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Valorization of olive mill wastewater using physical, chemical, and biological treatment

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ABSTRACT

The olive oil industry is considered one of the industries that pollute the environment. Because it is accompanied by solid and liquid waste that is difficult to deal with and causes much damage to the environment. Therefore, this research aims for the safe handling and disposal of high organic loads and phenolic compounds present in this type of industrial wastewater. The advanced oxidation represented by the Fenton reaction was used to get rid of phenolic compounds and organic loads. The concentration of organic loads expressed by chemical oxygen demand (COD), biochemical oxygen demand (BOD), and total suspended solids (TSS) in industrial wastewater was 71,000; 12,280 and 21,265 mg/L. The phenol concentration reached 8,754 mg/L. The Fenton reaction removes COD, BOD, TSS, and phenols by 93.6%, 76.5%, 99.7%, and 99.966%, respectively. The aerobic sand filter (ASF) was used as a secondary treatment stage. The ASF effectively removed COD, BOD, TSS, and phenol by 79%, 87.6%, 60%, and 97.7%, respectively. The combination of treatment using the advanced oxidation reaction (Fenton oxidation) with the ASF has proven to be very effective in treating olive mill water.

Keywords: Advanced oxidation; Fenton reaction; Aerobic sand filter; Biological treatment

1. Introduction

Olive trees (*Olea europaea* L.) are evergreen trees belonging to the family Oleaceae and comprise 30 genera. Although the genus Olea includes 30 species, *O. europaea* L. is the most common and the only species in this genus that bears edible fruits. Olive is an economically important crop with global consumption of olive oil and olives reaching more than 2×10^6 tons in 2010. The ten leading olive-producing countries are Spain, Italy, Greece, Turkey, Tunisia, Morocco, Syria, Egypt, Portugal, and Algeria [1]. During oil production, large amounts of by-products including raw olive cake, twigs, and leaves accumulate; the latter accounts for about 10% of the weight of olives during the production process. Olive leaves have no special use in this process, thus, increasing the cost to the producers, although these leftovers are certainly a cheap, available and rich source of phenolic compounds [2].

Olive oil production has deep roots in the history of the Mediterranean region. The centuries-old tradition represents a very important asset for many countries in the region, not only in terms of culture and health but also in terms of wealth. Olive oil production is increasing over time due to the increase in olive tree cultivation and customers' high demand [3]. Over the past few decades, olive oil represents a critical healthy dietary trend worldwide, because it is considered an important resource of essential fatty acids and antioxidant agents in the human diet [4].

Approximately 11×10^6 hectares of olive farms were cultivated in 2015 worldwide. About 50% of these farms'

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cultivations are in the EU region, especially Spain, Italy, and Greece. Total olive fruit production in the EU accounted for 13.24 × 10^6 tons. Spain is the major producer with 7.87 × 10^6 tons 60% of total EU production. The annual global olive oil production is around 3.32 × 10^6 tons, with 72% produced in Europe. Other Mediterranean countries' producers are Turkey, Syria, and Tunisia (6%) each and Morocco (4%), Jordan (3%), and Lebanon (1.5%). Other smaller producers outside the Mediterranean basin in Asia, Africa, and America are growing with 15%, 12%, and 2% of worldwide olive production, respectively [4]. Olive tree products have social and economic worth in the lives of people living in Mediterranean nations because of their nutritional content.

The process of production of olive oil results in the generation of olive mill wastewater (OMWW) [5–7]. In Mediterranean nations where the generation rate is extremely high and concentrated in a short amount of time, its treatment is a significant environmental challenge (winter season). Despite efforts to deploy two-phase, clean extraction technology, the yearly OMWW production is predicted to be over 30 × 10^6 m^3 [8]. Production of olive oil using a three-phase machine produces a huge amount of solid as well as liquid wastes. One ton of olive fruit requires an equal amount of water. The resultant wastes are olive mill wastewater (OMWW), and husk. The treatment of OMWW is becoming a serious environmental problem, due to its dark color and high organic matter content which consists mainly of polysaccharides, sugars, polyphenols, polyalcohols, proteins, organic acids, and oil [5,9]. OMWW's composition varies greatly depending on the type of olive used, how ripe the fruit is, and whether it was extracted using a press or a centrifuge [10]. OMWW typically consists of 83%–94% water, 4%–16% organic molecules, and 0.4%–2.5% mineral salts in terms of weight. The organic fraction includes, among other things, 2%–15% of phenolic compounds, which are divided into low- and high-molecular weight compounds (tannins, anthocianins, etc.) [11,12]. Due to its high organic load [13] and presence of phenolic compounds that are phytotoxic and antibacterial and resist biological destruction [14,15], the disposal and treatment of this liquid waste is the key issue for the olive oil business. These qualities make it impractical to dispose of OMWWs in urban sewage treatment plants. Recycling of olive mill wastewater (OMWW) should be encouraged in Mediterranean nations with low organic matter soils and active desertification processes [4].

This work aims to reduce the environmental threats via the treatment of OMWW and find beneficial uses for olive leaves.

2. Materials and methods

2.1. Olive mill wastewater

The OMWW was collected from an olive mill in Sharkia City, Egypt. Characterization of OMWW includes chemical, biochemical oxygen demand (COD, and BOD), total Kjeldahl nitrogen (TKN), total phosphorus (TP), total phenol (TPh) and total suspended solids (TSS). The chemical oxygen demand (COD) was carried out using digester LoviBond (Model RD 125), and UV-Vis spectrophotometer (Edinburgh, DS5). The analyses were carried out according to standard methods for the examination of water and wastewater [16].

2.2. Treatment scenario

The advanced oxidation (Fenton reaction) was used as the primary treatment process followed by aerobic sand filter (Fig. 1). Ferrous sulphate and hydrogen peroxide mixture was used to carry out the Fenton reaction.

2.3. Microbial activity of OMWW extract

This step was carried out according to different previous work [17,18]. The OMWW sample of 10 mL was dried at 45°C to concentrate the volume for the next step of extraction. The dried OMWW sample was macerated with 70% ethanol (1:10 w/v) for 72 h at ambient temperature with occasional shaking. The extract was filtered with Whatmann filter paper (No. 1). Ethanol was evaporated at 45°C, and the extract was tested for microbial activity against bacteria (*E. coli* and *Staphylococcus aureus*) and fungus (*Aspergillus* sp. and *Penicillium* sp.) [19,20].

2.4. Chemical treatment

The advanced oxidation process (Fenton reaction, FR) was used as an option for the primary treatment process. The optimum ratio of ferrous sulfate ($FeSO_4$) and hydrogen peroxide (H_2O_2) was selected according to previous work [5].

2.5. Biophysical treatment

An aerobic sand filter (ASF) was used as a secondary treatment step for the chemically treated OMWW effluent. The ASF (Fig. 1) was a cylindrical shape with dimensions of 2 m in length and 0.23 cm internal diameter. The AFS was packed with fine sand (0.4–0.6 mm). The material of ASF was PVC and fractionated into four compartments as mentioned by El-Khateeb et al. [21,22].

3. Results and discussion

The OMWW is characterized by its greenish color, which turns into black brownish which is viewed as a negative characteristic. Another negative impact of this wastewater is its extremely high content of organic compounds, which is reflected by the high values of COD. The organic loads expressed by COD and total phenols (TPh) were 71,000 and 8,754 mg/L (Table 1), respectively. Such wastewater is hardly biodegradable as the BOD/COD ratio ranged from 0.17 to 0.18. The BOD/COD ratio was found to be lower than that obtained by Haouas et al. [23]. Biological treatment of OMWW is not a suitable solution at ordinary conditions. This attributed to the presence of high concentration of TPh (8,754 mg/L) and the deficiency of nitrogen and phosphorus content (415 and 91 mg/L, respectively). Consequently, Zakoura et al. [24,25] studied the anaerobic biological treatment of OMWW at elevated temperature (mesophilic and thermophilic conditions).

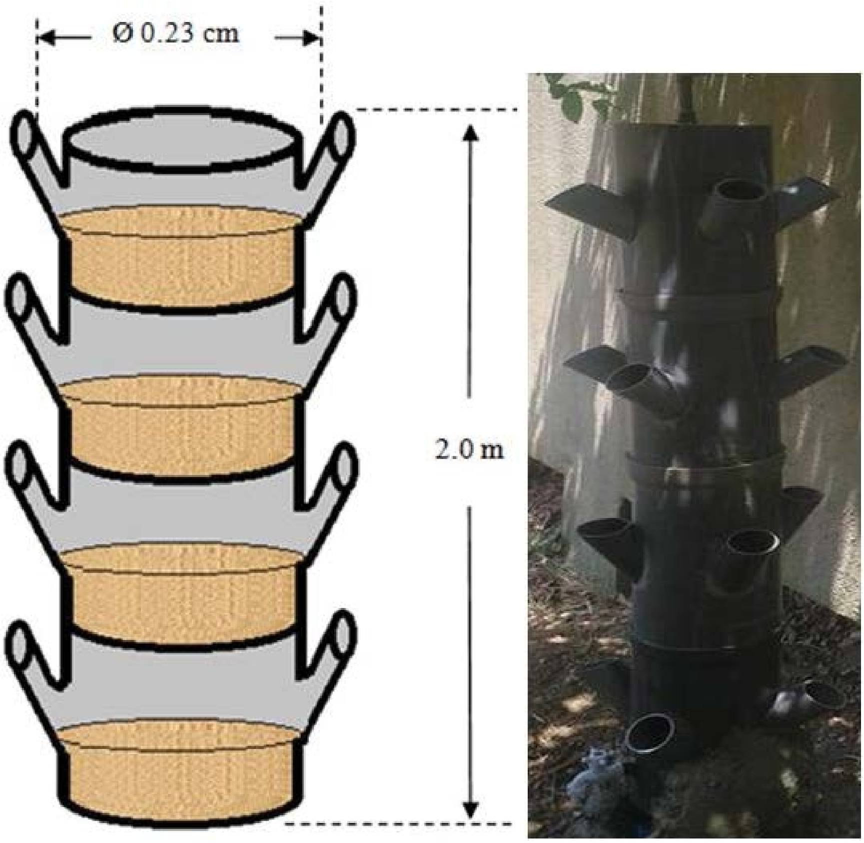


Fig. 1. Schematic diagram for the ASF.

Table 1
Characterization of OMWW collected from the Sakaka City region

Parameter	Unit	Min.	Max.	Average
pH		3.9	5.3	4.8
COD	mgO_2/L	65,000	78,000	71,000
BOD	mgO_2/L	11,255	14,025	12,280
BOD/COD	–	0.17	0.18	0.173
Settleable Matter	mL/L	Colloidal	Colloidal	Colloidal
TSS	mg/L	12,550	25,000	21,265
TKN	mgN/L	356	450	415
TP	mgP/L	89	96	91
Oil&Greases	mg/L	3,250	5,560	4,890
TPh	mg/L	4,526	13,256	8,754

3.1. Microbial activity of the extract

Figs. 2 & 3 and Tables 2 & 3 show the inhibition zone of the OMWW extract. The diameter of the inhibition zone in the control (amoxicillin antibiotic for bacterial strains test) and OMWW extract was 5 mm (stem and leaves) for *E. coli*. *Staphylococcus aureus* was more highly affected than *E. coli*. Roila et al. [26] attributed the inhibition of the growth of bacteria to the presence of diverse phenols with various biological effects. The OMWW is phytotoxic; however, it possesses antimicrobial activity due to the phenolic compounds present in the wastewater [26]. These findings were supported by the low biodegradability of the OMWW as depicted in Table 1 by the BOD/COD ratio. Consequently, the use of biological treatment as the primary treatment for OMWW was not feasible.

3.2. Chemical treatment

The FR was used to remove the toxic and hardily biodegradable organic matter in the OMWW. The optimum operating conditions were kept constant as follows: pH 3; $COD:H_2O_2$ = 1.0:1.1; $Fe^{2+}:H_2O_2$ = 1:50 [5]. Table 4 shows the performance of the FR for the treatment of the OMWW. The excellent COD removal efficacy of Fenton's reagent over short contact times suggests that this approach has a lot of potential for agro-industrial wastewater decontamination [27–29].

Fenton's reaction is an advanced oxidation process in which ferrous ions (Fe^{2+}) catalyze the breakdown of hydrogen peroxide (H_2O_2) to produce OH radicals:

$$H_2O_2 + Fe^{2+} \rightarrow OH^{\bullet} + Fe^{3+} \quad (1)$$

The OH radicals attach to the organic compounds leading to the reduction of COD as well as the TPh [29,30]. The concentration of the TPh was greatly reduced from 8,754 to 3 mg/L with a corresponding removal rate of

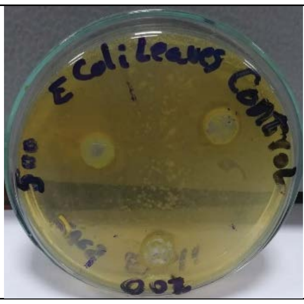
E.coli	***Staphylococcus aureus***
Stem	
	
Leaves	
	

Fig. 2. Bacterial activity of OMWW extract.

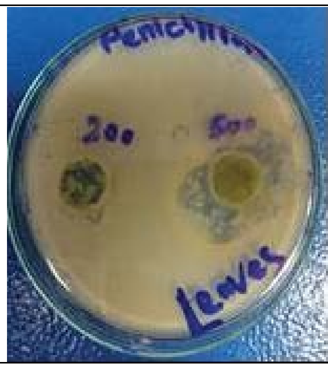
Asperagillus SP.	*Penicillium Sp.*
Stem	
	
Leaves	
	

Fig. 3. Mycological activity of OMWW extract.

Table 2
Inhibition zone (mm) of bacterial strains as affected by OMWW extract

Tested substance (μL)	Stem		Leaves	
	E. coli	*Staphylococcus aureus*	*E. coli*	*Staphylococcus aureus*
Control (amoxicillin antibiotic)	5	5	5	5
OMWW extract at 200 μL	5	5.1	5	9
OMWW extract at 500 μL	5	5.8	5	6

Table 3
Inhibition zone (mm) of fungal strains as affected by OMWW extract

Tested substance (μL)	Stem		Leaves	
	Aspergillus sp.	*Penicillium* sp.	*Aspergillus* sp.	*Penicillium* sp.
OMWW extract at 200 μL	2.41	3.11	0.76	3.2
OMWW extract at 500 μL	6.21	4.21	3.36	17.2

Table 4
Performance of the combined chemical and bio-physical OMWW treatment

Parameter	Unit	OMWW	FR effluent	%*R* by FR	ASF effluent	%*R* by ASF
pH		3.12	7.8		7.6	
COD	mgO_2/L	71,000	4,525	93.6	950	79.0
BOD	mgO_2/L	12,580	2,951	76.5	365	87.6
BOD/COD	–	0.18	0.65		0.38	
TSS	mg/L	21,265	65	99.7	26	60.0
TKN	mgN/L	415	36	91.3	18	50.0
TP	mgP/L	91	34	62.6	28	17.6
Oil & Greases	mg/L	4,890	3	99.9	0.3	90.0
TPh	mg/L	8,754	3	99.966	0.07	97.7

99.966% (Table 4). This leads to the increasing biodegradability of the OMWW as indicated by the BOD/COD ratio that increased from 0.18 to 0.65. The chemically-treated OMWW was suitable for the biological treatment process. These results were in good agreement with that reported by El-Gohary et al. [5], Al-Enazi et al. [7], Hodaifa et al. [31], and Lin et al. [32].

3.3. Biophysical treatment

The chemically-treated effluent was fed directly to the ASF for further treatment. Table 4 summarizes the characteristics of OMWW and the different treatment effluents.

The organic loads expressed by COD, BOD, and TSS were reduced greatly by 79%, 87.6%, and 60% with corresponding residual values of 950, 365, and 26 mg/L, respectively. The BOD/COD ratio decreased from 0.65 to 0.38. This was due to the depletion of the biodegradable part of the organic loads within the ASF treatment unit.

Fig. 4 shows the performance of the ASF for removing COD, BOD, and TSS. The TSS was more affected than COD and BOD, on the other hand, the BOD was the least affected as indicated by the trend line. This may be attributed to the degradation of the biodegradable part of the organic loads. These results are in good agreement with that obtained by El-Gohary et al. [5], El-Khateeb et al. [33], and Benamar et al. [34].

Fig. 5 reveals the performance of the ASF for the removal of TKN, TP, oil&grease, and TPh. The oil&grease and TPh were highly affected by the combined FR and the ASF treatment. The TKN, TP, oil and grease, and TPh were reduced from 36, 34, 3, and 3 to 18, 28, 0.3, and 0.07 with a removal rate of 50%, 17.6%, 90%, and 97.7%, respectively. The high removal efficiency by the ASF reactor was due to the combined mechanism of filtration as well as the biodegradation of organic matter by the attached microorganisms on the surface sand. The influent wastewater enters the ASF and trickles down over the attached growth (biofilm) that has formed on the packing material's surface. This movement sucks the air from the sidearms of the reactor. The surface of the sand material encourages biofilm adhesion. The ASF treatment of industrial wastewater results in a net reduction of organic loads (COD and BOD). Separation of liquid solids, on the other hand, was critical; hence the reactor bottom features a settling area. The ASF's high porosity was designed to prevent clogging and increase airflow [21,22,35,36].

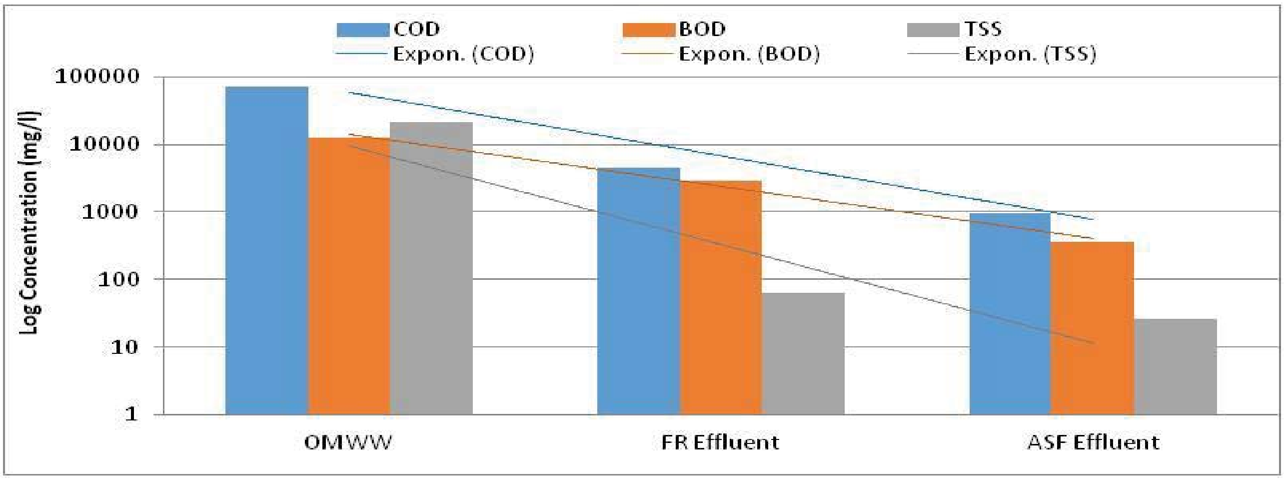


Fig. 4. Fate of organic loads (COD, BOD, and TSS) during the treatment process.

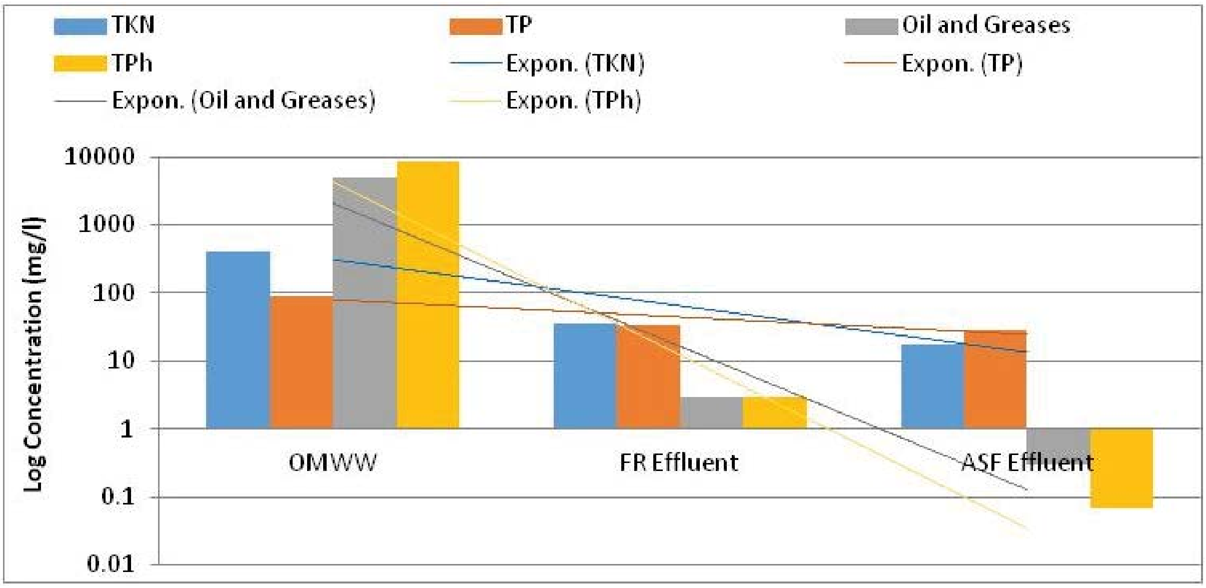


Fig. 5. Fate of TKN, TP, Oil&Greases, and TPh during the treatment process.

Table 5
Potential reuse of OMWW treated effluent

Parameter	OMWW treated effluent	Ministerial Decree 44/2000	Degree of treatment permitted for agriculture use [37]			
			Grade A	Grade B	Grade C	Grade D
pH	7.6	6–9.5				
COD (mgO_2/L)	950	1,100				
BOD (mgO_2/L)	365	600	15	30	80	350
TSS (mg/L)	26	800	15	30	50	300
TKN (mgN/L)	18	100				
TP (mgP/L)	28	25				
Oil&Greases (mg/L)	0.3	100				
TPh (mg/L)	0.07	0.05				

3.4. Valorization of the treated effluent

Table 5 shows the potential reuse of the treated effluent. The treated OMWW effluent was found to be complying with the National Regulation [37,38]. There are penalties and fines to be collected in case of violation of any of the criteria set out in Table 5 [1]. Consequently, the application of this technique saves olive oil production factories from paying fines by discharging the treated effluent on the sewage networks. There are no possibilities to reuse of treated effluent in irrigation the codes 501 [2].

4. Conclusions

The olive oil industry is of great importance in Mediterranean countries. This industry produces effluents containing high concentrations of organic loads, phenols, and oil and greases. Disposal of this waste without proper treatment poses significant environmental risks. This residue contains phenolic compounds that hinder the biological treatment process. Therefore, an advanced oxidation process (Fenton reaction) was used to remove the phenolic compounds and eliminate the existing high organic loads. The advanced oxidation process was used as the initial treatment stage and then the vertical sand filter was used. The results showed that the removal of organic loads represented by COD, BOD, and TSS was 79%, 87.6%, and 60%, respectively. While the phenolic compounds and oil and grease reached 97.7% and 90%, respectively. These results proved that the combination of the Fenton reaction and aerobic sand filter effectively removed organic loads and got rid of phenolic compounds in the final treated effluent and reducing their harmful impact on the environment.

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