use it as an adsorbent for the treatment of the olive mill waste water. It is an integrative technique with which we can manage the two-olive wastes.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflict of Interest

The authors declare that there is no conflict of interest regarding publication of this manuscript.

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