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Utilization of biobed for the efficient treatment of olive oil mill wastewater

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ABSTRACT

Simple and low-cost field systems called biobeds have been successfully developed in Sweden to reduce the environmental pollution caused by pesticides. Although these systems have been efficiently used for the treatment of a broad range of wastewaters worldwide, however, their utilization for olive oil mill wastewaters (OMWW) treatment is limited. The aim of this study was the examination of a pilot soil tank of 1 m^3 (biobed) operation for treatment, purification, and detoxification of a highly loaded olive oil wastewater effluent and the determination of the appropriate mixture of filling material consisted of compost and soil and the corresponding bed layer thickness for the optimum removal capacity of pollutants. A handmade irrigation system was developed to inject olive mill wastewater onto the biobed. Raw and effluent samples were collected in a feed and a storage tank, respectively. The effluent from the storage tank was recycled to the inlet of the system and the operation was carried out in subsequent treatment cycles; the concentration of various pollutants in the influent, the effluent, and the recycled stream was measured on a weekly basis together with the corresponding flow rates. The overall removal capacity as a function of the treatment cycles was studied, as well as the behavior of the biobed due to blockage and placental buildup of solids in the upper part. Among them, significant results were obtained, such as a great decrease in biochemical oxygen demand (96%), chemical oxygen demand (92%), total phenols (88%), total Kjeldahl nitrogen (85%), and NH_4^+ (100%) and important incensement of pH values. It was concluded that the recycling of the effluent was beneficial in pollutants removal; effluent collected after the third treatment cycle had the appropriate quality for being either reused in irrigation–watering or discharge into natural recipients, meeting the standards of relevant wastewater and soil conditioner legislation.

Keywords: OMWW treatment; OMWW purification; OMWW disposal; Biobed; Biomixture; Irrigation

1. Introduction

In the early 1990's Prof. Lennart Torstensson and Maria del Pilar Castillo, together with colleagues at the Swedish University of Agricultural Sciences developed the first biobed system, intended to collect, and degrade spills of pesticides on farms, in order to reduce environmental contamination from pesticide use. They designed and implemented a simple underground biological filter, filled with a clay layer at the bottom, a biomixture of soil, peat, and straw as an intermediate layer, and a grass layer at the surface [1,2].

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Its effectiveness lies in its capacity to reduce pesticide concentrations under certain conditions, due to their adsorption and degradation by the organic components and the microbial load of the biomixture, respectively [2,3]. Since the 2000's the Swedish biobed has been adopted in many countries in Europe (Belgium, Italy, France, and UK) and in Latin America, (Chile, Peru, Ecuador, and Guatemala), where it has been adapted to the local needs and often renamed (biofilter in Belgium, biomass in Italy, phytobac and biobac in France, and biodep in Guatemala). The composition of the biomixture may be modified in response to the needs of each application and therefore change the amounts and/or the activity of the microbial community that is developed, leading to different system performance.

Meanwhile, disposal and handling of huge quantities of olive mill waste waters (OMWW) remain one of the crucial issues for the Mediterranean basin countries, which have to face severe environmental problems, due to the production and irrational disposal of immense amounts of olive mill wastes in short periods of time [4]. The situation gets worse because olive oil production is seasonal, so the treatment process should be flexible enough to operate in a non-continuous mode, otherwise olive mill wastewaters should be stored until further processing. Currently, OMWW are improperly treated, being left-out in outdoor storage/evaporation lagoons, while during periods of high precipitations, they may reach water bodies and cause serious deterioration to the underground water properties [5]. Due to cost restrictions and seasonality of the flowrates, usually, no treatment plants are available at the mills [6]. Despite the fact that in all EU countries the direct discharge of OOMWW into rivers, lakes, valleys, and uncontrolled ponds is strictly forbidden and although in many cases, the illegal direct disposal of OOMWW into nearby aquatic resources and ecosystems has been observed, there is still no common EU legislation for olive oil mill wastes (OOMW) regulation and management.

The main factors that are responsible for OMWW associated pollution problems are their high organic load, their strong offensive smell, and their high content of phenolic compounds. Deterioration of natural waters' quality and soil degradation are serious problems, as indicated by coloring, the appearance of an oily layer, increased oxygen demand (high levels of biochemical oxygen demand (BOD) and chemical oxygen demand (COD)) and toxic effect on plants and soil microflora, when disposed on soil [7]. Therefore, due to their chemical characteristics, OMWW requires specific treatment in order to reduce their potentially negative environmental impact. High values of acidity, organic load (COD and BOD_5), and specifically elevated concentrations of organic matter of low degradability (COD/BOD_5 ratio between 2.5 and 5) [8], high electrical conductivity [9], high values of solid matter [10], organic compounds (lignins and tannins, related to its dark color), long-chain fatty acids and phenolic compounds [11,12] contribute to their hard degradability and their imminent toxicity to most crops and microorganisms [13,14]. Additionally, acidic conditions in combination with the polyphenols' complexing abilities increase the mobility of heavy metals in the environment [15].

A lot of researchers have used several mechanical, physical, chemical, biological, and thermal methods, as single process or in combination, such as oxidation, membrane filtration, centrifugation, flocculation/coagulation, incineration, ultrafiltration, reverse osmosis, ozonation, and photolysis, in order to treat OMWW [16]. Most of them are quite useful as pretreatment methods, but inefficient on pollutants removal and moreover, they do not generate valuable sub-products [17], while conventional biological processes (aerobic or anaerobic) have shown moderate efficiencies in terms of OMWW mineralization [18]. Furthermore, several studies have been carried out in order to use this waste as a renewable resource [19,20] and to convert OMWW from waste to bio-fertilizer, irrigation water, and green fuels. A lot of management methods have been developed in terms of recycling OMWW as a soil amendment, either in fresh untreated form or after further treatment, in order to recover organic materials to be applied in the field [6,21]. However, these methods are expensive and laborious, require monitoring, and may generate recalcitrant byproducts.

The proposed study aims at designing, installing, and operating a simple, experimental, and inexpensive biobed system for the treatment, purification, and detoxification of olive oil wastewaters. The biobed is a soil tank system filled in with a special biomixture consisting of compost, soil, and straw, which behaves as a natural biological filter for degradation of organic matter and solids retention. Moreover, handling of treated effluent was investigated, either for irrigation or discharge to water bodies, while the potential application of the produced biomixture was also assessed in terms of soil amendment or composting. Meanwhile, rejuvenation and reactivation of the used biomixture after 1 y of inactivity took place, to study its effectiveness.

2. Materials and methods

2.1. Raw materials

The applied OMWW was collected from a three-phase centrifugal olive oil mill located at Ormylia, Halkidiki, and Greece. Three batch oil centrifugal decanters with heating were used for olive oil extraction and raw wastewater from the olive oil production process was stored onsite in a cement tank, in order to avoid potential contamination by sewage and soil, for less than 1 month at the time sample of one tonne was collected for the experiment. The collected sample was stored at 10°C–12°C and was used in the experiments without any prior treatment or purification.

Commercial wheat straw suitable for animal feed was shredded into small pieces (2–3 cm length) and passed through a special crusher machine before mixing with the soil, in order to achieve more efficient homogenization of the biomixture and to facilitate carbon absorption by microorganisms. A sandy loam soil (sand 59%, silt 24%, and clay 17%) was collected from the upper soil layer (0–20 cm) of a cultivated field next to the premises of Soil and Water Resources Institute (SWRI) of Hellenic Agricultural Organization "DEMETER" in Sindos, Greece, then it was manually cleaned from the stones and finally, it was left for a few days to dry (soil moisture was monitored until it was reduced to 30%). Earthworm compost from cows manure was taken from a soil amendment company, while clay and gravel were obtained from a brick factory.

2.2. Biomixture preparation

A biomixture of straw, soil, and compost (50:25:25 vol %) was prepared in order to fill the biobed. Preparation of the biomixture took about 4 months (February–March 2016). This period of year was chosen because it was considered more appropriate, taking into account the fact that olive growers are faced with the management of their waste after the completion of the olive production process. The materials were mixed with a concrete mixer. Approximately 800 L of biomixture were prepared, which were left outside, under shed, for 12 weeks. The initial biomixture water was about 10%, so the biomixture was wet day by day with 50 L of water in order to increase its humidity and shake manually with a shovel, for 1 week. This first week during which the water content of the biomixture reached a level of 47%–48%, was considered to be the first week of digestion. For the remaining 11 weeks, the temperature was monitored and the water content was maintained to the desired level by adding water and stirring the biomixture manually with a shovel. During this period, C/N ratio was regularly measured and monitored. At the end of the 4th week the C/N ratio was 14%, at the end of the 6th week it was 16.5%, while at the end of the 7th and 12th week, C/N ratio was 19.7% and 18.2%, respectively. The main physico-chemical characteristics of the biomixture prepared after the 12 weeks period and before inserting it into the biobed are reported in Table 1. The pH was measured in a mixture of air-dried substrate and deionized water (1:5 w/v), based on methods of soil analysis [22]. Organic carbon (OC) content was analyzed using the dry combustion method, based on methods of soil analysis [23] and total N was determined after digestion with H_2SO_4 according to ISO 11261:1995 [24]. Total phenols were analyzed according to the method of Folin–Ciocalteu using gallic acid for standard curve preparation [25].

2.3. Biobed preparation

The biobed was constructed with galvanized steel. The dimensions of the biobed were: 1.00 m × 0.95 m × 0.79 m (height). Six drainage pipes were placed inside the biobed, which ended up in a drainage container fitted next to the biobed (1.00 m × 0.20 m × 0.50 m) and three ventilation pipes were fitted above them, to ensure the existence of aerobic conditions. Dry clay was placed at the bottom of the biobed at a height of 3 cm (Fig. 1). Above the clay layer and at a height of about 8 cm, gravel was placed halfway through the drain pipes (making sure that the slits on the top of the pipes were not covered) in order to allow the easier drainage of the liquid waste (Fig. 2). Six tubes of appropriate size were fitted on the biobed to allow smooth and uniform inflow of the liquid waste into the biobed (Fig. 3). About 560 L of biomixture were carefully

Table 1
Characteristics of the biomixture

Parameters	Raw biomixture
pH	7.7
Humidity (%)	45
Bulk density (g/mL)	1.23
O.C. (%)	11
N_{total} (%)	0.54
C/N ratio	20

Bulk density in the biobed was almost the same with the bulk density of the biomixture.

Fig. 1. Clay and gravel placement at the bottom of the biobed.

Fig. 2. Biomixture placement above the gravel.

Fig. 3. Input of OOMWW into the biobed.

placed over the gravel (Fig. 2). The biobed was connected in series with two tanks each with 1 ton capacity (Fig. 4). One of the tanks was filled with raw OOMWW, while the other tank was empty. A rotary pump was used in order to transfer and introduce the liquid waste from the tank to the biobed. The drains collected in the corresponding container were transferred to the empty tank with the help of an automatic submersible drainage sump pump so that the collection tank does not flood since the drainage time could not be calculated. Pump picked up the signal from the dipstick level inside the container. Valves were installed at each inlet and outlet of the pipes to regulate flow and reverse fluid flows, to avoid overflow on the surface of the biobed.

3. Experiment setup

The study was carried out at Soil and Water Resources Institute (SWRI) – Land Reclamation Department of Hellenic Agricultural Organization DEMETER in Sindos, Greece. The operation of the biobed occurred for two subsequent periods: from May to June 2016 and from April to May 2017, referred to the as first and second period, respectively. A trial operation of the biobed took place in the first period. One tonne of fresh liquid waste was transported from a three-phase centrifugal olive oil mill into the tank and four cycles of the experiment were carried out. Each time, the liquid waste was discharged from the tank into the biobed (Fig. 4) and the drainage (filtered waste) was discharged from the biobed container into the empty tank. In both periods, it was observed that after the end of the first cycle, it was possible to filter 85% of the liquid waste, since the rest was deposited at the bottom of the tank, forming a thick crust (mourga), which was removed before the beginning of the second cycle. Mourga made up about 15% of the waste, settled in the tank, and due to its dense composition could not be filtered. However, this material was analyzed and used for another experimental projects, concerning composting, and feed additives. Three drainage samples were collected after the end of each cycle to increase the reliability of the results, which were further analyzed in the laboratory. A fifth wash-out cycle with 100 L of tap water was also performed in 1.17 h during the first period and analysis of the drainage was also conducted. The above experimentation of the first period resulted in six sets of liquid samples (three replicates per set) and nine biomixture samples. The liquid samples were the raw OOMWW and the five drainages and the biomixture samples were the raw biomixture, four biomixture samples from the four cycles, collected from the upper layer of the biomixture (0–15 cm depth), while at the end of the fifth cycle, biomixture samples were collected from four different depths (0, 15, 30, and 45 cm) in order to conclude whether the biomixture could be used for more cycles or periods.

For the second experiment period, 1 ton of fresh liquid waste from the same olive oil mill was applied and five cycles of the experiment were carried out. Three drainage samples were collected after the end of each cycle for chemical analysis. Consequently, six sets of liquid samples were collected (including the raw OOMWW), as well as six biomixture samples (including the previous year biomixture). "Rejuvenation" of the biomixture (achieving desired humidity, temperature, etc.) took place with controlled wetting, but without disturbing the material. Upon completion of the first cycle, all the placenta (materials that cannot be filtered and stand on the surface of the biomixture) created on the upper surface layer of the biomixture was subtracted (6.5 kg of placenta/m^3 of biomixture). A complete chemical analysis of the placenta took place. This intervention prevented placenta re-creation in subsequent cycles, resulting in improved biobed function (based on the results of both periods), higher application speed, and shorter residence time, as well as a higher rate of detoxification of the liquid waste (Table 2). Each time, the liquid waste was discharged from one feed tank into the biobed and the drainage was discharged from the biobed container into the empty tank. Details on fluid supplies and application speed for each of the two experimentation periods and the corresponding cycles are presented in Table 2.

The physicochemical parameters of OOMWW and the drainages were determined according to Standard Methods for the Examination of Water and Wastewater Handbook [26]. The parameters of the outputs measured in the various batches were: BOD, COD, total phenols, total solids, volatile solids, total suspended solids, electrical conductivity,

Fig. 4. Biobed system with an OOMWW tank on the right and a leaches collection tank on the left.

Table 2
Fluid supply and application speed of both periods

	Fluid supply (L/h)		Application speed (m/h)	
Cycles	First period	Second period	First period	Second period
First cycle	83.16	86.54	0.08	0.09
Second cycle	81.17	94.21	0.08	0.09
Third cycle	94.06	97.05	0.09	0.10
Fourth cycle	90.20	92.31	0.09	0.09
Fifth cycle	–	94.10	–	0.09

First period refers to the period from May to June 2016, when the experiment took place for the first time, while second period refers to the period from April to May 2017, when the experiment took place for the second time.

pH, total nitrogen Kjeldahl, nitrates, ammonia, phosphorus Olsen, sulfate, K, Na, Ca, Mg, Fe, Mn, Cu, Zn, and B. Total phenols were assessed according to the method of Folin–Ciocalteu using gallic acid for standard curve preparation [25].

The physicochemical parameters of the biomixtures and the placenta were determined according to Methods of Soil Analysis Handbook [27] and Standard Methods for The Examination of Water and Wastewater Handbook [26]. The parameters of the samples measured were: total phenols, total solids, volatile solids, organic matter, calcium carbonate, electrical conductivity, pH, total nitrogen Kjeldahl, nitrates, phosphorus Olsen, exchangeable K, Na, Ca, Mg, Fe, Mn, Cu, Zn, and B. Total phenols were assessed according to the method of Folin–Ciocalteu using gallic acid for standard curve preparation [25].

4. Results

The results of the physico-chemical analysis of the OOMWW and the drainages for each period are presented in Tables 3–5.

An increase in the pH of the raw OOMWW and the drainages was observed for both periods, moving from acidic to alkaline, while electrical conductivity decreased by 53% at the end of the fourth cycle in the first period and 62% at the end of the fifth cycle of the second period.

Regarding COD concentration and as presented in Fig. 5, a reduction of up to 78% was observed for the first period of experiments, while a higher reduction was observed for the second period (88%). COD reduction was found to increase from the first to the fourth cycle for both periods, while a 92% reduction of COD was indicated for the fifth cycle of the second period.

Similarly, BOD reduction ranged between 57% and 74% for the first period, while for the second period, BOD reduction was higher, ranging between 65% and 88% (Fig. 6). BOD reduction indicated an increasing trend from the first to the fourth cycle in both periods, while a remarkable reduction of 96% was indicated for the fifth cycle of the second period (Fig. 6).

Concentration of total phenols for the first period of experiments ranged between 160 ppm (fourth cycle drainage) and 1,080 ppm (Raw OOMWW) (Table 3), thus indicating a reduction of up to 85% (Fig. 7). Similarly, for the second period of experiments, the concentration of total phenols varied from 72.9 mg/L (fifth cycle drainage) and 663 mg/L (raw OOMWW) (Table 4), with the corresponding reduction being up to 89% (Fig. 7). Total phenols reduction was found to increase from the first to the fourth cycle for both periods.

As far as total nitrogen is concerned and as presented in Tables 3 and 4, depletion of 68% was noted at the end of the third cycle of the first period, while the depletion was higher (86%) at the end of the third cycle of the second period. There was also a significant downward trend in nitrate concentration in both periods, reaching an 81% decrease at the end of the third cycle in the first period and 47% in the second period. Noteworthy was the fact that the concentration of ammonia was almost eliminated in the second period, while a significant decrease was observed in sulfate ions and in phosphate.

Table 3
Chemical parameters of OOMWW and drainages of the first period

First period	Raw OOMWW	First cycle drainage	Second cycle drainage	Third cycle drainage	Fourth cycle drainage
pH	4.7	5.8	6.1	6.8	7.1
EC (mS/cm)	8.9	6.1	5.1	4.7	4.2
COD (g/L)	40	21	14	12	9.4
BOD_5 (g/L)	18	7.7	6.3	6.1	4.6
Total phenols (mg/L)	1,080	390	280	200	160
TKN (mg/L)	373	102	110	143	118
NO_3^- (mg/L)	1,360	701	593	510	458
$N–NO_3^-$ (mg/L)	307	158	134	115	103
NH_4^+ (mg/L)	26	8	13	10	19
SO_4^{2-} (mg/L)	165	65	33	29	58
P (mg/L)	106	77	33	25	22
B (mg/L)	21.2	15.7	12.7	12.7	9.2
K (mg/L)	1,900	1,180	1,140	1,160	1,100
Na (mg/L)	1,040	730	750	760	750
Ca (mg/L)	225	720	820	780	670
Mg (mg/L)	84	235	268	253	230
Fe (mg/L)	55	7.8	12	8.9	9.7
Mn (mg/L)	5	10.3	12.6	9.3	5.3
Cu (mg/L)	35	67	46	41	39
Zn (mg/L)	1.23	0.743	0.729	0.236	0.150

Table 4
Chemical parameters of OOMWW and drainages of the second period

Second period	Raw OOMWW	First cycle drainage	Second cycle drainage	Third cycle drainage	Fourth cycle drainage	Fifth cycle drainage
pH	4.9	6	6.9	7.3	7.6	7.6
EC (mS/cm)	5.8	3.4	2.6	2.4	2.2	2.2
COD (g/L)	20	8	5.3	4.6	2.4	1.6
BOD_5 (g/L)	9.1	3.2	1.6	1.4	1.1	0.35
Total phenols (mg of gallic acid/L)	633	211	152	113	87.7	72.9
TKN (mg/L)	213	207	41.7	24.5	39.9	31.22
NO_3^- (mg/L)	280	181	183	173	148	132
$N–NO_3^-$ (mg/L)	63.2	40.8	43.6	39.1	33.6	30.0
NH_4^+ (mg/L)	32.7	0.38	0.25	0.28	0.11	0.15
SO_4^{2-} (mg/L)	161	404	204	44.9	16.6	19.9
P (mg/L)	28.8	20.7	9.17	6.95	6.05	8.15
B (mg/L)	4.14	2.96	2.77	2.62	2.63	2.78
K (mg/L)	560	660	690	640	690	660
Na (mg/L)	216	330	345	313	350	345
Ca (mg/L)	105	176	154	145	142	150
Mg (mg/L)	34	74	76	68	70	71
Fe (mg/L)	31	3.3	4.4	6.2	3	0.83
Mn (mg/L)	500	1,780	1,766	979	724	587
Cu (mg/L)	2.0	18.0	4.00	5.00	1.30	1.00
Zn (mg/L)	0.09	0.22	0.02	0.1	0.02	0.02

Table 5
Solids of OMWW and drainages of the first and the second period

Parameter	Period	Raw OOMW	First cycle drainage	Second cycle drainage	Third cycle drainage	Fourth cycle drainage	Fifth cycle drainage
TS (g/L)	First	22.6	14.7	12.9	12.2	10.7	–
	Second	8.65	6.33	5.29	5.09	4.34	3.86
VS (g/L)	First	14.4	8.52	6.66	6.16	4.84	–
	Second	7.13	4.93	3.89	3.79	2.92	2.58
TSS (g/L)	First	9.30	4.36	1.82	1.14	1.04	–
	Second	6.32	2.32	1.61	1.72	1.28	0.70
VSS (g/L)	First	8.86	4.04	1.88	1.28	0.66	–
	Second	10.7	7.18	5.60	5.42	5.71	5.11
BOD/COD	First	0.47	0.37	0.44	0.49	0.49	–
	Second	0.46	0.40	0.30	0.30	0.46	0.22
TS/COD	First	0.61	0.7	0.9	0.99	1.14	–
	Second	0.43	0.79	1	1.1	1.81	2.41
VS/COD	First	0.39	0.41	0.46	0.5	0.51	–
	Second	0.36	0.62	0.73	0.82	1.22	1.61
VS/TS	First	0.64	0.58	0.52	0.5	0.45	–

Regarding potassium concentration, a relatively small decrease of 40% was indicated at the end of the first cycle in the first period and then remained almost stable, while similar behavior occurred in the second period, after a small initial increase (20%). Likewise, sodium concentration followed a similar trend, since it remained stable after an initial decrease (28%) in the first period and an initial increase (56%) in the second period. The concentration of calcium in the first period ranged between 225 mg/L (raw OOMWW) and 670 mg/L (fourth cycle drainage), noting from the very first cycle a remarkable increase of 220%. Similarly, for the second period of experiments, the concentration of calcium varied from 105 mg/L (raw OOMWW) and 150 mg/L (fifth cycle drainage), as an initial increase of 68% took place. Calcium concentration build-up was observed for both periods. Similarly, magnesium concentration ranged between 84 mg/L (raw OOMWW) and 230 mg/L (fourth cycle drainage) in the first period, as an increase of 180% from the first cycle was observed, while in the second period, magnesium concentration raise was slightly lower, ranging between 34 and 71 mg/L (118% increase at the end of the first cycle).

Concerning total solids, volatile solids, total suspended solids, and volatile suspended solids a great decrease has been observed in both periods (Table 5). Solids reduction indicated an increasing trend from the first to the last cycle in both periods, while a great reduction in TSS of 89% was indicated at the end of the fifth cycle of the second period.

Regarding the chemical analysis of the fifth drainage of the first period (Table 6) corresponding to the drainage after a wash-out cycle with 100 dm³ of tap water, an increase in pH was observed comparatively to the fourth drainage value (Table 3), while the great decrease of all the other parameters took place.

Meanwhile, results from chemical analysis of the placenta created on the upper surface layer of the biomixture during the second period of biobed operation are presented in Table 7, pointing out that the placenta consists of a proper concentration of organic matter and nutrients, taking into account the other chemical parameters, thus, it could be treated with other plant materials as a high-value material for composting or soil conditioner.

The results of the chemical analysis of surface biomixture samples for each period are presented in Tables 8 and 9.

Chemical determinations in the biomixture samples of the two periods are presented in Tables 8 and 9.

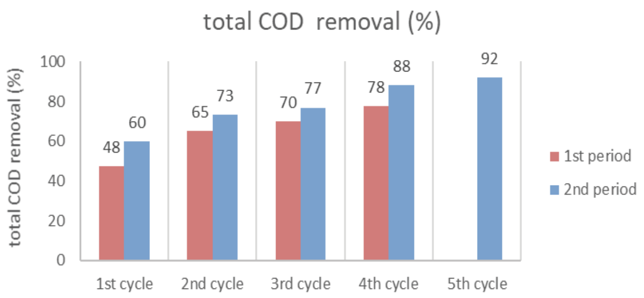

Fig. 5. Comparison of total COD removal (%) of the first and the second period.

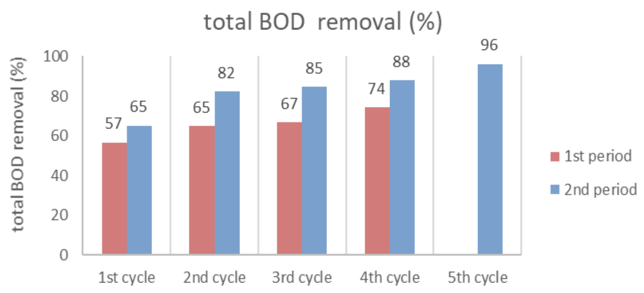

Fig. 6. Comparison of total BOD_5 removal (%) of the first and the second period.

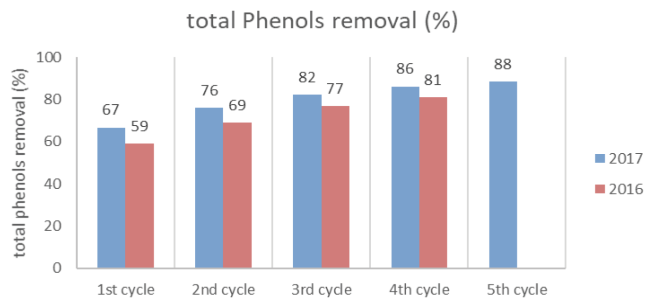

Fig. 7. Comparison of total phenols removal (%) of the first and the second period.

Table 7
Chemical parameters of placenta of the second period

Placenta (upon completion of the first cycle)			
pH	6.1	Na (mg/kg)	445
EC (mS/cm)	1.13	K (mg/kg)	2,700
O.C. (%)	12.4	Ca (mg/kg)	241
O.M. (%)	24.8	Mg (mg/kg)	483
$CaCO_3$ (%)	0.84	B (mg/kg)	4.73
Total Phenols (mg of gallic acid/L)	138	Fe (mg/kg)	483
N_{total} g/kg dry sample	8.65	Mn (mg/kg)	109
NO_3–N (mg/kg)	0.05	Zn (mg/kg)	74.7
P_{total} (mg/kg)	352	Cu (mg/kg)	5.60
C/N = 14.3			

Table 6
Chemical parameters of fifth wash-out cycle drainage of the first period

pH	7.5	SO_4^{2-} (mg/L)	1.1	Cu (mg/L)	23
EC (mS/cm)	2.4	Na (mg/L)	425	TS (mg/L)	4,780
COD (g/L)	2.5	K (mg/L)	630	VS (mg/L)	1,880
BOD_5 (g/L)	0.7	Ca (mg/L)	290	TSS (mg/L)	800
Total phenols (mg of gallic acid/L)	100	Mg (mg/L)	95	VSS (mg/L)	800
TKN (mg/L)	81	B (mg/L)	4.6	BOD/COD	0.28
NO_3^- (mg/L)	222	Fe (mg/L)	3	TS/COD	1.91
N–NO_3^- (mg/L)	50	Mn (mg/L)	0.9	VS/COD	0.75
NH_4^+ (mg/L)	10	Zn (mg/L)	0.04	VS/TS	0.39

Table 8
Chemical parameters of biomixture samples of the first period

Biomixture samples 2016	pH	EC (mS/cm)	O.M. (%)	$CaCO_3$ (%)	Na (mg/kg)	K (mg/kg)	Ca (mg/kg)	Mg (mg/kg)	N_{total} (g/kg) dry sample	NO_3–N (mg/kg)	P_{Olsen} (mg/kg)	B (mg/kg)	Total phenols (mg/kg)	Fe (mg/kg)	Mn (mg/kg)	Zn (mg/kg)	Cu (mg/kg)
Biomixture raw	7.7	0.7	22	1.3	186	1,349	3,852	742	5.4	9.8	282	2.51	105	132	56.9	39.3	4.36
Biomixture of first cycle	5.6	3.7	24	0.5	651	1,849	2,314	350	6.6	1.1	178	5.87	160	208	153	75.6	5.24
Biomixture of second cycle	5.6	3.3	25	0.3	1,150	3,050	1,590	442	6.1	0.2	149	4.77	185	219	158	112	5.60
Biomixture of third cycle	5.8	3.4	21	0.4	1,051	3,100	2,200	480	5.5	0.4	176	5.16	197	254	159	76.1	5.59
Biomixture of fourth cycle	6.5	2.9	21	0.8	899	2,550	2,600	712	4.4	0.4	205	5.82	210	223	159	65.1	6.57
Biomixture of fifth cycle 0 cm	6.5	1.9	18	0.6	750	2,648	2,846	586	4.3	1	200	4.86	190	235	160	64.2	5.50
Biomixture of fifth cycle 15 cm	7.0	2	20	0.7	750	2,600	3,400	564	3	1.6	265	5.40	195	215	173	51.5	6.37
Biomixture of fifth cycle 30 cm	7.2	2	20	1.3	651	2,340	3,384	582	3	1.1	246	5.35	200	182	161	51.5	5.56
Biomixture of fifth cycle 45 cm	7.1	1.9	21	1	699	2,200	3,190	580	3	0.5	251	5.81	206	175	162	54.9	5.64

Table 9
Chemical parameters of surface biomixture samples of the second period

Biomixture samples 2017	pH	EC (mS/cm)	O.M. (%)	$CaCO_3$ (%)	Na (mg/kg)	K (mg/kg)	Ca (mg/kg)	Mg (mg/kg)	N_{total} (g/kg) dry sample	NO_3–N (mg/kg)	P_{Olsen} (mg/kg)	B (mg/kg)	Total phenols (mg/kg)	Fe (mg/kg)	Mn (mg/kg)	Zn (mg/kg)	Cu (mg/kg)
Last year biomixture	7.1	2.0	20	1.0	700	2,447	3,325	578	3.0	1.1	241	5.52	151	191	166	52.6	5.85
Biomixture of first cycle	7.1	0.6	15	0.3	285	2,250	2,078	359	5.9	0.05	224	2.52	99	302	129	71.6	4.92
Biomixture of second cycle	7.2	1.0	15	0.4	490	2,600	2,965	603	6.6	1.3	209	3.75	144	275	97.1	77.8	6.27
Biomixture of third cycle	7.6	1.3	17	0.4	600	3,000	3,430	685	7.6	3.5	252	3.12	110	242	58.1	59.9	4.91
Biomixture of fourth cycle	8.0	1.1	18	0.4	540	2,800	3,389	708	7.8	1.8	275	3.43	118	187	92.1	52.9	4.80
Biomixture of fifth cycle	7.9	1.4	17	1.2	520	2,550	3,015	561	7.9	2.0	291	3.82	120	19.8	2.77	33.1	1.37

Regarding the biomixture samples from the biobed after each period of operation, a slight decrease in pH values was observed in the first period (Table 8), but pH remained stable at the beginning of the second period and slightly increased after the end of the third cycle (Table 9). It is worth noting that the pH value was 6.5 at the end of the first period, while at the end of the second period was 7.9.

Concerning the organic matter content of the samples, values ranged between 20% and 25% in the first period and between 15% and 20% in the second period.

Total phenols content increased during the first period from 105 to 210 ppm at the end of the fourth cycle and decreased in the second period from 151 to 110 ppm at the end of the third cycle.

As far as boron concentration, an increase was observed in the first period from 2.51 to 5.87 ppm at the end of the first cycle and then it remained almost stable, though in the second period a decrease took place from 5.52 to 2.52 ppm at the end of the first cycle and then remained stable at 3–4 ppm.

The percentage of $CaCO_3$ samples decreased slightly in both periods.

Potassium content of the samples increased, while sodium, calcium, and magnesium content declined slightly but their concentrations still remained high.

Concerning the N_{total} of the biomixture samples, a decline in the first period was observed, while an increase occurred in the second period, noticing that N_{total} concentration was 4.4 g/kg at the end of the first period and 7.9 g/kg at the end of the second period.

Phosphorus content of the samples remained relatively stable. Iron and zinc content rose slightly in both periods, while copper remained almost constant. Similarly, manganese concentration increased slightly after the end of the first cycle and then remained stable in the first period, while it decreased slightly in the second period.

Regarding the chemical determinations in the layers of the biomixture (samples collected from four different depths) at the end of the first period, no load is observed in any layer, leading to the conclusion that the biomixture can be used safely for the third period.

5. Discussion

The biobed system presented here proved to be very efficient for the treatment, purification, and detoxification of olive oil wastewaters, since a significant decline of BOD_5, COD, and total phenols was observed from the second operating cycle of the biobed operation. Moreover, there was a significant improvement in pH from acidic to neutral (first period) and alkaline (second period), while electrical conductivity declined markedly.

More specifically, total COD removal was 78% in the first period and 88% in the second period at the end of the fourth cycle, whereas it reached 92% removal at the end of the fifth cycle in the second period, while total phenols reduction reached 86% at the end of the fourth cycle and 88% at the end of the fourth cycle in the second period.

Spectacular drop occurred in the concentration of NH_4^+, since their absence was observed even from the first cycle of the second period. NH_4^+ accumulation was not observed in both periods, in contrast to the results from the majority of studies dealing with effluents with high organic-N [28].

Concerning BOD_5, great removal was observed in the second period, since higher rates of decrease have been observed after placenta substruction: 82% decrease after the end of the second cycle, 85% decrease after the end of the third cycle, 88% decrease after the end of the fourth cycle and 96% decrease after the end of the fifth cycle. Besides that, BOD_5 values of both periods meet the restrictions of the legislation concerning the treatment of olive oil mill wastes [29,30].

The BOD/COD ratio ranges between 0.3 and 0.4, indicating that the liquid waste is biodegradable in each cycle of the experiment [31,32]. The VS/COD ratio is also an indicator of the anaerobic biodegradability of the drainages. The values of these ratios increase gradually, starting from the value of 0.434, which indicates that drainages are becoming easier to biodegrade in each cycle, as less oxygen is required for the decomposition of the organic substance and since values greater than 0.4 show high biodegradability of the organic matter [33]. In addition, the VS/TS ratio is an indicator of the organic matter in the waste [34]. It is observed that the ratio decreases in each cycle, which indicates the decrease of the organic matter in the drainage of each cycle.

In the light of the above considerations, the final drainages (even from the third cycle) could be used as they are or diluted [29,30] either in irrigation or in watering or fertilizer–water–irrigation. Additionally, they could be discharged into natural recipients after proper dilution. Likewise, at the end of both the first and second period, biomixture's content in nutrients and high organic matter (20% and 17%, respectively), as well as its other chemical characteristics (pH, EC, etc.), nominates that the used biomixture may be reused satisfactorily for crop fertilization and for soil enrichment with organic matter, as many other organic substances like sewage sludge [35,36], dewatered sewage sludge vermicomposting [37], or sludge-based biosolids [38] or disposed under certain conditions.

The subtraction of all the placenta created on the upper surface layer of the biomixture (which content was only 6.5 kg/m^3), before the second period of biobed operation, upon completion of the first cycle, prevented placenta re-creation in subsequent cycles and resulted in better biobed function, higher application speeds, and fluid supplies, as well as higher rates of detoxification of the waste, due to the increase of the permeability of the surface of the biomixture. Moreover, the nutrient and polyphenol content of the placenta as well as its pH, EC, and C/N values, as well as its high content in organic matter (25%), indicate that this organic material can be reused as a soil amendment in soils with low organic matter content as it is or after composting.

Mourga made up about 15% of the waste, settled in the tank, and due to its dense composition could not be filtered. Thus, it was removed in both periods, analyzed, and used for another experimental projects, concerning composting and feed additives.

High organic loading rates applied were not found to be a limiting factor for the biobed's efficiency, since available O_2 was enough in order to maintain aerobic conditions and consequently satisfactory performance of the biobed, while maintenance of aerobic conditions in waste treatment in other studies has been problematic [39]. From the nitrogen balance sheets of the drainages and the biomixture of each cycle, it is indicated that nitrogen compounds are used by biomass microorganisms as nutrients.

The biomixture constitutes an important component of biobed systems and its correct composition is a prerequisite for successful detoxification of wastewaters. This study shows that low-cost composted materials could successfully behave as a filter in olive oil wastewater treatment. The "rejuvenation" of the biomixture that took place in the second period proved to be easy, quick, and efficient, which proves that biomixture can be reused effectively for more years. After all, its reuse follows the principles of the circular economy, given that its disposal would be an additional problem that the olive oil mill owner would have to deal with.

The chemical analysis of the drainages and biomixture samples of the fifth wash-out cycle in the first period, showed that purification of the biomixture takes place, since pH value increased, as well as all other parameters decreased. Specifically, pH increased from 7.1 to 7.5, values within acceptable limits for irrigation water (6.5–8.0), while EC decreased from 4.2 to 2.4. The water of this quality is used in irrigation with small to moderate restrictions [40]. In case of high salinity, dilution with tap water is recommended. These outcomes indicate the fact that a possible rainfall may satisfactorily purify the biomixture.

6. Conclusions

This study points out that biobeds show high potential for the reduction of COD, BOD_5, total phenols, TKN, NH_4^+, SO_4^{2-}, TS, VS, TSS, and VSS, provided that removal of all the placenta created on the upper surface layer of the biomixture takes place, upon completion of the first cycle. Pollutants concentrations are decreased, approaching 92% for COD, 96% for BOD_5, 88% for total phenols, 85% for TKN, 100% for NH_4^+, 88% for SO_4^{2-}, 55% for TS, 64% for VS, 89% for TSS, and 52% for VSS. Furthermore, biobed's efficiency is not limited by the high organic loading rates, since aerobic conditions are developed within the system built, and therefore adequate aeration is maintained. Biobeds can be considered as a novel, simple, expeditious, inexpensive, and effective method for treatment, purification, and detoxification of olive oil wastewaters, in order to minimize environmental impacts (contamination) from their uncontrolled discharge or inadequate treatment. The promising outcomes combined with the fact that the method is zero-waste and follows the circular economy principles, may facilitate the arduous and intractable treatment of OMWW in Greece. The use of dilution of OMWW can also improve the system performance. The reused biomixture, when replaced, and the placenta removed, can be used as a soil amendment in soils with low organic matter content, either as it is or after vermicomposting or composting.

Finally, as the results of the experimental process prove, the reuse of the useful wastes of the olive oil mills and biobed by-products (mourga, placenta, and used biomixture) as fertilizer and soil amendment offers a sustainable solution to chronic problems of the olive oil production and the olive oil mill owners, in an economic viable and environmentally friendly process. According to the principles of the circular economy, with this zero-waste method, a by-product of oil production, which until now has been considered to be a hazardous waste, is transformed into valuable fertilizer. Throughout the economic circle, what has been until now called "waste", can be turned into valuable components for the rest of the production process. In addition, it supports the competitiveness of a valuable export product, improving its quality, but also the income of olive oil mill owners, especially small and medium enterprises that cannot afford other waste treatment methods. For small island areas and touristic areas, it is also a sustainable method from a spatial point of view, since it requires a very small area for OMWW treatment.

Further future research may be conducted on the treatment and utilization of the by-products of the biobed (mourga, placenta, and used biomixture), by vermicomposting to produce soil amendment, so that it can be used in crop fertilization.

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The authors have no conflicts of interest to declare that are relevant to the content of this article.

Financial interests

Any undeclared financial interest that could embarrass the authors were it to become publicly known after the work was published.

Data transparency

All data and materials support their published claims and comply with field standards.

Code availability

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