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Scientific paper

Efficient Treatment of Olive Mill Wastewater using a Three-Column Continuous System Packed with Different Adsorbents

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Abstract

This study aimed to investigate the feasibility and effectiveness of using a three-column continuous system for treating olive mill wastewater (OMW). The methodology involved passing the wastewater through three successive columns packed with various adsorbent materials. The efficiency was evaluated by measuring several chemical and physical properties before and after treatment, including pH, electrical conductivity (EC), total dissolved solids (TDS), acidity, turbidity, chloride, nitrate, nitrite, sulphate, phosphate, Na, K, Ca, Fe, Cu, total phenols, and total flavonoids. The initial values of these properties varied among the samples. After treatment, the measured properties were significantly changed, including an increase in pH from 4.8 to 6.9, EC from 12990 µS/cm to 1407 µS/cm, turbidity from 7328 NTU to 2791 NTU, chloride from 3400 ppm to 422 ppm, nitrate from 307 ppm to 4.34 ppm, Na from 293 ppm to 178 ppm, K from 2243 ppm to 86 ppm, Ca from 91 ppm to 60 ppm, and a significant reduction in total phenols from 9821 mg/g to 35 mg/g. The results suggest that the proposed treatment is effective for purifying OMW and offers insights for developing sustainable and eco-friendly wastewater treatment methods.

Keywords: Olive mill wastewater, Sustainable wastewater treatment, Adsorption methods, Activated carbon.

1. Introduction

Environmental contamination resulting from toxins has significantly increased in recent years, primarily due to rapid industrialization.[1] The industrial sector's growing demand for resources and raw materials has intensified ecological and economic imbalances.[2] Urban industrialization has reduced land areas for waste disposal, leading to the unchecked discharge of untreated industrial and domestic wastes into the environment. This practice negatively affects soil and groundwater quality, rendering them unsuitable for use.[3] Industrial wastewater typically contains hazardous substances such as heavy metals and toxic organic compounds, which are carried into the environment.[4] The olive oil industry is a significant contributor to environmental contamination due to the presence of hazardous materials in the wastewater it produces.[5]

The olive oil industry holds significant economic and social importance for numerous Mediterranean countries, including Spain, Italy, Greece, Turkey, Tunisia, and Morocco.[6] These countries continue to dominate olive oil production, accounting for approximately 94% of the world's total output. Updated statistics indicate that global production reached 3.2 million tons in 2020. According to the Food and Agriculture Organization of the United Nations (FAO), Libya produced approximately 8,000 tons of olive oil in 2019.[7,8] This represents a significant decline from previous years, as production was estimated at around 100,000 tons in the 1970s.[9] The decline in production is mainly due to the disruption of agricultural activities caused by the ongoing conflict in the country. In the Mediterranean region, olive oil production generates approximately 30 million tons of olive oil mill wastewaters annually (OMW).[10]

The process of extracting olive oil yields three distinct components: olive oil, solid residue, and aqueous liquor, which can represent between 20-50% of the total weight of the processed olives.[11] Regardless of the extraction method used, whether discontinuous or centrifugation system, the olive oil industry generates two by-products in addition to its primary product, namely extra virgin oil and pomace oil. The first by-product is solid waste resulting from the squeezed residue, which consists of pulp and olive stones, known as "pomace". This waste can be transformed into animal feed or further processed into "olive pomace oil" through chemical extraction. The second by-product is a dark red to black liquid effluent known as "Olive Mill Wastewater" (OMW).[12] The production of olive oil generates significant quantities of liquid and solid waste during a relatively short period of time, typically from November to March. On average, pressing one ton of olives produces 1.5 tons of OMW with modern production methods.[13,14] The main challenge confronting the olive oil industry is the disposal and treatment of the liquid waste produced during olive oil production. Olive mill wastewater (OMW) is a highly polluting effluent, marked by its acidic nature, high salinity, and elevated levels of organic matter, phenolic compounds, and other toxic substances.[10] These characteristics render OMW resistant to natural degradation and pose significant risks to the environment. When untreated olive mill wastewater (OMW) is released into soil or water bodies, it results in significant ecological consequences.[15] The high levels of chemical oxygen demand (COD) and biological oxygen demand (BOD) deplete oxygen in aquatic systems, leading to eutrophication and endangering aquatic life. Additionally, the phenolic compounds present in OMW are both phytotoxic and antimicrobial, disrupting soil microbial communities and inhibiting plant growth, which ultimately reduces soil fertility. OMW also contributes to groundwater contamination, soil salinisation, and the deterioration of natural ecosystems. Therefore, addressing the environmental impact of OMW is a pressing concern for both the olive oil industry and regulatory bodies.[13,14]

Various methods have been explored for the removal of pollutants from Olive Mill Wastewater (OMW), which can be broadly categorized into biological, chemical, and physical methods.[16] Each method has its own advantages and drawbacks, and due to the high cost and disposal concerns, many conventional methods have not been widely adopted at large scales in olive oil mills.[17] Various methods have been explored for treating Olive Mill Wastewater (OMW), which can be broadly categorised into biological, chemical, and physical approaches.[12] Biological methods, such as aerobic and anaerobic digestion, rely on microbial processes to degrade organic pollutants. However, these methods are often impeded by the antimicrobial nature of phenolic compounds in OMW, which suppress microbial activity.[16] Chemical treatments, including advanced oxidation processes and chemical precipitation, can effectively remove specific pollutants but are associated with high operational costs and may generate harmful secondary pollutants.[17] Physical methods, such as filtration, sedimentation, and adsorption using conventional materials like activated carbon, can also reduce pollutants; however, they are limited by high costs and the need for frequent regeneration.[11] To overcome these challenges, researchers have investigated the use of cheaper and more effective adsorbents, including natural materials, biosorbents, and waste materials from industries and agriculture.[18] A number of low-cost adsorbents have been proposed for the removal of phenolic compounds from solutions, such as activated charcoal, coal, dried activated sludge and fly ash, palm pith carbon, and beet pulp.[19]

Biosorbent materials have emerged as an eco-friendly and cost-effective alternative for treating Olive Mill Wastewater (OMW). Derived from natural or waste biomass, these materials have demonstrated significant potential in adsorbing a wide range of pollutants, including phenolic compounds, heavy metals, and organic contaminants. Their effectiveness is attributed to their high surface area, porous structure, and the presence of functional groups that enhance their adsorption capabilities.[11] Examples of biosorbents include agricultural residues such as olive leaves, palm pith carbon, and beet pulp, as well as industrial by-products like dried activated sludge and fly ash.[5,10,16] Olive leaves are particularly noteworthy; they are rich in cellulose, hemicellulose, and lignin, which provide active binding sites for pollutant adsorption. Research has shown that olive leaf biosorbents can effectively remove over 90% of phenolic compounds and reduce turbidity by more than 60% under optimal conditions. Similarly, beet pulp and palm pith carbon have been found to significantly reduce total dissolved solids and colour intensity in wastewater.[9] In addition to their efficiency, biosorbents provide several advantages, such as low cost, availability, and minimal environmental impact. Many biosorbents can be regenerated and reused, which further enhances their sustainability. By using agricultural and industrial residues as biosorbents, the olive oil industry not only tackles wastewater treatment challenges but also encourages the valorisation of waste materials, contributing to a circular economy.[13]

Our objective is to assess the feasibility and effectiveness of using a three-column continues system for treating olive mill wastewater through infiltration percolation. The system involves passing the wastewater through three successive columns that are packed with various adsorbent materials, including fine sand, coarse sand, limestone, and two types of biosorbents made from olive leaves (dry powdered material and activated carbon material). The treatment method's efficiency was evaluated by measuring several chemical properties before and after the treatment, such as density, turbidity, acidity, pH, electrical conductivity (EC), total dissolved solids (TDS), ash, total phenols, total flavonoids, nitrate, sulphate, phosphate, chloride, nitrite, K, Na, Ca, Fe, and Cu.

2. Experimental

2. 1. Collection and Preservation of Olive Mill Wastewater

From December 2022 to March 2023, olive mill wastewater (OMW) samples were collected for this study from five olive oil extraction processes located in the Msallata area of Libya. These processes used both traditional and continuous methods, as indicated in Table 1, and no chemical additives were used during olive oil production. The OMW was collected using aseptic techniques in a closed plastic container to prevent contamination. The samples were immediately analysed for physicochemical parameters, including pH, acidity, electrical conductivity, TDS, density, turbidity, and ash content. To minimize the risk of biodegradation, the samples were stored at 4°C until further analysis.

Table 1. OMW samples

No.	Type of olive mill	Area	Symbol
1	Continuous	Sam Aldays (1)	S1
2	Traditional	Aleamud (1)	A1
3	Traditional	Sam Aldays (2)	S2
4	Continuous	Aleamud (2)	A2
5	Continuous	Qasr Aljadid	Q

2. 2. Analytical Methods

The chemical and physical properties of the five samples of OMW were assessed before the treatment process. Subsequently, one sample was subjected to the treatment process, and the efficiency of the separation process was evaluated by analyzing its physical and chemical properties.

2. 2. 1. Determination of pH, Electrical Conductivity, Total Dissolved Solids (TDS), and Turbidity

The pH measurement was performed using a Jenway pH-meter (model 3505). Prior to measuring the samples, the device was calibrated at room temperature using buffer solutions with pH values of 4, 7, and 9. The electrical conductivity and total dissolved solids were measured using a Jenway 4510 conductivity meter. The device was calibrated using a 0.1 M potassium chloride solution. The turbidity of the samples under study was measured using a Hach 2100N turbidity meter from Hach. Turbidity is expressed in NTU (Nephelometric Turbidity Units).

2. 2. 2. Determination of Ash

The ash content was estimated using the techniques outlined in earlier research.[20] A sterile and dehydrated crucible was employed to measure 10 g of the OMW sample. The sample was then subjected to drying in an oven for 3 h at 105 °C and subsequently transferred to a muffle furnace for 3 h at 550 °C. Following this, the crucible was allowed to cool in a desiccator for 15 min before being weighed. The percentage of ash content was determined using the formula below:

$$\% \text{ ash} = \frac{\text{wt of ash}}{\text{wt of sample}} \text{ x } 100 \quad (1)$$

2. 2. 3. Determination of Acidity

The acidity percentage was determined using volumetric techniques by conducting a titration against a standardized solution of potassium hydroxide (0.1 M) with phenolphthalein serving as the indicator.[21]

2. 2. 4. Determination of chloride

The chloride was estimated by volumetric titration[22] against a standardized solution of silver nitrate (0.1 M) in the presence of potassium chromate as an indicator (Mohr's method).

2. 2. 5. Determination of sulphate

The sulfate was estimated using spectroscopic methods (turbidity measurement).[22] The method relies on the reaction between sulfate anions and barium cations, which results in the formation of a precipitate (barium sulfate), and the turbidity is monitored at a wavelength of 420 nm using an LED-based photometer of the type 2100Q Turbidimeter from Hach.

2. 2. 6. Determination of Nitrate and Nitrite

Estimation of nitrates and nitrites in the samples under study was performed using the published spectroscopic methods proposed.[22] These methods rely on the formation of a colored complex between nitrate or nitrite and certain reagents, such as sulfanilamide and N-(1-naphthyl) ethylenediamine dihydrochloride, which can be measured at specific wavelengths using a spectrophotometer.

2. 2. 7. Determination of Total Phenols

The total phenolic content was determined using a modified Folin-Ciocalteu method[23] in the current study on OMW samples. A 0.2 mL aliquot of the diluted OMW water sample was mixed with 1 mL of 10% diluted Folin-Ciocalteu reagent. The mixture was kept in the dark for 4 min and then 0.8 mL of 7.5% sodium carbonate solution was added. The volume was completed to 10 mL with solvent. The absorbance of the solution was measured at 765 nm after 30 min, and several dilutions of each OMW sample were used to calculate the average total phenolic concentrations. Gallic acid was used as a reference standard in this method, and the total phenolic equivalent to gallic acid was calculated. A standard calibration curve was prepared using gallic acid concentrations of 10, 20, 30, 40, 50, and 60 mg/L.

2. 2. 8. Determination of Total Flavonoids

The total flavonoids were estimated using the modified aluminum chloride method,[24] with rutin as a reference standard, and the total flavonoid equivalent to rutin was calculated. A standard calibration curve was prepared using the following concentrations of rutin: 1-5-10-20-40-60 mg/L. The total flavonoids were also estimated in all studied OMW samples. To estimate the total flavonoids, 1 mL of diluted OMW or rutin solution was mixed with 0.3 mL of sodium nitrite solution ($NaNO_2$) and 4 mL of distilled water. After 5 min, 0.3 mL of aluminum chloride solution was added. The mixture was left for 6 min, and then 2 mL of 1 M sodium hydroxide solution was added. The volume was completed to 10 mL with distilled water after 10 min, and the absorbance of the solution was measured at 510 nm. Different dilutions of each sample were prepared, and the average concentrations were calculated.

2. 2. 9. Determination of Minerals

The concentrations of some major elements (sodium, potassium, and calcium) were estimated using a Flame Photometer, specifically the Jenway™ PFP7 Industrial Flame Photometer. The concentrations of some minor elements (iron, copper, zinc, nickel, and cadmium) were measured using a Flame Atomic Absorption Spectrometer (VARIAN AA240FS) to determine the atomic absorption in the flame. Prior to analysis, samples were digested using the wet digestion method available in literature.[25–27]

2. 3. Treatments of Olive Oil Mill Wastewater

The OMW sample was purified using a three-column successive system, each containing different natural materials. The columns were filled successively with fine and coarse sand (first column), limestone (second column), olive leaf powder and activated carbon from olive leaves (third column). All materials were washed several times with distilled water (Until the effluent from the materials was devoid of impurities) and dried before use. The olive leaves were dried and ground, while the activated carbon was burned in an air-isolated combustion furnace at 600 °C.[28–30] Figure 1 illustrates a schematic diagram of the columns used in the treatment process. The columns used in this experiment were made of polyvinyl chloride with an inner diameter of 2.5 cm and a length of 50 cm. The columns were sealed from the bottom using tightly secured cotton. The materials were packed in the columns to a height of 15 cm. The columns were vertically fixed on a holder, and each treatment system was repeated three times. The cumulative purified water was collected over time for analysis. Physical and chemical properties were measured (for one sample) to evaluate the efficiency of the treatment.

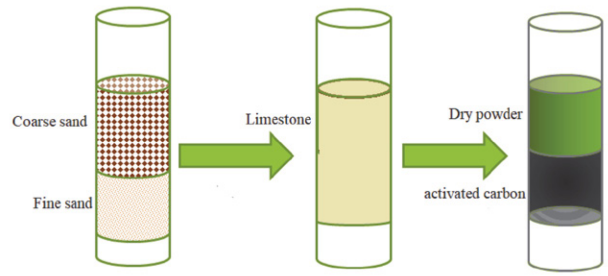

Figure 1. System used for OMW treatment

2. 4. Statistical Analysis

The current study utilized Microsoft Office Excel 2016 for statistical analysis of the data. The reported results were obtained as the mean value of a minimum of five independent replicates, with the corresponding standard deviation (±) also being calculated.

A t-test (paired two sample for means was) employed to assess the significance of differences in measured parameters before and after treatment. This paired t-test method takes into account the dependence between paired observations, ensuring a precise evaluation of treatment effectiveness. Statistical significance was established using a threshold p-value of 0.05, with p-values below this threshold indicating significant differences between the compared means.

3. Results and Discussion

Table 2 presents the chemical property measurements obtained from five samples of olive oil mill wastewater (OMW) collected from five distinct olive oil extraction processes. The first sample (S1 Sample) underwent a proposed treatment method, after which the same chemical properties were measured to evaluate the effectiveness of the treatment.

The physicochemical properties of olive oil mill wastewater (OMW) are important indicators of its quality and environmental impact. The pH values of the five OMW samples ranged from 4.0 to 5.04, indicating that they are acidic. This is consistent with previous studies that have reported pH values ranging from 3.8 to 5.2 for OMW.[31–33] Electrical conductivity (EC) values ranged from 4475 to 12990 μS/cm, which is also in agreement with previous studies that reported EC values ranging from 6400 to 18000 μS/cm for OMW.[32–34] Total dissolved solids (TDS) values ranged from 3980 to 11980 ppm, which is within the range reported in other studies.[32–34] Turbidity values ranged from 3170 to 12216 NTU, which is consistent with previous studies that reported turbidity values ranging from 2000 to 16800 NTU for OMW.[32–35] The density values ranged from 0.931 to 1.730 g/mL, which is also consistent with previous studies that reported density values ranging from 0.9440 to 1.8100 g/mL for OMW.[33,34] Finally, the % acidity values ranged from 0.91 to 2.12%, which is within the range reported in other studies,[32,34,36] and the % ash values ranged from 0.33 to 1.64%, which is also consistent with previous studies that reported ash values ranging from 0.22 to 3.1% for OMW.[33,34] Azzam et al.[37] and Al Bsoul et al.[38] studied the physicochemical properties of OMW from different regions in Jordan and reported pH values ranging from 4.6 to 5.9, EC values ranging from 2500 to 2700 μS/cm, and TDS values ranging from 2000 to 3500 ppm. Similarly, Azzam et al.[33] investigated the physicochemical properties of OMW from different regions in Jordan (2022) and reported a mean pH value of 5.03, EC mean value of 9520 μS/cm, and TDS mean value of 8760 ppm. Ramires et al.[39] investigated the physicochemical properties of OMW from different regions in Italy and reported pH value of 5.1, EC value of 7600 μS/cm, and TDS values ranging from 3210 to 14160 ppm. They also reported a chemical oxygen demand value of 286.33 g/L and Trolox equivalent antioxidant capacity of 1008 μM TE/mL.

Likewise, the levels of some ions were measured, namely Cl^-, NO_3^-, SO_4^{2-}, and PO_4^{3-}, in the OMW samples. The Cl^- levels ranged from 1150 to 6100 ppm, with an average of 4130 ppm. These levels are consistent with those reported in other studies, which have reported Cl^- levels ranging from 1000 to 8000 ppm in OMW.[31,32,40] The NO_3^- levels ranged from 116 to 2010 ppm, with an average of 737 ppm. These levels are also consistent with those reported in other studies, which have reported NO_3^- levels ranging from 200 to 2000 ppm in OMW.[32,37] The SO_4^{2-} levels ranged from 30 to 750 ppm, with an average of 185 ppm. These levels are within the range reported in other studies, which have reported SO_4^{2-} levels ranging from 10 to 1500 ppm in OMW.[32,36] Finally, the PO_4^{3-} levels ranged from 35 to 190 ppm, with an average of 87 ppm. These levels are also within the range reported in other studies, which have reported PO_4^{3-} levels ranging from 20 to 200 ppm in OMW.[32,37] The levels of these ions in OMW can vary depending on several factors, such as the type of olive oil production process, the location of the olive groves, and the weather conditions during the olive harvesting season. Hodaifa et al.[40] reported higher Cl^- and SO_4^{2-} levels in OMW samples collected from regions with high agricultural activity, while Flores et al.[41] reported higher Cl^-, NO_3^-, and SO_4^{2-} levels in OMW samples collected from regions with high industrial activity.

Additionally, the levels of five minerals, namely Na, K, Ca, Fe, and Cu, in the OMW samples have been determined. The Na levels ranged from 227 to 381 ppm, with an average of 301 ppm. The K levels ranged from 771 to 3111 ppm, with an average of 2215 ppm. The Ca levels ranged from 91 to 169 ppm, with an average of 120 ppm. Fe was detected only in some samples at very low levels, while Cu levels ranged from 0.025 to 0.056 ppm, with an average of 0.038 ppm. The levels of Na and K in OMW are of particular interest, as they can have implications for the potential use of OMW as a source of nutrients for plants or as a feedstock for biogas production.[10] The K levels reported in this study are generally consistent with those reported in other studies, which have reported K levels ranging from 1600 to 4300 ppm in OMW.[42,43] However, the Na levels reported in this study are lower than those reported in some other studies, which have reported Na levels ranging from 1000 to 24000 ppm in OMW.[42,43] This may be due to differences in the olive oil production process or the location of the olive groves. The Ca levels reported in this study are also consistent with those reported in other studies, which have reported Ca levels ranging from 20 to 300 ppm in OMW.[32,34] Iron (Fe) is typically present in low levels in OMW, and its presence can be influenced by factors such as the pH and the oxidation-reduction potential of the wastewater.[42] The Cu levels reported in this study are generally consistent with those reported in other studies, which have reported Cu levels ranging from 0.01 to 0.1 ppm in OMW.[34,43]

Finally, the total phenols levels ranged from 1250 to 12474 mg/g, with an average of 8576 mg/g. The total flavonoids levels ranged from 182 to 2560 mg/g, with an average of 1275 mg/g. The levels of total phenols and flavonoids in OMW can vary depending on several factors, such as the type of olive oil production process, the location of the olive groves, and the weather conditions during the olive harvesting season. Mekki et al.[31] reported total phenols levels ranging from 840 to 12800 mg/g in OMW samples, while Leouifoudi et al.[44] reported total flavonoids levels ranging from 0.8 to 12.9 mg/g in OMW samples collected from different regions in Morocco. Also, Dermeche et al.[42] characterized OMW samples collected from different olive mills in Algeria and reported total phenols levels ranging from 6.9 to 73.7 g/L, with an average of 34.5 g/L. They also reported total flavonoids levels ranging from 0.1 to 5.0 g/L, with an average of 1.6 g/L. Solomakou et al.[45] reviewed the literature on the phenolic composition of OMW and reported that the levels of total phenols in OMW can range from 0.5 to 14 g/L, while the levels of total flavonoids can range from 0.01 to 0.5 g/L.

3. 1. The Properties of OMW After Treatment

In the current study, the proposed treatment method (illustrated in Figure 1) was applied to a sample of olive oil mill wastewater (OMW). To evaluate the effectiveness of the treatment process, the aforementioned properties were measured to determine the extent of the method's impact

Table 2. OMW physicochemical characteristics

Parameter*	S1	A1	S2	A2	Q
pH	4.40±0.13	5.04±0.15	4.60±0.13	4.09±0.17	4.80±0.14
EC (µS/cm)	12990±390	4475±170	10690±213	9848±295	11790±236
TDS (ppm)	3980±89	7717±189	11980±399	4200±210	7600±211
Turbidity (NTU)	7328±146	9125±253	12216±366	8750±262	3170±63
Density (g/mL)	1.003±0.040	1.035±0.031	1.730±0.009	1.009±0.040	0.931±0.028
% Acidity	0.91±0.04	0.94±0.05	2.12±0.10	1.87±0.09	0.97±0.04
% Ash	1.41±0.04	0.33±0.02	1.64±0.08	1.64±0.08	0.88±0.04
Cl^- (ppm)	3400±68	3500±105	6100±183	1150±23	700±28
NO_2^- (ppm)	2.73±0.15	4.25±0.33	1.89±0.08	2.66±0.11	1.44±0.12
NO_3^- (ppm)	307±12	1000±20	2010±60	116±4	150±5
SO_4^{2-} (ppm)	60±2.5	30±1.5	53±2.7	750±22.5	30±2.0
PO_4^{3-} (ppm)	79±3.2	75±3.0	190±5.7	35±0.7	57±2.3
Na (ppm)	293±10	227±8	381±7	259±11	345±13
K (ppm)	2243±67	771±23	2340±93	2752±82	3111±95
Ca (ppm)	91±3.6	91±3.0	126±5.1	122±3.7	169±6.8
Fe (ppm)	0.33±0.01	ud	0.03±0.00	0.13±0.01	0.07±0.01
Cu (ppm)	0.025±0.001	0.039±0.002	0.056±0.002	0.033±0.001	0.034±0.001
Total phenols (mg/g)	9821±392	12150±380	8883±266	12474±375	9254±370
Total flavonoids (mg/g)	2560±51	182±7	942±28	1439±43	1452±47

* the measured value is a mean of 5 replicates, ud: under instrument detection limits

3. 1. 1. Physicochemical Properties

The treatment of OMW sample with the three-column system (shown in Figure 1) resulted in significant changes in its physicochemical properties as revealed in Figure 2. This figure compares various physicochemical parameters of olive mill wastewater (OMW) before and after treatment. The measured parameters include pH, electrical conductivity (EC), total dissolved solids (TDS), turbidity, density, acidity, and ash content. The data are presented as the mean of five independent replicates, with error bars indicating the standard deviation. After treatment, significant reductions were observed in most parameters, including pH, EC, TDS, turbidity, total phenols, and total flavonoids.

The pH of the treated sample increased from acidic to near-neutral, indicating an increase in the concentration of hydroxide ions and a decrease in the concentration of hydrogen ions. The pH increase observed in the current study is likely due to the neutralizing effect of the limestone used in the second column of the treatment system. The increase in pH; as shown in Figure 2, is consistent with the alkaline nature of limestone and its ability to neutralize acidic solutions. This is in contrast to the acidic conditions in the composting process used in Mekki et al.,[31] who reported a reduction in pH from 4.8 to 4.4 after treating OMW with composting. Additionally, the EC of the treated sample decreased from 12990 to 1407 µS/cm, indicating a reduction in the concentration of dissolved salts. This is similar to the findings of Aly et al.,[32] who reported a decrease in EC after using various treatment methods for OMW.

The turbidity of the treated sample also decreased from 7328 to 2791 NTU, indicating a reduction in the concentration of suspended solids (displayed in Figure 2). This is consistent with the findings of Aly et al.,[32] who reported a decrease in turbidity from 4000 to 2000 NTU after treating OMW with composting. Furthermore, the TDS of the treated sample decreased from 3980 to 1901 mg/L, indicating a reduction in the concentration of dissolved solids. This is consistent with the findings of Achak et al.,[34] who reported a decrease in TDS after treating OMW with a combination of electrocoagulation and electroflotation.

The ash content of the treated sample also decreased from 1.41 to 0.02%; as shown in Figure 2, indicating a reduction in the concentration of inorganic residues. This is similar to the findings of Solomakou et al.,[45] who reported a decrease in ash content after treating OMW with Lactobacillus plantarum. Additionally, the acidity of the treated sample decreased from 0.91 to 0.53 g/L, indicating a reduction in the concentration of acidic compounds. This is consistent with the findings of El-Sawi et al.,[46] who reported a decrease in acidity after treating OMW with composting.

The change in density of the treated OMW sample from 1.003 to 1.000078 g/mL after treatment with the three-column system indicates a decrease in the density of the wastewater. This decrease in density may be attributed to the removal of suspended solids, organic matter, and other pollutants from the wastewater during treatment. Al-Hmoud et al.[35] investigated the use of a hybrid system combining coagulation-flocculation and membrane filtration for the treatment of OMW. They reported a decrease in density from 1.012 to 1.008 g/mL after treatment, which they attributed to the removal of suspended solids and organic matter. Similarly, Achak et al.[34] investigated the use

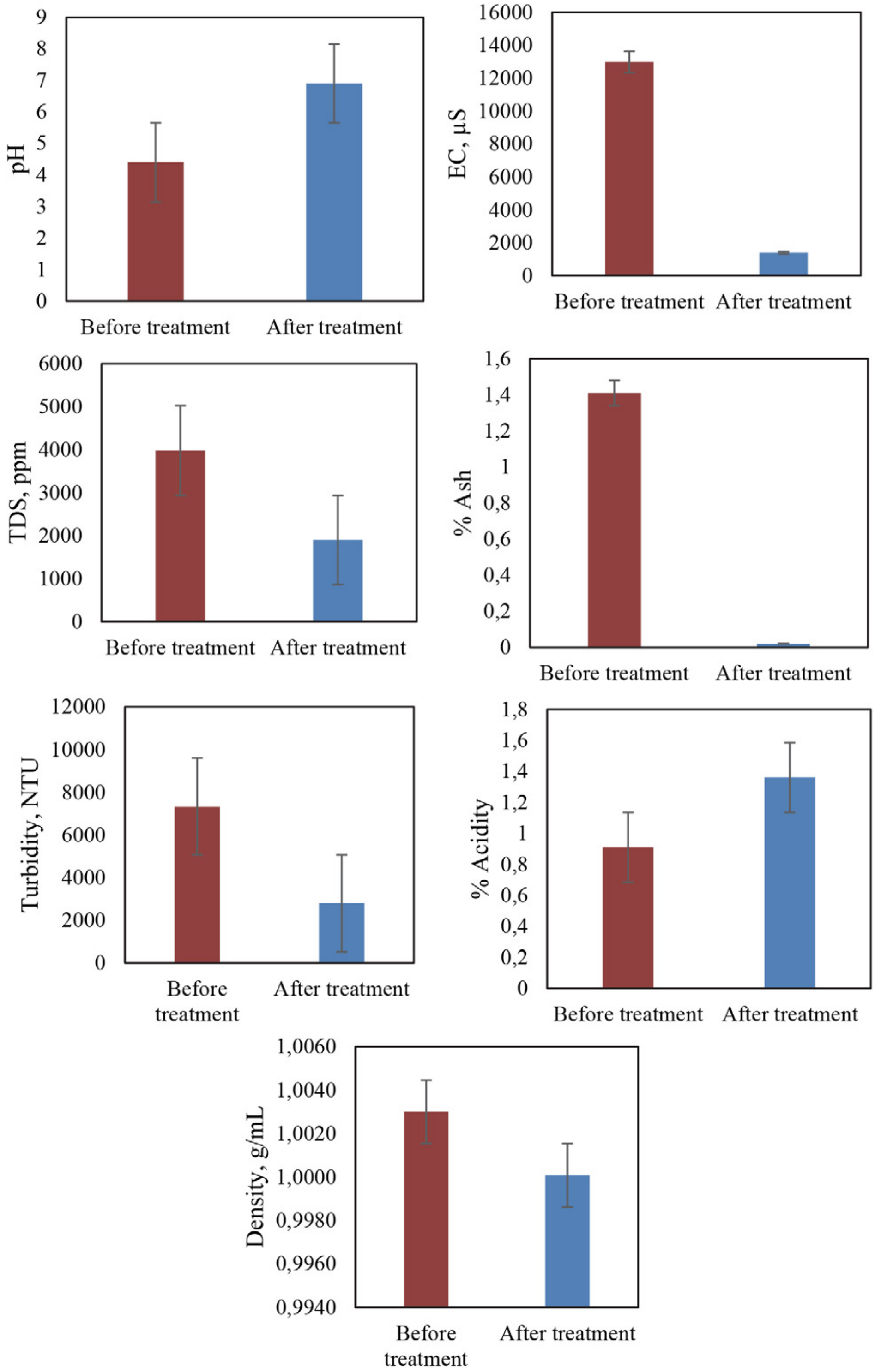

Figure 2. Physicochemical parameters of OMW sample before and after treatment

of a two-stage treatment system involving an anaerobic digestion followed by a sequencing batch reactor for the treatment of OMW. They reported a decrease in density from 1.023 to 1.008 g/mL after treatment, which they attributed to the removal of organic matter and the production of biogas.

3. 1. 2. The Levels of Anions

The levels of significant anions, such as chloride (Cl^-), nitrite (NO_2^-), nitrate (NO_3^-), sulphate (SO_4^{2-}), and phosphate (PO_4^{3-}), in olive mill wastewater (OMW) before and after treatment are depicted in Figure 3. Error bars show the standard deviation, and the data are displayed as the

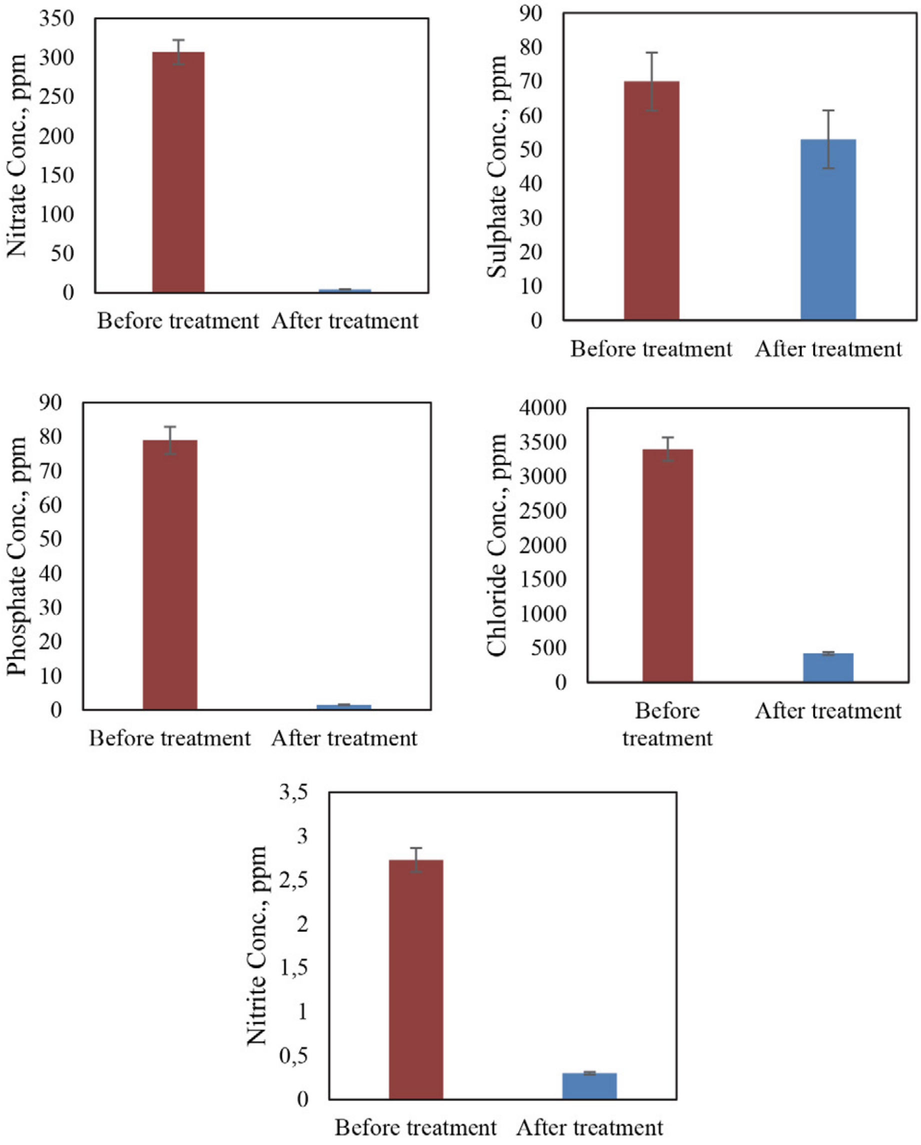

Figure 3. Some anions levels of OMW sample before and after treatment

mean of five separate replicates. Following treatment, there were significant decreases in these anions' concentrations, demonstrating the efficacy of the suggested treatment approach. The nitrate concentration decreased from 307 to 4.3 mg/L, indicating a significant reduction in the concentration of nitrogen compounds. The decrease in nitrate concentration is consistent with the findings of other studies that have investigated the use of various treatment methods for OMW, such as the use of constructed wetlands and electrocoagulation.[47]

Similarly, the nitrite concentration decreased from 2.73 to 0.3 mg/L, indicating a significant reduction in the concentration of nitrogen compounds. The decrease in nitrite concentration is consistent with the findings of Aly et al. (2014),[32] who reported a decrease in nitrite concentration after treating OMW with electrocoagulation.

The sulphate concentration decreased from 60 to 53 mg/L, indicating a reduction in the concentration of sulphate compounds. This reduction in sulphate concentration is consistent with the findings of other studies that have investigated the use of various treatment methods for OMW, such as the use of reverse osmosis and integrated membrane bioreactors.[48]

Furthermore, the phosphate concentration decreased from 79 to 1.5 mg/L, indicating a significant reduction in the concentration of phosphorus compounds. This reduction in phosphate concentration is consistent with the findings of other studies that have investigated the use of various treatment methods for OMW, such as the use of integrated membrane bioreactors and electrocoagulation.[49]

The treatment of OMW sample using the proposed method resulted in a significant decrease in chloride levels, from 3400 to 422 mg/L. This decrease in chloride concentration is consistent with the findings of other studies that have investigated the use of various treatment methods for OMW, such as the use of reverse osmosis[48], biological treatment with fungi,[50] and electrocoagulation.[41] The decrease in chloride concentration observed in the current study is particularly significant, as high chloride levels can cause environmental and health problems. High chloride levels in wastewater can cause corrosion of pipes and other infrastructure, as well as damage to aquatic ecosystems. Moreover, the decrease in chloride concentration observed in the current study is comparable to the findings of other studies that have investigated the use of various treatment methods for OMW. Mekki et al.[31] reported a decrease in chloride concentration from 3100 to 85 mg/L after treating OMW with electrocoagulation. Similarly, Aly et al.[32] reported a decrease in chloride concentration from 3500 to 525 mg/L after treating OMW with reverse osmosis. Furthermore, Hodaifa et al.[40] reported a decrease in chloride concentration from 1700 to 75 mg/L after treating OMW with biological treatment using fungi. Additionally, Azzam et al.[33] reported a decrease in chloride concentration from 1800 to 50 mg/L after treating OMW with composting.

3. 1. 3. The Levels of Minerals

Key mineral levels in olive mill wastewater (OMW), such as sodium (Na), potassium (K), calcium (Ca), iron (Fe), and copper (Cu), are shown in Figure 4 both before and after treatment. Error bars indicate the standard deviation, and the data are displayed as the mean of five separate replicates. The efficacy of the treatment system in eliminating mineral pollutants from the OMW was demonstrated by the considerable reduction in these minerals' concentrations following treatment. The potassium (K) concentration decreased from 2243 to 86 mg/L, calcium (Ca) concentra-

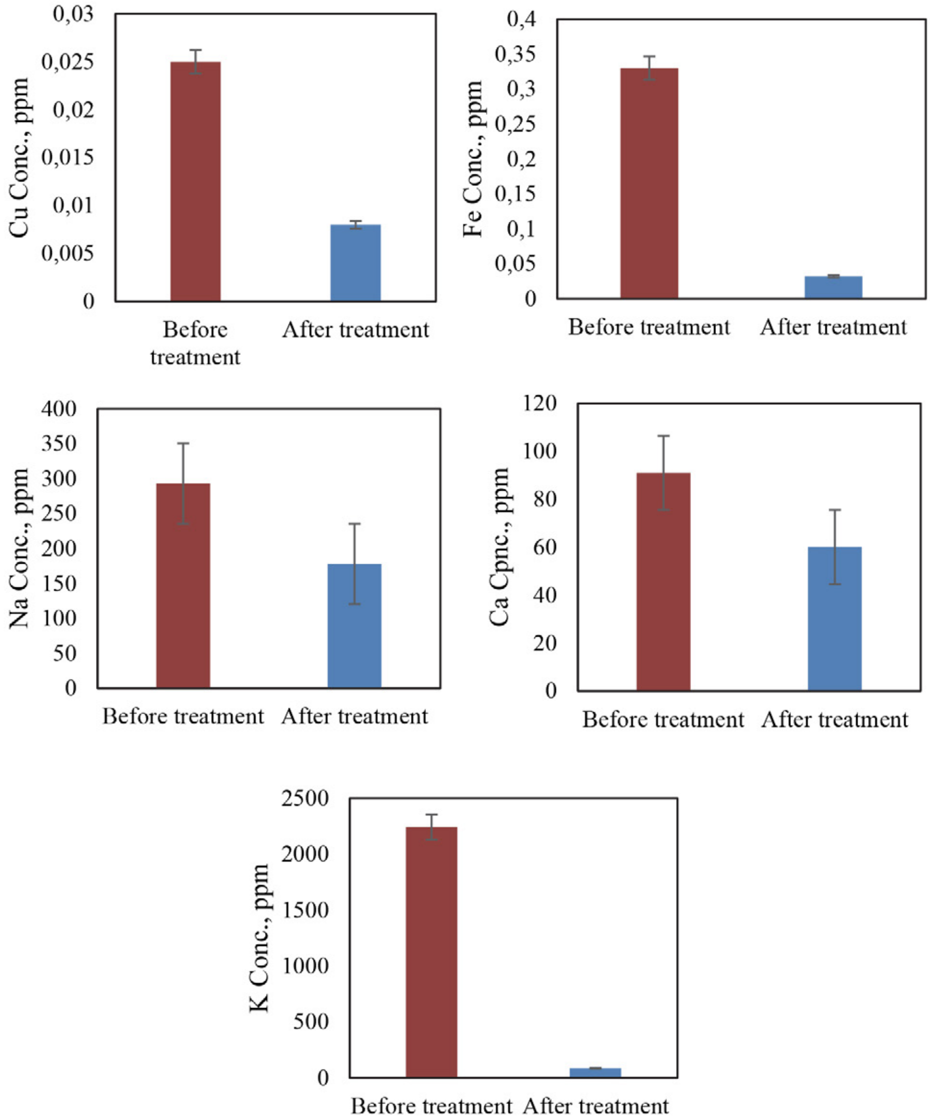

Figure 4. Minerals levels of OMW sample before and after treatment

tion decreased from 91 to 60 mg/L, sodium (Na) concentration decreased from 293 to 178 mg/L, iron (Fe) concentration decreased from 0.33 to 0.032 mg/L, and copper (Cu) concentration increased from 0.025 to 0.008 mg/L.

These changes in mineral concentrations are consistent with the findings of other studies that have investigated the use of various treatment methods for OMW, such as the use of electrocoagulation,[41] reverse osmosis,[48] and biological treatment.[50] Rakhmania et al. (2022)[49] reported a decrease in K concentration from 1970 to 119 mg/L after treating OMW with electrocoagulation. Similarly, Aly et al.[32] reported a decrease in K concentration from 2320 to 58 mg/L after treating OMW with reverse osmosis. Moreover, Hodaifa et al.[40] reported a decrease in Ca concentration from 217 to 115 mg/L after treating OMW with biological treatment using fungi. Additionally, Dermeche et al. (2013)[42] reported a decrease in Na concentration from 309 to 79 mg/L after treating OMW with electrocoagulation. Furthermore, the decrease in Fe concentration observed in the current study is consistent with the findings of other studies that have investigated the use of various treatment methods for OMW. For instance, Dermeche et al.[42] reported a decrease in Fe concentration from 0.33 to 0.032 mg/L after treating OMW with reverse osmosis.

3. 1. 4. The Levels of Total Phenols and Total Flavonoids

The concentrations of total flavonoids and total phenols in olive mill wastewater (OMW) before and after treatment are shown in Figure 4. The standard deviation is shown by the error bars, and the results are the mean of five separate replicates. Both total phenols and total flavonoids significantly decreased as a result of the treatment procedure, demonstrating how well the system works to remove organic contaminants. As shown in the figure, a significant decrease in the levels of total phenols and total flavonoids was observed. The total phenols concentration decreased from 9821 to 35 mg/L, and the total flavonoids concentration decreased from 2560 to 9 mg/L.

These changes in phenolic compounds are consistent with the findings of other studies that have investigated the use of various treatment methods for OMW, such as the use of membrane filtration,[51] adsorption,[45] and biological treatment.[50] Solomakou et al.[45] reported a decrease in total phenols concentration from 9820 to 30 mg/L after treating OMW with adsorption method. Similarly, Aly et al.[32] reported a decrease in total phenols concentration from 10200 to 350 mg/L after treating OMW with zeolite-based adsorption method. Moreover, Nogueira et al.[50] reported a decrease in total flavonoids concentration from 320 to 20 mg/L after treating OMW with biological treatment using fungi. Additionally, Flores et al.[41] reported a decrease in total flavonoids concentration from 460 to 20 mg/L after treating OMW with a single electrocoagulation with different electrodes.

However, the decrease in both total phenols and total flavonoids concentrations observed in the current study is particularly significant, as their presence in OMW in high levels can have negative impacts on the environment and human health.[3] These bioactive compounds can be toxic to aquatic organisms and can cause soil and water pollution. Moreover, the discharge of OMW into the environment can lead to the proliferation of harmful bacteria and pathogens, which can pose a risk to human health. Therefore, reducing the concentration of these compounds in OMW is important to mitigate their negative impacts on the environment and human health.[6]

3. 2. Chemical Profile of OMW Before and After Treatment

The three-column continuous system, utilizing various adsorbent materials, including biosorbents made from olive leaves, was efficient in treating olive mill wastewater. The treatment process resulted in significant reductions in most of the measured properties, including pH (56.8%), electrical conductivity (89.2%), total dissolved solids (52.2%), turbidity (61.9%), density (0.22%), acidity (41.8%), ash content (98.6%), chloride (87.6%), nitrite (89.0%), nitrate (98.6%), sulfate (11.7%), phosphate (98.1%), sodium (39.2%), potassium (96.2%), calcium (34.1%), iron (90.3%), copper (68.0% increase), total phenols (99.6%), and total flavonoids (99.6%).

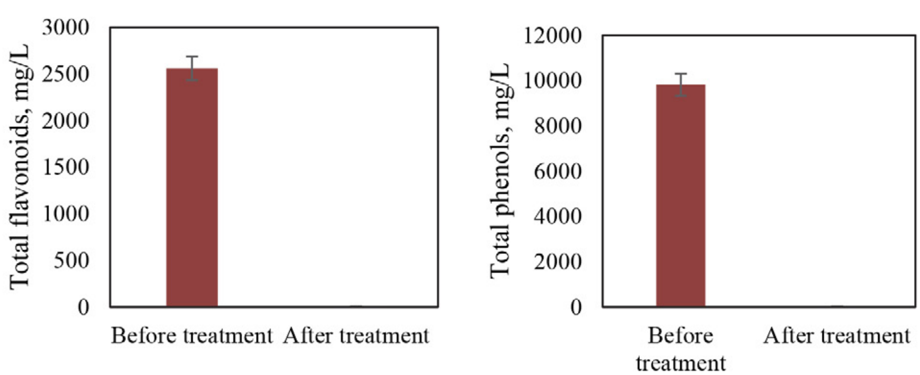

Figure 5. Total phenols and total flavonoids levels of OMW sample before and after treatment

These results are desplayed in Table 3. To enhance understanding of the treatment's effectiveness, the table now includes literature values for permissible limits and previously reported concentrations for each parameter for comparison.

The reduction of total phenols from 9821 to 35 mg/g, and total flavonoids from 2560 to 9 mg/g, highlights the efficiency of the treatment method in removing the organic pollutants from the wastewater. The reduction of chloride from 3400 to 422 ppm, nitrate from 307 to 4.34 ppm, and nitrite from 2.73 to 0.3 ppm, demonstrates the significant reduction of the nitrogen-based pollutants in the wastewater. The reduction of turbidity from 7328 to 2791 NTU, electrical conductivity from 12990 to 1407 μS, and total dissolved solids from 3980 to 1901 ppm, indicates the efficiency of the treatment method in removing the suspended and dissolved solids from the wastewater.

Statistical analysis using paired t-tests confirmed the significance of the reductions observed for all parameters before and after treatment, with p-values below 0.05 for each parameter. The t-statistic values significantly exceed the critical t-value, further emphasising the substantial differences between the untreated and treated samples.

These findings suggest that the three-column continuous system is an effective and eco-friendly method for treating olive mill wastewater, which is a significant environmental issue in the olive oil industry. The reduction percentages highlight the efficiency of the treatment method in removing the pollutants, which could contribute to preserving the environment and promoting sustainable practices in the olive oil industry.

3. 3. Green Profile Analysis of the OMW Purification System

In response to the growing environmental impact of chemical processes and products, green chemistry (GC) was developed in the 1990s. It focuses on creating chemical products and processes that, from design to disposal, reduce or completely eliminate the use and production of hazardous materials. Green analytical chemistry (GAC), which aims to minimise environmental effects during laboratory processes, is an evolution of this concept within analytical chemistry. GAC adheres to the 12 principles of green chemistry and prioritises minimising energy use, eliminating solvent-intensive procedures, and avoiding unnecessary derivatisation. To assess the sustainability of analytical techniques and facilitate comparison and development, a number of green metrics have been created, including the analytical Eco-Scale, GAPI, and AGREE. These tools are crucial for evaluating and enhancing the green profile of procedures such as the OMW treatment process outlined here. They are widely used across techniques like spectrophotometry, chromatography, and fluorometry.[52,53]

The environmental impact of the olive mill wastewater (OMW) treatment process was evaluated using several green analytical chemistry tools, focusing on material use, energy consumption, and waste generation. As shown in Figure 6, which provides a comprehensive green profile assessment of the treatment process, the pie chart (Figure 6a) indicates that the materials used in the treatment process primarily consist of natural substances. Coarse sand, fine sand, and limestone together account for 37.5% of the total material usage, while olive leaf powder and activated carbon represent smaller portions (12.5% each). Distilled

Table 3. Chemical Profile of OMW Before and After Treatment Compared to Literature Values

Parameter	Literature	Before	After	% reduction	t-Statistic	p-Value (Two-Tail)
pH	3.80–5.20[31]	4.40±0.13	6.90±0.22	56.8	79	2E-5
EC (μS/cm)	6400–18000[34]	12990±390	1407±61	89.2	2E3	2E-13
TDS (ppm)	3200–30000[32]	3980±89	1901±75	52.2	668	3E-11
Turbidity (NTU)	2000–16800[35]	7328±146	2791±113	61.9	2500	2E-13
Density (g/mL)	0.944–1.810[33]	1.0030±0.0401	1.0001±0.0000	0.22	3	0.04
% Acidity	–	0.91±0.04	0.53±0.03	41.8	38	3E-10
% Ash	0.33–1.64[34]	1.41±0.04	0.02±0.001	98.6	440	2E-10
Cl^- (ppm)	1000–8000[40]	3400±68	422±19	87.6	2E3	2E-13
NO_3^- (ppm)	200–2000[37]	307±12	4.3±0.18	98.6	476	1E-10
NO_2^- (ppm)	–	2.73±0.15	0.3±0.01	89.0	768	2E-11
SO_4^{2-} (ppm)	10–1500[36]	60±2.5	53±0.75	11.7	16	3E-7
PO_4^{3-} (ppm)	20–200[37]	79±3.2	1.5±0.08	98.1	271	1E-9
Na (ppm)	1000–24000[43]	293±10	178±6.9	39.2	364	3E-10
K (ppm)	1600–4300[42]	2243±67	86±3.3	96.2	3E3	7E-14
Ca (ppm)	20–300[34]	91±3.6	60±3.0	34.1	69	2E-12
Fe (ppm)	0.9–9.9[42]	0.33±0.01	0.032±0.002	90.3	118	3E-8
Cu (ppm)	0.01–0.1[43]	0.025±0.001	0.008±0.0002	68	13	2E-4
Total phenols (mg/g)	840–12800[31]	9821±392	35±1.25	99.6	3E4	7E-18
Total flavonoids (mg/g)	0.8–12.9[44]	2560±51	9±0.35	99.6	8E3	1E-15

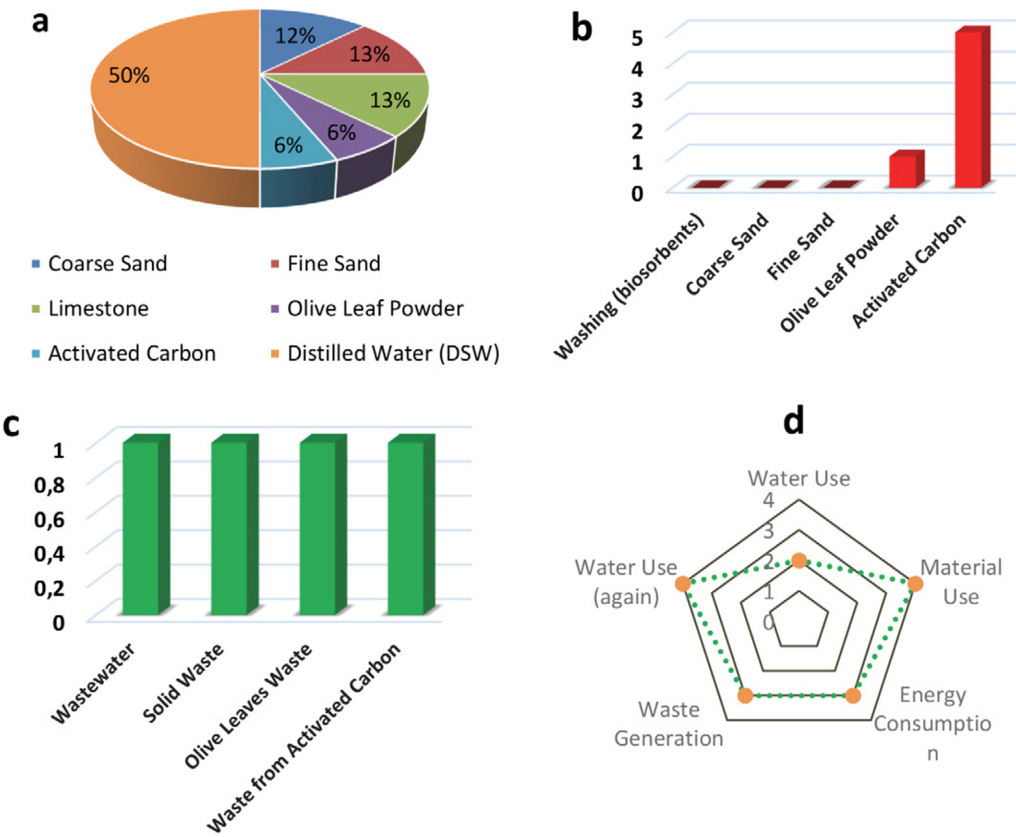

Figure 6. Green profile assessment of the OMW treatment process: a) Material usage breakdown, b) Energy consumption profile, c) Waste generation, d) Overall greenness of the method

water, mainly used for washing the biosorbents, constitutes the largest share (50%) of the material usage.

Energy consumption during the process was quantified in arbitrary units, as illustrated in the bar chart (Figure 6b). The washing and preparation of the materials (coarse sand, fine sand, and limestone) required minimal energy, whereas the activation of the carbon material in an air-isolated furnace at 600 °C resulted in a significantly higher energy demand. The highest energy consumption (value 5) was linked to activated carbon production, as highlighted in the chart.

In terms of waste generation, the process produced relatively low waste across various categories, including wastewater, solid waste, olive leaf waste, and waste from activated carbon, each represented equally in the bar chart (Figure 6c). Waste generation from the entire process was measured at a uniform level (arbitrary unit of 1), reflecting the eco-friendly nature of the procedure. Finally, the radar chart (Figure 6d) illustrates the overall green profile of the treatment method, indicating its low environmental impact. The treatment process achieves a balance of low water use (value 2), moderate material use (value 4), and minimal waste generation (value 3), making it a sustainable alternative for the purification of OMW. The most significant challenge to its green profile is the high energy consumption associated with activated carbon production, which is essential for enhancing the effectiveness of the treatment system.

3. 4. Environmental Impact and Valorisation of Used Adsorbent Materials

In addition to its high efficiency in purifying olive mill wastewater (OMW), the proposed three-column system provides opportunities for the valorisation of the retained compounds, especially phenolic compounds and oligosaccharides, which are known for their potent antioxidant and antimicrobial properties and can be extracted from the adsorbent materials for use in integrated pest management and plant protection. According to recent studies, including those by Greco et al., phenolic extracts from olive mill waste have been shown to induce plant innate immunity, decrease the need for chemically synthesised pesticides, and improve resistance to phytopathogens like Botrytis cinerea and Pseudomonas syringae.[54] Additionally, the oligosaccharides that were retained during treatment, such as arabino-oligosaccharides and oligogalacturonides, have demonstrated potential as glycan elicitors, capable of priming plant defence mechanisms and enhancing crop resilience.[55]

OMW may be converted into a source of valuable bioproducts by extracting and recovering these bioactive components, which is in line with the principles of the circular economy. In addition to addressing waste management issues, this strategy lowers pollution levels in the environment and encourages the sustainable use of agricultural waste. Future research might concentrate on improving recovery strategies for these substances, utilising scalable approaches and eco-friendly solvents to optimise their ecological and economic worth.

4. Conclusions

The three-column continuous system, proposed in the current study, using various adsorbent materials, including biosorbents made from olive leaves, was found to be an effective method for treating olive mill wastewater. The treatment process resulted in significant reductions in total phenols (from 9821 to 35 mg/g), chloride (from 3400 to 422 ppm), nitrate (from 307 to 4.34 ppm), turbidity (from 7328 to 2791 NTU), K (from 2243 to 86 ppm), Na (from 293 to 178 ppm), and the other measured properties. These results highlight the potential of using eco-friendly and sustainable methods for treating olive mill wastewater, a significant environmental issue in the olive oil industry.

This continuous treatment system has proven to be highly effective in treating wastewater from olive mills, resulting in notable reductions in important pollutants and highlighting its potential for widespread industrial use. We advise the olive oil sector to use this approach for practical implementation, especially because environmentally friendly biosorbents like olive leaves offer a more affordable choice to conventional treatment techniques. Scalability and the possibility of adsorbent fouling over time, however, are significant drawbacks that need additional investigation. Future studies should concentrate on improving the regeneration of biosorbents in order to increase sustainability and lower costs. Furthermore, investigating the recovery of important substances from the treated wastewater, such as oligosaccharides and phenolic compounds, may have financial advantages and enhance the process' sustainability. This method may have a greater environmental impact if it is used to clean wastewater from other agricultural industries.

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Povzetek

Namen študije je bil preveriti izvedljivost in učinkovitost uporabe kontinuirnega sistema s tremi kolonami za čiščenje odpadne vode iz oljarn (OMW). Metodologija je vključevala prehajanje odpadne vode skozi tri zaporedne kolone, polnjene z različnimi adsorbenti. Učinkovitost je bila ocenjena z merjenjem več kemičnih in fizikalnih lastnosti pred in po obdelavi, vključno s pH, električno prevodnostjo (EC), skupnimi raztopljenimi trdnimi snovmi (TDS), kislostjo, motnostjo, kloridom, nitratom, nitritom, sulfatom, fosfatom, Na, K, Ca, Fe, Cu, skupnimi fenoli, in skupnimi flavonoidi. Začetne vrednosti teh parametrov so se med vzorci razlikovale. Po obdelavi so se izmerjene količine bistveno spremenile, vključno z zvišanjem pH s 4,8 na 6,9, z znižanjem EC s 12990 μS/cm na 1407 μS/cm, motnosti iz 7328 NTU na 2791 NTU, kloridov s 3400 ppm na 422 ppm, nitratov s 307 ppm na 4,34 ppm, Na s 293 ppm na 178 ppm, K s 2243 ppm na 86 ppm, Ca s 91 ppm na 60 ppm in znatno zmanjšanje skupnih fenolov s 9821 mg/g na 35 mg/g. Rezultati kažejo, da je predlagana obdelava učinkovita za čiščenje OMW in ponuja vpogled v razvoj trajnostnih in okolju prijaznih metod čiščenja odpadne vode.