Olive mill wastewater treatment in Jordan: A Review

Abeer Al Bawab1,2*, Noor Ghannam1, Saida Abu-Mallouh2, Ayat Bozeya2, Rund A. Abu-Zurayk2, Yazan A. Al-Ajlouni3, Fida’a Alshawawreh1, Fadwa Odeh1, Muna A. Abu-Dalo4*

1 Chemistry Department, The University of Jordan, Amman, 11942, Jordan,
2 Hamdi Mango Center for Scientific Research, The University of Jordan, Amman, 11942, Jordan
3 New York University Abu Dhabi, Saadiyat Island Abu Dhabi 129188, United Arab Emirates
4 Department of Applied Chemical Sciences, Jordan University of Science & Technology, Irbid-22110, Jordan

drabeer@ju.edu.jo
maabudalo@just.edu.jo

Abstract The environmental impact of olive mill wastewater (OMW) pollution is a public concern. OMW contains high levels of phenols, organic compounds, chemical oxygen demand (COD), biological oxygen demand (BOD), microorganisms, nutrients, and toxic compounds. The treatment of OMW has been investigated by many researchers in the Mediterranean region, using several treatment techniques to remove contaminants from OMW. These techniques include chemical, biological, physiochemical, and biophysical techniques. Surfactants and some adsorbents were used in chemical techniques, anaerobic and aerobic in biological techniques, while the combined treatment methods used Electroosmosis, ozonation and electrocoagulation processes as physiochemical methods, and ultrasonic irradiation combined with aerobic biodegradation as biophysical method. The effects of OMW, whether treated or untreated, have been evaluated on both plants’ growth and soil properties. The treatment methods as well as the environmental impact of OMW in Jordan were summarized in this review.

1. Introduction

Olive oil production is considered as a major industry in Jordan with approximately 21.5 thousand tons per year. The process of olive oil extraction generates around 200,000 m³ of olive mill wastewater (OMW), which also known as Zebar [1,2].

OMW generally characterized as acidic dark colored water which contain ample amounts of organic compounds (COD, BOD), total dissolved solids (TDS), and high concentration of phenols and potassium [3-5]. In Jordan, according to a recent study carried out by Azzam et al., 2015, OMW contains high phenolic contents (360 mgL⁻¹), high COD (40,000 mgL⁻¹), high total suspended solids TSS (20,700 mgL⁻¹), high total dissolved solids TDS (27,700 mgL⁻¹), high concentrations of cations and anions (Ca 2.11 mgL⁻¹, Cu 0.94 mgL⁻¹, K 25.18 mgL⁻¹, Mg 1.90 mgL⁻¹, Na 0.68 mgL⁻¹, Zn 33.43 μgL⁻¹) and a low pH value, estimated to be approximately 4.6.

Majority of mills in Jordan discharge their OMW without any treatment due to lack of knowledge, complexity, and costs of treatment or transport to a landfill site. The treatment of OMW is not only desirable, but also necessary. This is because if untreated OMW is discharged into rivers or streams, it will deplete the dissolved oxygen, thereby causing fish death and other undesirable effects, including malodorous gases produced due to the decomposition of organic materials. Other compounds also contribute to the toxicity of OMW, these include for instance, lipids and humic-like substances. Untreated OMW also contains numerous pathogenic, microorganisms and toxic compounds (especially phenolic compounds), which affects human health negatively.
On the other hand, the disposal of OMW to the environment has beneficial effects, as they are rich in nutrient, especially potassium. Potassium can stimulate plant growth and could be used as soil conditioner, biomass fuel, compost, or as starting material to obtain valuable products such as antioxidants, enzymes, and biogas fuel [6-10]. Additionally, olive mill solid residue offers a potential for the removal of heavy metals by biosorption [11].

Several methods have been proposed to reduce the environmental impact of OMW. These methods are classified into physical, chemical, and biological methods, including floatation and sedimentation [12], sand filtration [13,14], ozonation [15], membrane filtration [16-18], neutralization with addition of acid advanced chemical oxidation (Fenton reaction) [19-20], adsorption by activated carbon, aerobic and anaerobic digestions [9,10,21], and magnetic nanoparticles [22], surfactant enhanced aquifer remediation technology [23], and modified surfactant [24,25].

In recent years, there has been growing interest to develop environmentally friendly and cost-effective treatment methods for OMW. Upon these bases, the aim of this review is to explore various methods and technologies for treatment, recovery, and reuse of OMW in Jordan.

2. Treatment methods of OMW

In Jordan, many researches in the literature have focused on attempting to find efficient and cost-effective treatment method for OMW. Previously, various methods have been tested to achieve this aim. These methods have drawn on aspects from chemical, biological, and physical sciences, and have often used an inter-disciplinary approach combining more than one method.

2.1. Biological treatment methods

Biological treatment methods used for OMW treatment are ultimately aerobic activated sludge and anaerobic digestion, focusing heavily on the ability to remove organic matter and inorganic nutrients. These processes are reliable, environment friendly, and cost-effective [26-30].

2.1.1 Anaerobic

During anaerobic processes, biomass waste is converted to biogas (by bacteria in the absence of oxygen) and compost [29]. The produced biogas (mainly methane) has an economic importance, as it can be used to generate heat and electricity [31].

Up-flow anaerobic sludge blanket reactor (UASB) is considered as the most popular bioreactors to treat agro- industrial wastewaters characterized with high organic load [32].

The impact of the removal efficiency and the observed biomass yield on nutrient requirements for anaerobic treatments of OMW was studied by Ammary, 2004 using laboratory scale anaerobic sequencing batch reactors. The researcher found more than 80% of COD removal could be achieved by varying the COD: N: P ratio. In addition, the observed biomass yield was about 0.06 kg volatile suspended solids (VSS) per kg of COD degraded. Thus, it was concluded that OMW have sufficient nitrogen and phosphorous concentrations, and the addition of such nutrients was not necessary [33].
These results were again supported when the characteristics of Jordanian OMW were investigated [34]. In this study, the use of anaerobic sequencing batch reactor (ASBR) was also tested for its suitability for onsite treatment, and a COD removal of 83% was achieved.

Furthermore, Sobhi et al. conducted a study in 2007 aiming to identify the most efficient anaerobic treatment for OMW. It was found that the (UASB) reactor achieved COD removal efficiency in the range 75–85% at a Hydraulic Retention Time (HRT) of 5 days with an influent COD concentration of about 40 gL⁻¹ and Organic Loading Rate (OLR) of 7–8 g CODL⁻¹ d⁻¹ [35].

As the COD is a major OMW pollutant, an UASB was constructed in 2009 by Khatib et al. to reduce COD level under the specified parameters. After eight months of operation program, it was shown that 46%–84% of COD removal was achieved and that the organic load was reduced from 27,000 mgL⁻¹ to less than 5,000 mgL⁻¹, which permits the direct discharge of OMW municipal wastewater treatment plants. Biogas (mainly methane) was also collected and used [36].

### 2.1.2 Aerobic

Naturally occurring microorganisms work as an effective factor in wastewater treatment. These microorganisms include bacteria, fungi, protozoa and other microbes. They ultimately live on wide variety of complex compounds contained in wastewater. In aerobic treatment processes, bioreactors are used to provide microorganisms with optimum growth conditions by the addition of soluble oxygen as well as organic and nitrogenous compounds. During this process, microorganisms act as decomposers that are responsible for the oxidation of complex organic compounds to return them to simple forms of carbons that could safely be returned to the environment [37]. Additionally, other microorganisms are capable of producing biofuel by oxidizing phenolic compounds [38].

For example, the fungus Pleurotus sajor-caju was used to treat OMW and reduce its toxicity, making it suitable as a fermentation medium by yeast to produce ethanol. The maximum ethanol production (14.2 gL⁻¹) was obtained after 48 hour of yeast fermentation using 50% diluted OMW that was thermally processed and pretreated with P. sajor-caju [38].

Another study using P. sajor-caju was performed to evaluate its effect on OMW treatment. In this study, it was reported that the OMW could be a suitable media for P. sajor-caju ligninolytic enzymes, which is involved in the reduction of toxic compounds in OMW. It was possible using this fungus to significantly reduce phenolic compounds content (from 5.7 to 2.1 gL⁻¹) along with enzymes secretion and action. By the end of this treatment process, a complete degradation of some phenolic and non-phenolic compounds was achieved and the color of OMW was reduced by more than 60% [39].
2.2. Chemical treatment methods

2.2.1 Adsorption

Continuous packed bed of zeolite was used to investigate the possibility of OMW treatment by Abu Al-Haija, 2008. Pilot-scale column was designed and operated under various feed flow rates using different zeolite particles sizes. Jordanian mined zeolite packing the bed was treated and activated as a part of this investigation. The results showed a high reduction in phenolics compound and COD using the smaller zeolite particles size, the removal was 98% for both terms and decreased with increasing particle size as well as operating condition [40].

Sedimentation, filtration, and flotation experiments were conducted by Gharaibeh et al., 2008 to treat OMW, using Azraq bentonite (AB), reddish volcanic tuff (RVT), lime (CaO), aluminum sulfate (alum), ferric chloride, and sodium carbonate. Two parameters were studied; OMW turbidity removal and COD removal. The results revealed that alum, lime and AB have a high efficiency in turbidity and COD removal, while RVT, ferric chloride, and sodium carbonate did not have significant effects. The turbidity removal using alum, lime and AB was 95%, 99%, and 96%, respectively, while the COD removal was 65%, 69%, and 37.5%, respectively. However, given its high cost it was not recommended to use alum, whereas lime and AB are inexpensive and locally available and could be used easily [41].

The adsorption of phenols and organic compounds was also investigated. Different pretreatment processes of OMW such as sedimentation and filtration were used. This was followed by batch adsorption process using activated clay as the adsorbent. The maximum reduction of organic load was about 71%, while the removal of phenols was about 81% [42]. In another study, Azzam et al., using activated carbon as the adsorbent, the maximum adsorption capacity for the tested concentrations of activated carbon is reached in less than 4 hour. Phenols were about 94% removed, while for organic matter in general it reached about 83% [43]. This was followed by batch adsorption process using activated carbon for one, two and three stages process. The pretreatments showed an effective reduction of COD, phenols, and total solids content. It was more effective to use a two stage countercurrent adsorption process compared to a one-stage adsorption process, however, the use of a three-stage adsorption showed only a slight improvement compared to the two-stage process. For example, a treatment protocol composed of a three-stage countercurrent adsorption process using activated carbon of concentration 24 gL\(^{-1}\) of OMW was able to reduce the COD from 60000 mgL\(^{-1}\) down to 22300 mgL\(^{-1}\), while phenols were reduced from 450 to 15 mgL\(^{-1}\) [44].

Naturally occurring Jordanian clay was also tested as an adsorbent material for OMW treatment, while COD and phenols levels were monitored. Natural clay was calcined at different temperatures (350–550°C) and some were further treated with different HCL concentrations (1, 3, and 5 M-HCL solutions) at 85°C. The treatment was done in batch experiment to identify the optimum conditions, which were then used for a continuous packed bed treatment system. In this investigation, both COD and phenols of raw OMW were reduced by 10–20% in batch experiments. Additionally, the reduction in COD in the continuous packed bed experiment reached 50%. Such reductions are considered relatively significant, considering the large amounts of organic matter involved (COD~40,000 mgL\(^{-1}\)) [45].
2.2.2 Coagulation advanced oxidation

Lafi et al., 2010 proposed the use of a combination of two treatment processes for the removal of organic pollutants from the OMW. These processes included a single coagulation step in which they used $\text{Al}^{3+}$ and $\text{Fe}^{3+}$ ions as coagulant, followed by a single advanced oxidation process, AOP, step. The study used UV, $\text{O}_3$, $\text{O}_3$/UV and $\text{H}_2\text{O}_2$/UV depending on the operating time. For the AOPs, the concentration of the chemical oxygen demand (COD) was measured in the effluent of the treated wastewater. It was found that the COD removal achieved using $\text{Al}^{3+}$ was 54%, while for $\text{Fe}^{3+}$ ions it reached 58% at pH 9. However, when advanced oxidation process was used alone, the percent COD removal ranged between 10 and 39%. The percent removal of the COD concentration was achieved using the combined processes, coagulation and AOPs ($\text{O}_3$ 90%, $\text{O}_3$/UV 95% and $\text{H}_2\text{O}_2$/UV 94%) [46].

2.2.3 Surfactants

Surfactant enhanced aquifer remediation (SEAR) technology was applied to reduce phenolic compounds content in OMW using modified surfactant, which is a novel extended surfactants sodium polypropylene oxides sulphate of the type (branched hydrocarbon chain)-(propoxyl group)-(sulphate). Phase diagram was modeled using phenol as a standard compound. This study indicated that the needed amounts of surfactants to extract the phenol can be determined using the phase behavior of phenol, water, and proposed surfactants [23].

Another study in the literature proposed new modified surfactants. These included surfactants combined with cationic hydrotropes tetra butyl ammonium bromide (TBAB) in different molar ratio, namely: sodium linear alkyl- polypropylene-oxide-polyoxyethylene- sulfate (X-AES), Sodium polypropylene oxides sulfate (L167–4s), and Linear alkyl benzene sulfonate (LAS). Similarly, the amount of the needed surfactant to extract the phenol was determined using the three-dimensional phase behavior of the phenol, water, and the proposed surfactants. The results of this study showed that only polypropylene oxides sulfate (L167–4s) modified surfactant can be used for real sample which showed a 95 % COD removal [24].

Moreover, Al Bawab et al., 2018 used cost effective media of two types of granular carbons. Granular Activated Carbons non-oxidized (GAC), and Granular Activated Carbons oxidized (GAC-OX), particles impregnated with surface active materials (nonionic surfactants (TWEEN 80, Span 20, Span 80, Span 85, and Brij 93) at three different concentrations (10, 30, 55 mM) for OMW treatment. Experimental results for 24 hours showed that 10 mM nonionic surfactant concentration was the optimum concentration used in media preparation, which is below Critical Micelles Concentration (CMC). The results of percent phenol removal using the 10 mM impregnated media GAC-OX-Span 20, GAC-OX-Span 80, GAC-OX-Brij93, GAC-OX- TWEEN 80, and GAC-OX-Span 85, were 83.64, 79.55, 73.97, 66.54, and 31.96%, respectively. While, on GAC for GAC-Span 20, GAC-Brij 93, GAC-Span 80, GAC-TWEEN 80, and GAC-Span 85, were 82.56, 67.65, 64.68, 33.45, and 29.36%, respectively. The results of media, which was soaked with OMW for 15 days, showed an increase in percent removal of phenol using of GAC-OX-Span 20, GAC-OX-Span 80, and GAC-OX- Brij 93, were 92.94, 91.45, and 78.07% respectively. While for GAC-Span 20, GAC-Span 80, and GAC-Brij 93, percent removal was 97.03, 86.25, and 79.25% respectively. After 15 days, the percent removal was relatively reduced.
Finally, the media were tested at different temperatures (7, 25, and 35°C). The results showed that there was no change in percent removal with temperature. Additionally, the shaking condition was not significant. Therefore, these media provide an economical treatment strategy [25].

2.3. Physicochemical treatment methods

2.3.1. Electroosmosis process

Electro-osmosis dewatering (EOD), is a technique that extracts water by placing a colloidal material between two electrodes. This technique is based on the electrostatic effect of the electrochemical double layer that is formed at the particle water interface of the colloidal material. Near the solid-liquid interface the polarization of water molecules causes an electrical double layer to form [47].

Additionally, this process that OMW goes through affects its pH, voltage, current and bed height, and these effects have been investigated independently. This process resulted in converting the pH of the water from acidic to basic and reduction in COD and total dissolved solid (TDS) levels. EOD process was enhanced by increasing the voltage or the current and by the addition of alum or an electrolyte. Furthermore, it was reported that sodium chloride is the most effective electrolyte in EOD process [48].

2.3.2 Ozonation and electrocoagulation processes

OMW could be successfully treated by applying electrocoagulation accompanied with ozonation. This was manifested by Bani Salameh 2015, where the electrocoagulation process resulted in 82.5% and 47.5% removal of TSS and COD respectively after 70 minutes at 45mA/cm², by using coupled iron–aluminum electrodes. The subsequent ozonation reveals high levels of organic compound elimination [49].

2.4. Biophysical treatment methods

A combined ultrasonic irradiation and aerobic biodegradation treatment was used for the reduction of toxic phenolic compounds from greenish black (GB) and dark brown (DB) OMW by Al-Qodah et al., 2014. Several parameters were tested for their effect on phenols, COD and BOD degradation. Parameters tested include the duration of ultrasonic irradiation, ultrasonic power intensity, and ultrasonic frequency. The results revealed that total phenols were degraded by 81% when it was exposed to 90 min to ultrasonic field. It was demonstrated that phenol degradation followed a first-order kinetics model with rate constant, \( k \), of 0.0083 and 0.0077 min\(^{-1}\) for (GB) and (DB) OMW samples, respectively. In the aerobic degradation step, the COD consumption followed the Grau kinetic model. The order of COD degradation rate, is about 1.13 and 1.27, whereas, the Grau kinetic constant, \( k = 0.0218 \) and 0.0149 h\(^{-1}\) for GB and DB OMW, respectively. The maximum removal of COD was approximately 80% [50].

2.5. Biochemical treatment methods

Different stage processes of advanced oxidation with ozone (O\(_3\)), an aerobic biodegradation treatment and photodegradation by UV radiation was used for COD removal from OMW. For both single-stage treatment of O\(_3\) and two-stage treatment of O\(_3\)/UV, the COD remains quite high. However, a combination of biological and UV/O\(_3\) process for the OMW treatment showed an alteration in the reduction of the COD. Biodegradation of UV/O\(_3\) pretreated OMW had the highest removal levels while the percent of COD removal reached approximately 91%. The decay of chemical oxygen demand follows a first-order models for advanced oxidation and pseudo first-order models for biodegradation processes as the kinetic study showed [51].
3. Environmental impact of OMW

Given the high abundance of organic substances, phenols, and other toxic compounds and microorganisms in OMW, OMW has significant polluting concerns on both environmental and biological systems. Often, OMW is collected in open ponds and left to dry throughout the summer season, thus producing pollution to underground water and the soil surface in addition to unpleasant odor [52, 53].

Consequently, there is an urgent need to raise awareness, improve technical skills and change the public’s attitude toward this issue. Change must be advocated for and policy-makers must focus on intervening to utilize and manage OMW, especially given its economic value [54]. However, other studies did not recommend the use of either treated or untreated OMW in agriculture because of the high dissolved solids concentration within it, and it was advised to use natural occurring materials such as bentonite and lime as a solution to treat OMW [41].

Generally, to achieve sustainable agricultural practices, OMW applications should be carefully adapted, while taking into consideration several issues, including climatic conditions, soil properties, and biological systems.

In an experiment carried out by Rousan, 2007, barley crops were treated with OMW for two years during 2005 and 2006. During this time period, several factors were studied, including dry matter, leaf area index, plant population, and grain yield at harvest. The results of dry matter and grain yield were comparable when treated and untreated OMW used, however the untreated OMW produced necrosis of barley leaves and led to slow secondary stems emission [55].

The effect of untreated OMW on soil properties, oil quality, plant performance, oil content, and fruit yield was studied by Ayoub et al., 2014. Fresh OMW was applied on the soil surface of orchards planted with 15 years old olive trees (cultivar Nabali Muhassan) at five different application rates; 0 Lm⁻², 5 Lm⁻², 10 Lm⁻², 20 Lm⁻² and four equal doses of 20 Lm⁻². It was indicated that OMW could increase the concentrations of potassium, organic matter, phenols, and total microorganisms in treated soil when compared with control soil, but that was not considered a negative effect. Moreover, this soil could serve as an efficient fertilizer. Furthermore, and regarding plant performance, it was found that fruit set, shoot growth, fruit yield and photosynthesis were significantly enhanced [56].

Soil is considered an indicator for the health and integrity of ecosystem, thus its measurement provides valuable insights in research. Notably, any contamination or pollution in the soil will negatively affect plant growth and other vital living systems. Biological soil quality index (QBS-ar) was used as a valuable tool to study the impact of OMW on the physiochemical and biological properties of soil. Wahsha et al., 2014 used this tool to study soil samples collected from northwest Jordan. The results showed a decrease in the biological quality of soil samples polluted with OMW according to QBS-ar due the alteration in chemical and physical characteristics of soil microhabitat [57].

OMW contains a substantial amount of oil, which might affect soil thermal conductivity. Thus, an experimental work investigated the effect of OMW on thermal conductivity of three soil types (sandy clay, silty clay and clay) using heating and cooling methods under laboratory conditions. The results showed a decrease in thermal conductivity for all soil tested at different concentrations of OMW (20%-100%). Thermal conductivity ranged from 0.89-0.71, 0.77-0.63, 0.80-0.57 W/mK for sandy clay, silty clay, and clay, respectively. Higher thermal conductivities were obtained from heating methods [58].
4. Summary and Conclusion

The purpose of this exploratory review study was to provide an overview of the OMW treatment methods and the environmental impact of each method, with specific focus of the application of these methods in Jordan. In summary, there is no ideal solution in the area of treatment of OMW. Rather, several environmentally friendly and economically viable solution are presented and proposed. These methods include biological treatment (anaerobic and aerobic), chemical treatment (adsorption, surfactants), physicochemical treatment (Electroosmosis, ozonation and electrocoagulation) and biophysical treatment (combined ultrasonication irradiation and aerobic).

Previously, studies in the literature examined the efficiency of these methods by using phenols removal percentages and/or COD removal percentage as a measure of efficiency. The best removal percentages using different methods were reported in the literature as follows:

- Anaerobic: 80% COD removal.
- Aerobic: 63% Phenols removal.
- Adsorption: 98 % for both COD and phenols removal.
- Surfactants: 95% COD removal.
- Ozonation and electrocoagulation: 47.5% COD removal.
- Combined ultrasonication irradiation and aerobic: 80% for both COD and phenols removal.
- Chemical treatment methods (adsorption, surfactants) are found to show highest efficiency of treatment of OMW (98%, 95%).

As a conclusion from the literature reviewed above, it is plausible to state that OMW are important from an environmental perspective. Ultimately, OMW are currently considered to be a waste in most situations. However, with proper treatment, it can be become a recovered resource. The current study advances our knowledge of OMW treatment method by providing a rigorous review of the current methods, and providing a setting for potential combination of different methods. Future research should continue to explore various treatment methods and combine different approaches to utilize the most appropriate approach in Jordan.

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