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Original research article

Enhancing Olive Mill Wastewater Treatment: A Study of pH Influence in Multi-Step Column

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ABSTRACT

Olive mill wastewater (OMW), a byproduct of olive oil production, presents significant environmental challenges due to its high organic load and non-biodegradable polyphenols. In Libya, untreated OMW is frequently discharged into the environment, impacting soil and water quality. This study evaluates the pollution potential of OMW and examines a multi-step treatment process utilizing natural materials. The system comprised three columns: the first filled with iron slag and sand, and the second and third with activated carbon prepared from dried olive leaves. Parameters such as pH, electrical conductivity (EC), total dissolved solids (TDS), turbidity, biochemical oxygen demand (BOD_5), and total phenols (TP) were monitored. Results demonstrated significant improvements in water quality under basic pH conditions, with reductions in EC (61.9%), TDS (62.9%), and turbidity (99.8%) and complete removal of BOD_5. However, the reduction in TP was lower, showing a decrease of 43.2%. In contrast, the treatment without pH control achieved lower removal efficiencies for EC (50.5%), TDS (51.5%), and turbidity (97.1%), but a higher reduction in TP (80.9%). The lower TP removal at basic pH is due to phenol deprotonation, weakening adsorption. Acidic conditions improved turbidity removal but were less effective for EC, TDS, and BOD_5. pH influences adsorption and microbial activity, impacting treatment efficiency. This multi-step approach offers a promising solution for OMW treatment, reducing its environmental impact and enabling reuse in irrigation.

Keywords: Activated carbon; Biosorbents; Environmental pollution; Olive mill wastewater; Wastewater treatment

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1. Introduction

Olive oil production, a cornerstone of Mediterranean economies, generates substantial quantities of olive mill wastewater (OMW), a byproduct characterized by its high organic load, acidic pH, and recalcitrant phenolic compounds. While the cultivation of Olea europaea has ancient roots in the Mediterranean Basin, the industry's substantial environmental footprint is a contemporary concern. This region, responsible for approximately 98% of global olive oil production, faces the challenge of managing the substantial volumes of OMW generated by its agro-industrial processes. The two- or three-phase extraction systems employed in olive oil production yield an estimated 20 to 160 liters of OMW per 100 kg of olives, highlighting the scale of the environmental issue. The complex composition of OMW, including polyphenols, sugars, tannins, and lipids, many of which are resistant to biodegradation, contributes to its significant ecotoxic and phytotoxic effects. The effluent's high biological oxygen demand (BOD) and chemical oxygen demand (COD) further exacerbate its environmental impact, as it can contaminate water bodies, inhibit plant growth, and disrupt ecosystems [1-4].

Despite its well-documented toxicity, OMW is frequently disposed of improperly, either through direct discharge into freshwater systems or application to agricultural soils without adequate treatment, exacerbating environmental degradation. The effluent's antimicrobial properties and high levels of phenolic compounds render it resistant to natural degradation, compounding the environmental risks. Mediterranean countries thus face a dual challenge: substantial water consumption and the harmful environmental impact of OMW. The lack of consistent regulations regarding OMW disposal further complicates management efforts, leading to the common use of natural evaporation ponds or discharge into urban sewage systems [5-7].

Given the escalating water scarcity challenges in the Mediterranean region, exacerbated by climate change and rising demand for freshwater, the proper treatment and management of olive mill wastewater (OMW) are paramount. Effective treatment methods not only mitigate the environmental risks posed by OMW but also support water reclamation, ensuring sustainable access to water resources for future generations. Addressing the environmental footprint of the olive oil industry, particularly in relation to OMW, is critical to both ecological conservation and economic sustainability in the region [8-10].

The complex composition and high pollution load of olive mill wastewater (OMW) have posed a significant environmental challenge. Various methods have been proposed to manage OMW, including evaporation ponds, which, despite their cost-effectiveness, suffer from drawbacks such as odor development, leakage, and high land requirements, particularly in regions with low evaporation rates [11]. Other approaches include aerobic treatment, anaerobic digestion, composting [12], biological, physico-chemical, and advanced oxidation methods [13-15]. However, a universally adopted, environmentally safe, and economically feasible solution remains elusive [16]. The seasonal nature of olive oil production and the isolated locations of mills further exacerbate the challenges of OMW treatment [17]. Recent research has explored innovative approaches such as thermal conversion of OMW into biochar for use as a biofertilizer [18]. However, the high water content of OMW and the costs associated with drying and pyrolysis limit large-scale application [19]. Activated sludge (AS) processes, while widely used in aerobic treatment due to their simplicity and cost-effectiveness, require microbial acclimation to reduce the toxicity and chemical oxygen demand (COD) of OMW [20]. To enhance biodegradation, researchers have also investigated techniques like bioaugmentation and acclimatization of microbial communities [21]. Given the complexity of OMW, integrated treatment methods combining physical, biological, and chemical technologies are considered the most promising approach for effective management of this effluent [22].

Adsorption has emerged as one of the most promising and efficient techniques for treating olive mill wastewater (OMW), primarily due to its low maintenance costs, high efficiency, and ease of operation compared to other technologies, which are often expensive, time-consuming, and sometimes ineffective. While several treatment methods have been explored, such as aerobic and anaerobic processes, they are frequently not economically viable due to the seasonal nature of olive oil production and the isolated locations of olive mills [16, 17]. In contrast, adsorption has effectively removed hazardous organic and inorganic compounds from wastewater and groundwater, including toxic phenolic compounds in OMW. Various adsorbents have been studied, including industrial waste materials and natural biosorbents like dried sludge, coal, activated charcoal, olive leaves powder, orange peels, palm pith carbon, and beet pulp [23-29]. Moreover, synthesized adsorbents such as amine-modified mesoporous silica and activated carbon have been specifically employed for the removal of phenolic compounds from OMW [30]. Despite these advancements, most research has concentrated on simple phenols, while more complex natural phenolic compounds have been less thoroughly investigated. Thus, ongoing research into low-cost, efficient adsorbents remains crucial for tackling the challenges associated with OMW treatment.

In Libya, untreated OMW is often discharged into the sewerage system, while solid waste is dumped on nearby land, endangering soil, surface water, and groundwater resources. This study aims to assess the pollution potential of OMW by analyzing key parameters such as pH, conductivity, total dissolved solids, and biological oxygen demand (BOD). Additionally, it explores an efficient treatment method using successive columns of natural materials, specifically a mixture of iron slag and activated charcoal (AC), to enhance water quality. This treatment presents a promising approach for rendering OMW safe for irrigation use.

2. Materials and Methods

2.1 Samples collection and preservation

Olive mill wastewater (OMW) samples were collected from three olive oil mills located in Zliten; a city in the northwestern part of Libya and situated 160 km east of the capital, with five replicates from each site. To ensure accurate measurements of physical properties, the samples were obtained immediately after the pressing process. The samples were then stored in tightly sealed plastic bottles under refrigeration at 4 °C to maintain low temperatures and prevent any unintended reactions among their components

2.2 Analytical measurements

Key physical and chemical properties of fresh OMW samples, including pH, density, electrical conductivity (EC), total dissolved solids (TDS), turbidity, dissolved oxygen (DO), acidity, biological oxygen demand (BOD_5), and total phenolic content, were characterized before and after pretreatment.

Total phenolic compounds were quantified using the Folin-Ciocalteu method. Briefly, a 2.5 mL aliquot of 0.2M Folin-Ciocalteu reagent was mixed with 0.5 mL of the sample, followed by a 5-minute incubation in the dark. Subsequently, 2 mL of a 75 g/L sodium carbonate solution was added, and the mixture was incubated for one hour in the dark. The absorbance was measured at 765 nm using a UV-Vis spectrophotometer (Jenway model 6300) [31]. The total phenolic content was expressed as mg gallic acid equivalents in liter.

A pH meter (HACH MP-6, Hach Company, Loveland, CO, USA) was used to determine pH, while electrical conductivity and total dissolved solids were measured with an HACH EC MP-6 (Hach Company, Loveland, CO, USA). Turbidity in NTU (Nephelometric Turbidity Unit) was measured using a HACH Turbidimeter 2100Q (Hach Company, Loveland, CO, USA). Acidity was quantified using volumetric acid-base titration techniques [32].

2.3 Preparation of activated carbon

Activated carbon was prepared from dried olive leaves through a chemical activation process. Concentrated phosphoric acid (H3PO4) was added to the leaves for 24 hours, followed by drying in an oven at 250 °C and subsequent burning at 600°C in an oxygen-free environment. The resulting activated carbon was washed repeatedly with distilled water until the solution reached neutrality [33].

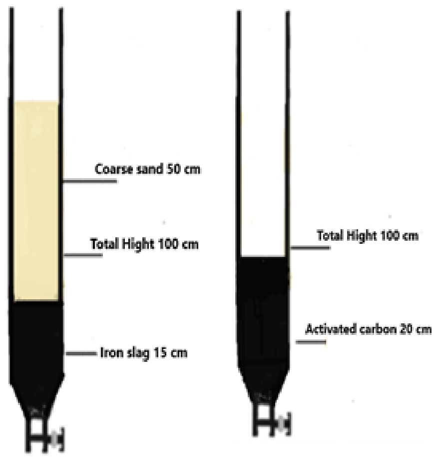

Fig. 1. Olive Mill Wastewater Treatment Model Components.

Fig. 2. The effect of treatment on OMW color.

2.4 Treatments of OMW

The treatment process involved a multi-step approach using columns made of polyvinyl chloride with an inner diameter of 3 cm and a height of 100 cm, as depicted in Fig. 1. The first column was filled with two successive layers: a 15 cm layer of iron slag and a 50 cm layer of coarse sand that had been washed thoroughly with distilled water. A thin layer of cotton was placed at the column's bottom to prevent material loss. OMW samples were then passed through the column, and the treated water was collected in a cup at the bottom for analysis. The effect of pH on the treatment process was also investigated.

The treated water from the first column was subsequently transferred to a second column filled with a 20 cm layer of activated carbon. A thin layer of cotton was placed at the bottom of this column, and the water was passed through it, collecting in a cup at the bottom. This treated water was then transferred to a third column, also containing a 20 cm layer of activated carbon. The necessary analyses were performed on the final effluent, and the impact of pH on treatment efficiency was studied. Fig. 2 illustrates the color change of the sample during the various treatment stages.

For the treatment conducted under controlled pH conditions, 1 M hydrochloric acid (HCl) and 1 M sodium hydroxide (NaOH) were used to adjust the pH of the OMW samples. For the acidic pH treatment, 1 M HCl was gradually added to the OMW sample until the pH reached approximately 3. For the basic pH treatment, 1 M NaOH was added to the sample until the pH reached approximately 9. These adjustments were carefully monitored to maintain the desired pH levels throughout each treatment stage.

2.5 Statistical analysis

The statistical analysis of the experimental data was performed using Microsoft Excel. Each parameter was measured in triplicate, and the results are presented as the mean of these three replicates. The standard deviation (±) is provided to indicate the variability of the measurements.

3. Results and Discussion

Fresh OMW samples were collected from a local olive mill and analyzed for their physicochemical properties as shown in Table 1. OMW is characterized by a dark brown to black color and a strong olive oil smell. Its complex chemical composition is due to the presence of vegetation water and olive fruit tissues. The collected OMW was characterized for various parameters, including pH, density, EC, TDS, turbidity, DO, acidity, BOD_5, and total phenolic content, prior to treatment with adsorption materials.

A three-column sequential flow system was used to treat the second sample. Physical and chemical properties were measured after the first column and at the end of the treatment, as shown in Table 2.

3.1 Analysis of pH

The pH values of the OMW samples analyzed in this study ranged from 4.88 to 5.05, with an average of 4.98±0.21 (as shown in Table 1). The acidic pH values (shown in Fig. 3) are consistent with findings from numerous previous studies, which reported pH values ranging from 3.8 to 6.5 [34]. For instance, Al-Essa [35], who used acid-activated bentonite for olive mill wastewater treatment, reported a pH of 4.63. Similarly, Achak et al. [32], utilizing wheat bran as a biosorbent for phenolic compound removal, recorded a pH of 5.06, while Aladham [36], employing activated olive stones for polyphenol removal, found pH values between 4.9 and 5.0. Víctor Ortega [37], who applied ion exchange technology for olive mill wastewater treatment, reported a pH of 5.4, and Al Bsoul et al. [28], using titanium oxide nanoparticles for adsorption, observed a pH of 4.9. Aktas et al. [38] recorded pH values ranging from 4.5 to 4.9. In con-

Table 1. Physical-chemical parameters of the raw OMW samples (mean ± standard deviation, n = 3).

Parameter	Unit	OMW1	OMW2	OMW3
pH	-	5.05±0.25	4.88±0.21	5.02±0.19
Electrical Conductivity (EC)	μS/cm	11290±50	18810±910	11740±485
Total dissolved solids (TDS)	mg/L	7200±320	12290±314	7700±35
Turbidity	NTU	11800±574	2270±112	8950±247
Density	g/mL	1.0082±0.03	1.0104±0.01	1.0076±0.02
Dissolved Oxygen Demand (DO)	mg of O_2/L	2.03±0.10	5.78±0.30	2.44±0.08
Biological Oxygen Demand (BOD_5)	mg/L	50.00±1.5	2.80±0.12	327.00±13.4
Acidity	%	0.97±0.04	0.99±0.06	2.21±0.11
Total phenol content	mg/L	214.43±8.5	173.30±7.6	126.10±5.3

Table 2. Main characteristics of OMW2 sample, before and after treatment (mean ± standard deviation, n = 3).

Parameter	Unit	Before treatment	After column 1	End treatment
pH	-	4.88±0.21	6.98±0.33	7.02±0.29
Electrical Conductivity (EC)	μS/cm	18810±910	12590±610	9314±210
Total dissolved solids (TDS)	mg/L	12290±314	8057±165	5960±98
Turbidity	NTU	2270±112	245±9.5	66.1±3.3
Dissolved Oxygen Demand (DO)	mg of O_2/L	5.78±0.30	1.50±0.07	0.14±0.01
Biological Oxygen Demand (BOD_5)	mg/L	2.80±0.12	0.00±0.0	0.00±0.0
Total phenol content	mg/L	173.30±7.6	168.2±5.4	33.07±1.3

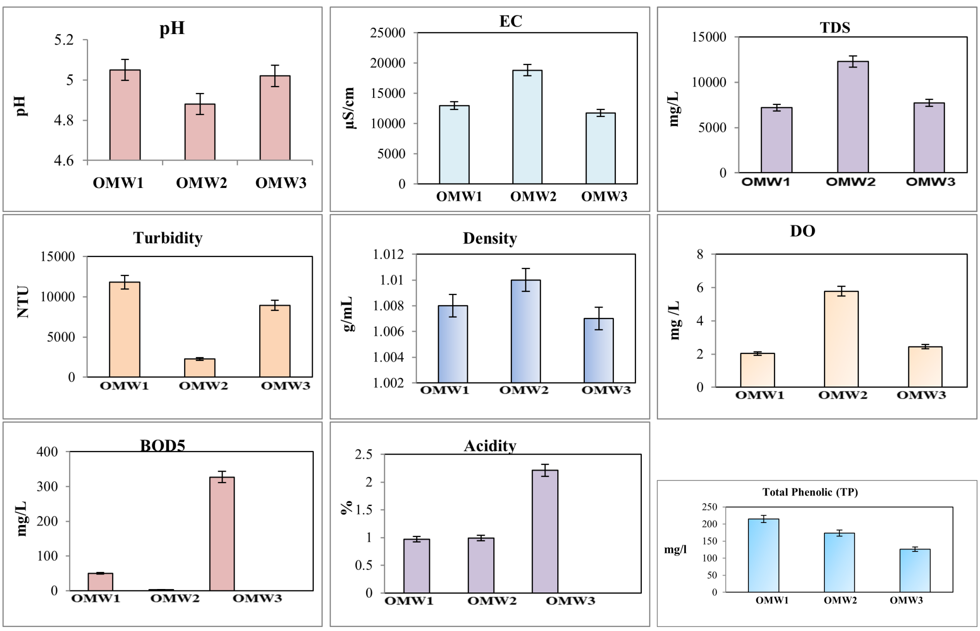

Fig. 3. Physical and chemical properties of untreated OMW samples, Data presented as mean ± SD (n = 3).

trast, Ait-Hmane et al. [39], who applied a combined treatment involving multi-soil-layering ecotechnology and adsorption on activated carbon/lime, reported a relatively higher pH of 7.66. Conversely, Aly et al. [40], employing a zeolite-based low-cost

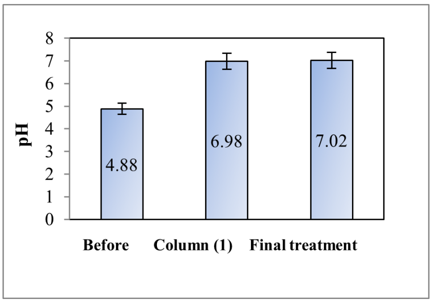

Fig. 4. Effect of different treatment steps on pH of OMW, Data presented as mean ± SD (n = 3).

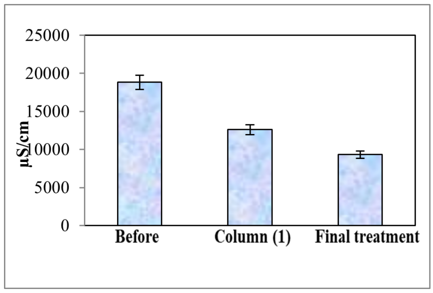

Fig. 5. Effect of different treatment steps on EC of OMW, Data presented as mean ± SD (n = 3).

treatment method, recorded a lower pH of 3.8.

Following the treatment of the sample (OMW2) with a sand and iron slag column, the the pH levels in this study transitioned from acidic to near-neutral, reaching 6.9 after initial treatment, and further increasing to 7.02 following activated carbon column treatment (Fig. 4). This pH elevation is attributed to the reduction in phenolic compounds and fatty acid concentrations during the treatment process [6].

These findings align with previous research. Víctor Ortega [37] reported a similar increase, with post-treatment pH values of 7.8. Mekki et al. [41] documented an average pH of 8.1 ± 0.2, and Hanafi et al. [42] observed a pH of 7.53. Fiestas et al. [43] also confirmed a shift to neutral pH, with values ranging from 6.5 to 7.0.

In contrast, Aly et al. [40] reported post-treatment pH values between 5.1 and 6.2, while Al-Essa [35] observed a range of 5.08 to 5.74. These studies indicate that their treatment processes were less effective in achieving neutral pH levels compared to the current study and several others.

3.2 Analysis of electrical conductivity

Before treatment, the electrical conductivity (EC) of the olive mill wastewater (OMW) samples (OMW1, OMW2, OMW3) ranged from 10,000 to 20,000 μS/cm, with an average of 14,503.3 ± 510 μS/cm (Fig. 3). These values align closely with those reported by Jahangiri et al. [44] (14,600 μS/cm), Al-Essa et al. [35] (19,890 μS/cm), Aharonov-Nadborny et al. [45] (11,000 μS/cm), and Galiatsatou et al. [34] (13,000 μS/cm).

In contrast, higher EC levels were reported by Ben Sassi et al. [46] (50,000 μS/cm), Aly et al. [40] (64,000 μS/cm), Achak et al. [32] (60,850 μS/cm), and Al Bsoul et al. [28] (25,100 μS/cm). Lower EC values were noted by Ait-Hmane et al. [39] (2,825 μS/cm), Dermeche et al. [47] (5,500–10,000 μS/cm), Víctor Ortega [37] (7,900 μS/cm), and Mandi et al. [48] (6,850 μS/cm). Variations in EC are likely influenced by factors such as irrigation water quality, soil type, and the salting of olives before pressing [13].

After initial treatment with sand and iron slag, the EC of OMW2 decreased to 12,590 μS/cm, a 33.1% reduction. A subsequent activated carbon treatment further reduced the EC to 9,314 μS/cm, representing an additional 26% reduction and an overall decrease of 50.5% (Fig. 5). The treatment process effectively reduces electrical conductivity (EC) by removing dissolved ionic species. After the first column is due to sand and iron slag filtration, which removes suspended solids, and iron slag, which adsorbs and precipitates certain ions. A further 26% reduction in the column containing activated carbon is achieved through the adsorption of residual ions and organic contaminants.

These results are consistent with Aly et al. [40], who reported a 48.4% reduction in EC (from 6,400 μS/cm to 3,300 μS/cm). Other studies, such as those by Al Bsoul et al. [28] and Al-Essa [35], have documented similar reduction trends.

3.3 Analysis of total dissolved solids

Fig. 3 presents the total dissolved solids (TDS) levels for the study samples (OMW1, OMW2, OMW3), with values ranging from 7200 to 12290 mg/L and an average of 9063.3 ± 253 mg/L. When compared to previous studies, discrepancies in TDS values are observed, with some studies reporting results close to those of the current study, while others report significantly lower or higher values. For instance, Al-Essa [35] reported a TDS level of 34700 mg/L, Alba [50] recorded 5900 mg/L, Passarinho [51] documented 103200 mg/L, Frascari et al. [49] found 13000 mg/L, Al Bsoul et al. [28] reported 71400 mg/L, and Aktas et al. [38] recorded 44400 mg/L. In a study by Aladham [36], the TDS value of peat water before treatment was 15,464 mg/L [7]. Overall, the TDS results in the current study fall within the mid-range of previously reported values.

After the first stage of treatment of the OMW2 sample with sand and iron slag, the total dissolved solids (TDS) level decreased by 34.4%, reaching 8,057 mg/L. Following the second stage of treatment with activated carbon, the TDS level further decreased by 26%, to 5,960 mg/L, as shown in Fig. 6. Overall, the total reduction in TDS was 51.5%, which is consistent with findings from several previous studies [28, 35, 38].

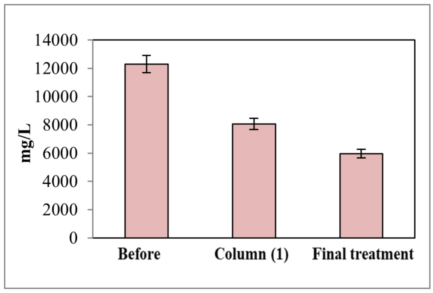

Fig. 6. Effect of different treatment steps on TDS of OMW, Data presented as mean ± SD (n = 3).

Here also, the reduction in total dissolved solids (TDS) following treatment indicates that the treatment process successfully removed a significant portion of dissolved substances, including salts and organic compounds. TDS levels are directly related to the concentration of ions and dissolved solids, which were likely removed through adsorption and ion exchange mechanisms.

3.4 Analysis of turbidity

The turbidity values of the studied OMW samples were notably high and exhibited significant variation, ranging from 2270 NTU to 11800 NTU with an average of 7663.3 NTU as depicted in Fig. 3. Sample OMW1 displayed the highest turbidity value, surpassing those reported in previous studies [52-54]. Conversely, the turbidity of sample OMW2 was more consistent with prior research findings. The elevated turbidity levels in the OMW samples can be attributed to the presence of suspended organic matter, a residual byproduct of the olive pressing process [52].

Following the initial treatment stage, the turbidity of the OMW2 sample decreased to 245 NTU, representing an 89.2%

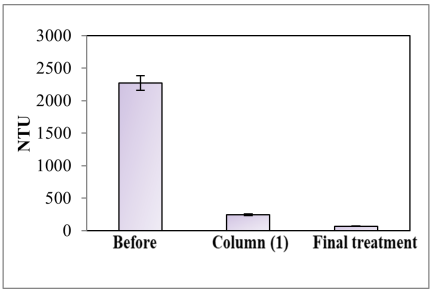

Fig. 7. Effect of different treatment steps on turbidity of OMW, Data presented as mean ± SD ($n = 3$).

reduction (shown in Fig. 7). Subsequent treatment with activated carbon further lowered turbidity to 66.1 NTU, a total reduction of 73%. In aggregate, the entire treatment process achieved a remarkable 97.1% decrease in turbidity. Turbidity is commonly attributed to suspended solids, which can be eliminated by adsorbing particulate matter onto biosorbents such as sand, iron slag, and activated carbon. The notable decrease in turbidity indicates the successful removal of suspended materials from the water sample, consistent with results reported in several earlier studies [6, 40, 52-55].

3.5 Determination of density

The density values of the studied samples ranged from 1.007 to 1.01 g/ml, as illustrated in Fig. 3. These findings are consistent with previous research by Achak et al. (1.1 g/ml) [32], Bouknana et al. (1.001 - 1.05 g/ml) [52], Al-helo et al. (1.01 - 1.02 g/ml) [56], and Eroğlu et al. (1.01 g/ml) [57].

3.6 Dissolved oxygen contents

The dissolved oxygen (DO) content of the studied samples varied, as shown in Fig. 3, ranging from 2.03 to 5.78 mg/L, with an average of 3.42 ± 0.15 mg/L. These levels are generally consistent with those reported by Achak et al. (0.48–0.69 mg/L) [32], Ben Sassi et al. (0.2–3.4 mg/L) [46], and Bouknana et al. (0.14–0.41 mg/L) [52]. However, the OMW2 sample exhibited a notably higher DO content compared to other samples. In contrast, Al-Essa [35] recorded a significantly higher DO value of 600 mg/L. The overall elevated levels of dissolved oxygen in the current study suggest a relatively low bacterial population, as microbial activity would typically deplete DO during the decomposition of biodegradable organic matter [6].

Following the initial treatment with sand and iron slag, the dissolved oxygen level in the OMW2 sample decreased to 1.5 mg/L, a 74% reduction. Subsequent treatment with activated carbon further lowered the oxygen content to 0.14 mg/L, representing a 90.7% decrease, as illustrated in Fig. 8. The efficient removal of organic contaminants and suspended particles during the treatment process is responsible for the observed decrease in DO. Sand and iron slag are used in the filtering stages to effectively remove suspended particles, which are a major cause of elevated DO levels. Furthermore, phenols, fatty acids, and other complex organic chemicals are among the organic molecules that the activated carbon used in the treatment system is essential for adsorbing. The organic load in the wastewater is significantly decreased by eliminating these contaminants, which results in a discernible drop in DO levels [28, 38, 58]. Overall, the treatment results align with findings from numerous previous studies [3-6, 32, 38].

3.7 Biochemical oxygen demand (BOD_5) contents

BOD_5 is a measure of the organic pollution in water. It indicates the amount of dissolved oxygen microorganisms need

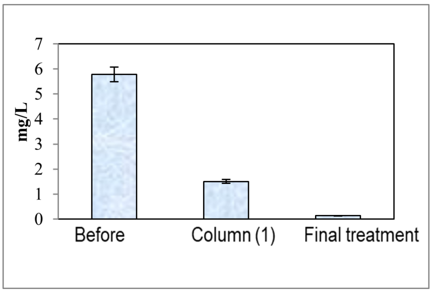

Fig. 8. Effect of different treatment steps on DO of OMW, Data presented as mean ± SD (n = 3).

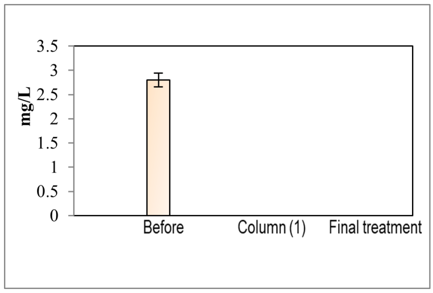

Fig. 9. Effect of different treatment steps on BOD5 of OMW, Data presented as mean ± SD (n = 3).

to decompose organic matter within five days at 20 °C. High BOD values indicate a significant amount of biodegradable organic matter in the water. This organic matter is consumed by microorganisms, which require dissolved oxygen for their metabolic processes. As microorganisms decompose the organic matter, they deplete the available oxygen in the water body. This can have detrimental effects on aquatic life, as oxygen is essential for their survival [59].

The BOD_5 values of the studied samples exhibited significant variation, with sample OMW3 displaying relatively high levels and sample OMW2 exhibiting relatively low levels. Prior to treatment, values generally ranged from 2.8 to 327 mg/L, with an average of 126.6 ± 9.5 mg/L, as depicted in Fig. 3. When compared to previous studies, a considerable discrepancy is evident. While some studies align with the results of sample OMW3, such as those by Achak et al. [32], Al-Essa [35], and Dermeche et al. [47], others, including the studies of Aladham [36], Víctor Ortega [37], and Aktas et al. [38], report higher values. Conversely, the results of sample OMW2 were more consistent with studies by Vaz et al. [6] and Ait-Hmane et al. [39]. Notably, the BOD_5 values in the present study are generally lower than those reported in previous research, indicating a reduced consumption of dissolved oxygen by microorganisms and consequently a lower level of water pollution [52].

The treatment of the OMW2 sample resulted in a significant reduction in BOD_5 levels, to the point of non-detection using the analytical method employed; as displayed in Fig. 9. This decrease was observed following the initial treatment stage. This finding aligns with the results reported by Víctor Ortega [37].

3.8 Acidity levels

The acidity levels of the OMW samples analyzed in this study varied, with values ranging from 0.97% to 2.21%, as shown in Fig. 3. The average acidity across all samples was 1.39%, which is consistent with the range of values reported in the literature. Elkacmi et al. [60] observed a similar acidity value of 1.3%, aligning closely with both the overall average and individual values for OMW1 and OMW2 in our study. Bouknana et al. [52] reported a slightly wider range of acidity (1.19–1.56%), which also falls within the range of our samples, particularly the lower acidity values of OMW1 and OMW2. However, the acidity of OMW3 was notably higher than both the average of our study and the typical range reported by Bouknana et al. The findings from Esmail et al. [61] further support the consistency of our data with prior studies on OMW acidity. The variations in acidity observed across different studies and our samples likely reflect differences in the olive oil extraction process, olive variety, and storage conditions, all of which influence the composition of phenolic compounds and organic acids responsible for the acidity in OMW [6].

3.9 Total Phenols (TP) contents

The total phenol content in the OMW samples analyzed in this study showed a clear variation, with values of 214.4 mg/L for OMW1, 173.3 mg/L for OMW2, and 126.1 mg/L for OMW3, as illustrated in Fig. 3. The average total phenol content across the three samples was 171.3 mg/L. When compared with literature data, our results fall on the lower end of the reported phenol concentrations, reflecting significant variability in the phenolic composition of OMW across different studies and geographic regions.

For instance, the total phenol content reported by Elkacmi et al. [60], while expressed as a percentage, can be assumed to be notably higher than our results. Similarly, Al-Essa [35] recorded a much higher concentration of 1340 mg/L, while Achak et al. [32] and Aly et al. [40] reported even higher values of 4500 mg/L and 13450 mg/L, respectively. These significantly higher phenol concentrations likely reflect differences in olive oil extraction processes, the maturity of the olives, and regional agricultural practices. On the other hand, some studies reported values that are closer to or even lower than our findings. For example, Frascari et al. [49] observed a range of 510–1100 mg/L, Al Bsoul et al. [28] measured 3100 mg/L, and Aktas et al. [38] recorded phenol levels between 2500 and 3800 mg/L. While these values are higher than our study’s results, they are not as extreme as those in some of the other studies mentioned. Interestingly, Ait-Hmane et al. [39] and Abu-Dalo et al. [62] found much lower phenol concentrations, with values of 55 mg/L and 51–350 mg/L, respectively. These results are more comparable to our lower range, particularly for OMW3 (126.1 mg/L), suggesting that phenol content in OMW can vary significantly depending on numerous factors such as olive variety, extraction method, and climate conditions.

The relatively lower phenol content observed in our samples may be attributed to differences in the maturity of the olives used, the extraction process employed, or perhaps even the handling and storage conditions of the OMW. Additionally, the type and intensity of treatments applied to the wastewater prior to sampling could have influenced the concentration of phenolic compounds, which are known to be degraded under certain conditions [6].

The OMW2 sample was treated using multi-step approach using columns containing different adsorption materials. Initially, OMW2 exhibited a TP concentration of 173.3 mg/L. After the first stage of treatment (with sand and iron slag), the TP concentration decreased to 168.1 mg/L, representing a modest reduction of 2.99% (shown in Fig. 10). A more substantial decrease in TP level was observed after the final treatment stage (using activated carbon), with the concentration dropping to 33.07 mg/L, corresponding to a significant reduction of 80.91% from the original value. This dramatic decrease demonstrates the high efficiency of activated carbon in adsorbing phenolic compounds from olive mill wastewater (OMW). Activated carbon effectively binds to phenolic compounds

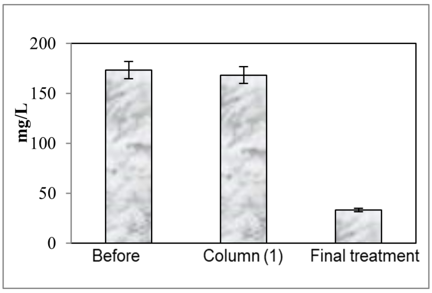

Fig. 10. Effect of different treatment steps on TP of OMW, Data presented as mean ± SD (n = 3).

through its large surface area, porous structure, and affinity for aromatic molecules, resulting in a significant decrease in TP levels. The total phenol reduction of 80.91% in OMW2 aligns with findings from other studies highlighting the effectiveness of other adsorbing materials in removing phenolic pollutants [23,25,30,32,39,49,62,63]. Factors such as pH may have also contributed to the adsorption efficiency, as pH can influence the ionization state of phenolic compounds, thus impacting their interaction with the adsorbent [63].

The obtained results from the treated wastewater were compared with the WHO drinking water quality standards [64]. The pH value of the treated water (7.02) falls within the WHO recommended range of 6.5–8.5, making it suitable in terms of acidity and alkalinity. However, the dissolved oxygen (DO) level of 0.14 mg/L is significantly lower than the WHO minimum requirement of 5 mg/L, which is critical for sustaining aquatic life and ensuring water quality. The total dissolved solids (TDS) concentration of 5960 mg/L exceeds the WHO permissible limit of 1000 mg/L, indicating a high level of dissolved substances that could affect water taste and quality. Additionally, the turbidity of 66.1 NTU is far above the WHO recommended maximum of 1 NTU for drinking water, signifying poor clarity. The biological oxygen demand (BOD_5) value of 0.00 mg/L indicates no immediate biodegradable organic pollutants, which aligns with drinking water standards. However, the total phenols (TP) concentration of 33.07 mg/L is significantly higher than the WHO limit of 0.001 mg/L, indicating the presence of phenolic compounds that could pose risks to health and the environment.

3.10 Effect of pH on treatment process

The effect of pH on the treatment process of OMW samples was investigated by modifying the medium with 1M HCl and 1M NaOH. This study examined how the pH of the medium influenced various physical and chemical properties of the OMW samples, including electrical conductivity (EC), total dissolved solids (TDS), biological oxygen demand (BOD_5), and total phenolic content (TP).

3.10.1 In acidic pH

As shown in Table 3, the EC of the OMW2 sample before treatment was 18810 μS/cm. After the first stage of treatment with sand and iron slag, the EC dropped to 14900 μS/cm, representing a reduction of 20.8%. Following the final stage of treatment using activated carbon, the EC further decreased to 13597 μS/cm, resulting in a total reduction of 27.7%. When comparing these findings to those without pH control, where the final EC was 9314 μS/cm (a 50.5% reduction), it is evident that treatment in an acidic medium resulted in a lower overall reduction in EC. This can be attributed to the impact of acidic conditions on ion dissociation and the surface chemistry of the adsorbent materials. Under acidic pH, the protonation of functional groups on the adsorbent surface may reduce

Table 3. Impact of Acidic pH on Treatment Efficiency (mean ± standard deviation, $n = 3$).

Parameter	Unit	Before treatment	After column 1	End treatment
EC	μS/cm	18810±910	14900±640	13597±379
TDS	mg/L	12290±314	9500±275	8668±233
Turbidity	NTU	2270±112	14.8±0.65	8.40±0.32
BOD5	mg/L	2.80±0.12	1.50±0.06	0.57±0.02
TP	mg/L	173.3±7.6	-	36.40±1.42

the availability of active sites for ion adsorption, thereby limiting the efficiency of the adsorption process and leading to less effective ion removal.

The initial TDS level in the OMW2 sample was 12290 mg/L (displayed in Table 3). After passing through the first treatment stage, the TDS dropped to 9950 mg/L, reflecting a decrease of 19.1%. The final treatment brought the TDS down further to 8668 mg/L, achieving a total reduction of 29.5%. In contrast, without pH control, the TDS decreased to 5960 mg/L (a 51.5% reduction). The greater reduction in TDS without pH control suggests that acidic pH medium may have influenced the solubility of salts and other dissolved solids, limiting the treatment's overall efficiency in reducing TDS.

Before treatment, the turbidity of the OMW2 sample was 2,270 NTU. After the first stage of treatment, the turbidity decreased dramatically to 14.8 NTU, marking a 99.35% reduction. After the final treatment stage, the turbidity further reduced to 8.4 NTU, resulting in an overall reduction of 99.63%. When comparing this to the treatment without pH control, where the final turbidity was 66.1 NTU (a 98.5% reduction), pH control slightly improved turbidity removal. The near-complete reduction in turbidity in both cases highlights the effectiveness of the column treatment process, particularly in removing suspended particles. The initial BOD_5 level of the untreated OMW2 sample was 2.8 mg/L. After the first stage of treatment, the BOD_5 was reduced to 1.5 mg/L, achieving a 46.4% reduction. The final treatment stage further decreased the BOD_5 to 0.57 mg/L, yielding a total reduction of 79.6%. Without pH control, the BOD_5 was reduced to 0.00 mg/L (a 100% reduction), indicating that the absence of pH control significantly enhanced the biodegradation of organic matter. This suggests also that acidic pH may inhibit the microbial activity responsible for breaking down biodegradable organic compounds, leading to a less effective reduction in BOD_5.

The initial total phenol concentration in the OMW2 sample was 173.3 mg/L. After the final treatment stage, TP was reduced to 36.4 mg/L, corresponding to a 79.0% reduction. Without pH control, the final TP concentration was 33.07 mg/L, resulting in a slightly higher reduction of 80.9%. The minimal difference between these two results suggests that pH adjustment had a marginal effect on phenol removal, with both conditions showing effective adsorption of phenolic compounds onto the activated carbon.

3.10.2 In basic pH

In the basic pH medium; as depicted in Table 4, the EC of the OMW2 sample was reduced from 18810 μS/cm to 14248 μS/cm after the first stage, a 24.2% reduction. The final treatment further decreased the EC to 7155 μS/cm, achieving an overall reduction

Table 4. Impact of basic pH on Treatment Efficiency (mean ± standard deviation, n = 3).

Parameter	Unit	Before treatment	First Treatment	Final Treatment
EC	μS/cm	18810±910	14248±512	7155±157
TDS	mg/L	12290±314	9081±254	4561±225
Turbidity	NTU	2270±112	8.40±0.32	5.65±0.27
BOD5	mg/L	2.8±0.12	0.07±0.01	0.00
TP	mg/L	173.3±7.6	-	98.54±2.9

of 61.9%. Comparatively, in the treatment without pH control, the final EC was 9314 μS/cm, with a lower overall reduction of 50.5%. The enhanced reduction in EC under basic pH conditions can be attributed to the increased dissociation of ions, which may enhance their interaction with the adsorbent materials. Additionally, the higher pH likely facilitated the adsorption of more ions onto the activated carbon surface, improving the ion removal efficiency. This suggests that the basic pH medium played a crucial role in enhancing the treatment's effectiveness in reducing EC.

From Table 4, the initial TDS of the untreated sample was 12290 mg/L. In the basic pH medium, TDS was reduced to 9081 mg/L after the first stage, a 26.1% reduction, and further decreased to 4561 mg/L after the final treatment, with a total reduction of 62.9%. In contrast, the final TDS without pH control was 5960 mg/L, corresponding to a 51.5% reduction. These results indicate that the basic pH improved TDS removal efficiency, likely because higher pH conditions facilitated the precipitation of dissolved ions, allowing for better adsorption by the activated carbon.

The turbidity of the untreated OMW2 sample was 2270 NTU. After the first stage of treatment in a basic medium, turbidity was reduced to 8.4 NTU, a 99.6% reduction. The final stage brought it down further to 5.65 NTU, corresponding to a total reduction of 99.8%. Without pH control, the final turbidity was 66.1 NTU, reflecting a reduction of 97.1%. This significant improvement in turbidity removal in the basic medium suggests that high pH conditions may have enhanced particle coagulation, allowing more efficient filtration and adsorption The results are shown in Table 4.

The BOD_5 of the untreated OMW2 sample was 2.8 mg/L. In the basic medium, it was drastically reduced to 0.07 mg/L after the first stage, a reduction of 97.5%, and to 0.00 mg/L after the final treatment, achieving 100% removal. Without pH control, BOD_5 was similarly reduced to 0.00 mg/L. While both processes were effective in completely eliminating BOD_5, the basic medium achieved near-complete removal after the first stage, indicating that the higher pH might have enhanced the degradation of organic matter earlier in the process.

The initial total phenol concentration in the OMW2 sample was 173.3 mg/L. In the basic medium, after the final stage, TP was reduced to 98.54 mg/L, corresponding to a 43.2% reduction. Without pH control, TP was reduced to 33.07 mg/L, achieving a much higher reduction of 80.9%. These results suggest that the basic pH medium was less effective for phenol removal, likely because phenolic compounds are more difficult to adsorb or degrade in basic conditions, and acidic conditions are typically more favorable for phenol adsorption onto activated carbon.

3.11 Sustainable management and valorization of spent biosorbents

To maintain environmental sustainability and maximise utilisation of resources, wasted biosorbents from the treatment of olive mill wastewater (OMW) need to be managed [65]. The environmental impact of the treatment process may be reduced while achieving further benefits through regeneration, recycling, and valorisation. After having been utilised in the treatment process, activated carbon made from olive leaves can be renewed to restore its adsorption ability. By efficiently desorbing the residual contaminants by heating at high temperatures, thermal regeneration makes it possible to reuse the activated carbon in later treatment cycles. As an alternative, weak acids or bases can be used to accomplish chemical regeneration [66]. Adsorbed pollutants can be removed from iron slag and sand, which are essential parts of the treatment system, by washing them with water or diluted acid. This allows them to be used again in subsequent treatment procedures [65, 66]. In addition to their use in water treatment, these materials have a large potential for recycling in the building industry. Recovering significant bioactive compounds that were retained throughout the treatment procedure is another opportunity presented by spent biosorbents. For example, it is possible to extract and use oligosaccharides with strong antibacterial and antioxidant activities in agriculture. Such compounds are useful for improving plant health, preventing pest damage to crops, and integrated pest control. Likewise, it is possible to extract and use the phenolic chemicals that are retained on the biosorbents as biostimulants [65, 67].

4. Conclusion

This study highlights the effectiveness of a multi-step column treatment process for improving the quality of olive mill wastewater (OMW), with a particular focus on the influence of pH conditions. The combination of sand, iron slag, and activated carbon demonstrated significant reductions in electrical conductivity (EC), total dissolved solids (TDS), turbidity, biochemical oxygen demand (BOD□), and total phenols (TP). The impact of both acidic and basic pH conditions was evident in the results.

In an acidic medium, the treatment process achieved enhanced turbidity removal, indicating that low pH improved the agglomeration of suspended particles and their adsorption onto activated carbon. However, the acidic conditions limited the reductions in EC, TDS, and BOD□ compared to treatment without pH control, likely due to inhibited ion exchange and microbial activity. Total phenol removal was marginally affected by pH adjustment, with effective adsorption observed in both acidic and neutral conditions.

In a basic pH environment, the treatment process significantly enhanced the removal efficiency for most parameters. Electrical conductivity (EC) decreased from 18,810 μS/cm to 7,155 μS/cm, resulting in a 61.9% reduction. TDS was reduced by 62.9%, and BOD□ was completely eliminated, showing a 100% decrease. Turbidity was reduced by an impressive 99.8%. However, the reduction in total phenols was lower, at 43.2%, compared to the 80.9% reduction achieved without pH control. The basic pH medium facilitated ion exchange and improved interactions between dissolved ions and the adsorbent materials, yielding better outcomes than the acidic medium.

Overall, this study demonstrates that pH plays a critical role in optimizing OMW treatment processes. While acidic conditions are beneficial for suspended particle removal, a basic pH environment significantly enhances the overall reduction of ionic content and biodegradable organic matter. These insights are valuable for tailoring wastewater treatment strategies depending on the specific pollutants targeted in OMW.

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