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Innovative solutions for reduction of olive mill wastewater pollution

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ABSTRACT

Olive oil is also an important activity for the inhabitants of Al Jouf, KSA. The production of olive oil is accompanied by the emission of wastes (liquid and solid). These wastes are discharged directly without treatment. This research work aims to study the treatment of such waste by the integration of physical and chemical methods. The organic loads expressed by chemical oxygen demand (COD) and biological oxygen demand (BOD) of the produced wastewater (olive mill wastewater) were evaluated. The quantity of wastes emitted from the three phase centrifuge decanters was found to be higher than that emitted from the two-phase centrifuge decanters. The olive mill wastewater (OMWW) was treated using plane sedimentation (PS) combined with advanced oxidation treatment. The effluent from AO was splitted and treated using complex sand filtration unit (CSF) and modified trickling filter (MTF) in parallel manner. The results indicated that the concentration of COD, BOD, TSS, oil & grease and phenol was reduced greatly by more than 67%, 75%, 20%, 97% and 91% and 65%, 77 3%, 96% and 86%, respectively for the effluent of the three phase OMWW. This work supports the application of such treatment technique for reducing the environmental threats of olive mill waste effluents.

Keywords: Olive mill wastewater treatment; Advanced oxidation; Modified trickling filter; Electric hoses pipes

1. Introduction

Olive oil extraction includes different activities (e.g, leaf removal, olive washing, grinding, beating as well as oil separation). The characteristics of the obtained oil and associated wastes depend greatly on the methods used for production. Olive oil is produced mechanically from the fresh fruit in order to conserve its natural characteristics according to the Global Standards [1]. Olive fruits must be processed quickly after harvesting to eliminate oxidation (as much as possible) and preserve low acidity [2]. Crushing of olives followed by malaxation of the obtained paste and separation of oil by centrifugation are the steps used during oil production. The two commonly used techniques to separate oil are the two phase or the three phase decanter centrifuge [3].

The application of the three phase centrifugation process in the seventies century increased the processing capacity and extraction yield and reduces labor [4,5]. This technology uses water as an essential component during the production of olive oil. The products of the three phase process are oil phase, solid residue and the green liquor olive mill wastewater (OMWW). The decanter is used to separate solid residue from the other two phases. The drawback of this process is the high consumption of water, about 1.25 to 1.75 times of the weight of olive used [5].

The failure to invent a suitable olive OMWW treatment technology derives technology manufacturers to develop the two phase process. It uses only washing water and delivers oil and the wet substrate (pomace) using a more effective centrifugation technology [6]. The lower water demand in this

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process has reduced environmental impact. However, olive mill waste (OMW) threats the environment, as leaves represent only 5% of olives' weight in oil extraction. The annual world olive mill waste production is expected to be in the range 10–30 million m^3 [3].

Threatening of natural water resources due to olive mill waste is a major problem. It affects the soil fertility, harm the plants and soil micro flora when disposed without treatment [7–9]. Consequently, direct discharge of olive mill wastewater into the environment is not allowed and certain constrains must be taken prior disposal of the olive mill waste into the environment [7,8,10].

Olive mill wastewater characterized by the high levels of both organic loads and phenolic compounds [7,8]. Jaafari et al. [9] studied the performance of anaerobic fluidized bed reactor (AFBR) for the treatment of high-strength wastewater. Application of biologica1 treatment of OMWW will not be efficient due to the presence of phenolic compounds. Consequently, the use of chemical or physical treatment coupled with biological treatment will be effectively remove high organic load as well as degrading the phenolic compounds in OMWW [7,8].

Carlos et al. [11] presented the application of Fenton's reagent process combined with anaerobic digestion for the treatment of olive mill wastewater (OMWW). The olive mill wastewater (OMWW) was primary treated by chemical oxidation in a batch reactor with Fenton's reagent, using a fixed H_2O_2/COD ratio of 0.20, pH = 3.5 and a H_2O_2/Fe^{2+} molar ratio of 15:1. The reductions of 17.6 chemical oxygen demand (COD) and 82.5% of total polyphenols (TP) were achieved. Then the olive mill wastewater (OMWW) secondary treated by anaerobic digestion using previously adapted microorganisms immobilized in Sepiolite. This combined process presented a significant improvement on organic load removal, reaching COD degradations from 64% to 88%.

The quantity and the physico-chemical characteristics of the produced wastes depend on the used oil extraction technique, the processed fruits, as well as the operating conditions. Several treatment techniques were used for the treatment of such waster. The obstacle which hindered the simple treatment of OMWW is the presence of toxic compounds such as phenolics. OMWW treatment have been carried out using several technologies such as aerobic and anaerobic treatment [7,8,11] as well as physico-chemical treatment [12]. Recently, Fenton oxidation processes have been applied for the destruction of resistant organic compounds. Using of Fenton and electro-Fenton oxidation to remove inhibitory compounds and increase the biodegradability of OMWW could offer a good solution at a reasonable cost [10,13,14]. The main objective of this work is to reduce the impact of olive mill wastewater that discharged without treatment and reduce the threats to the environment. This could be achieved by combining chemical and simple biological techniques for the treatment of such waste. The aims extended to study the performance of the combination between chemical, physical and biological treatment of the two phase and three phase centrifugation technique that used for the production of olive oil in Sakaka City, KSA.

2. Materials and methods

Field trips were carried out for collecting mill wastewater to carry out the physico-chemical characteristics. The analysis will include: pH, organic loads (biological oxygen demand (BOD), chemical oxygen demand (COD)), nutrients (total phosphorus (TP) and nitrogenous compounds expressed as total Kjeldahl nitrogen (TKN)). Also, phenols, total suspended solids (TSS) and oil contents (oil and grease). The characteristics of olive mill waste wastewater were done according to APHA, [15]. COD and BOD were measured in triplicates while, TSS, TKN, TP, oil and grease and phenolic compounds were measured in duplicates.

To determine phenolic compounds, olive mill wastewater was extracted by ethyl acetate and evaporated under vacuum at 40°C to remove organic solvent. The method described by Weidner et al., [16] and Troszynska and Ciskae [17] were used to determined the phenolic acids. HPLC, Agilent 1100 series supplemented with diode array detector (DAD) at wavelength 250 nm was used during this study.

Reversed phase C18 column (4.6 × 150 × 5 mm) was used. The composition of the mobile phase was 68% de-ionized water, 30% methanol and 2% acetic acid (25%) at 1.0 ml min^{-1} flow rate.

2.1. Treatability study

Olive mill wastewater was collected weekly from Gameia and Namaa as example for two phase and three phase decanters for three months. The samples were collected from the manhole from the storage tanks from both companies. The samples were fresh and collected during the production of the olive oil. Fig. 1 shows the schematic diagram illustrating the treatment steps for olive mill wastewater.

The primary treatment step is the plane sedimentation (PS). The detention time of PS is chosen to be 1 d. Fig. 2 shows the dimensions of PS tank.

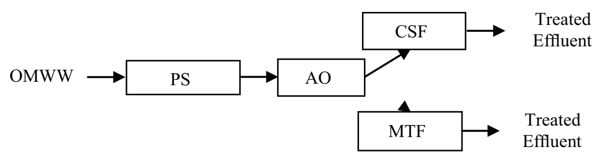

Fig. 1. Schematic diagram of the treatment steps.

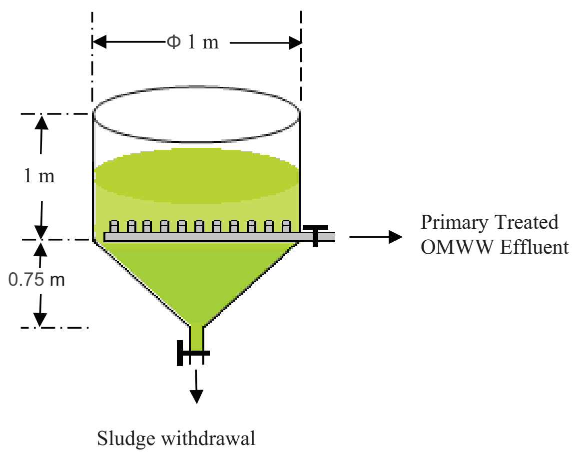

Fig. 2. Plane sedimentation process (PS) used in the study (slope of the base is 45°).

The proposed secondary treatment step is the advanced oxidation process (AO). The optimum operating conditions for Fenton's reaction (pH, H_2O_2/COD, Fe/H_2O_2 and reaction time) as well as the experimental work were chosen according to the results obtained by El-Gohary et al. [7].

The effluent from the AO was feed directly to both the complex sand filter unit (CSF) and modified trickling filter unit (MTF). The dimension of CSF is shown in Fig. 3. The CSF divided into three main sections. The upper section is fine gravel (4–8 mm), the middle section is pea gravel (2–4 mm) and the lower section is coarse sand (1–2 mm). The flow rate of the complex sand filtration unit is 2 l min^{-1}, which corresponds to surface loading rate to 0.611 $m^3/m^2/h$.

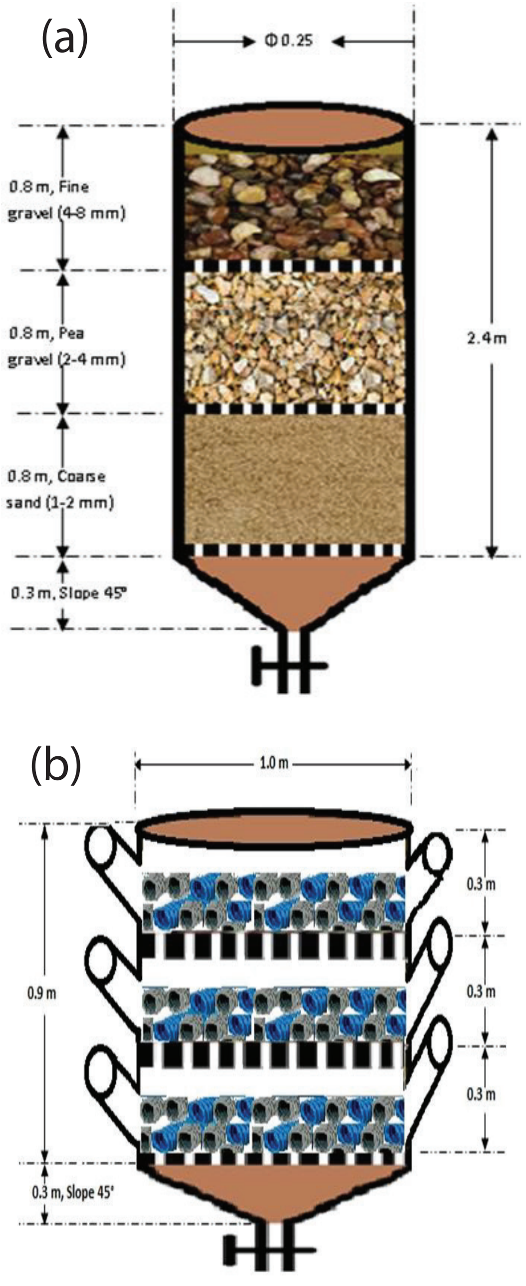

Fig. 3. Dimensions of the post treatment units. (a) complex sand filtration unit (CSF), and (b) modified trickling filter unit (MTF).

The MTF unit is divided into three sections separated by perforated baffle. The unit packed with small pipes of electric hoses. These pipes were cut into 4 cm parts. The unit provided with opening arms to enhance the passive aeration. The ratio of air to packing materials inside the unit is about 75% to 25%, respectively. The MTF is a modification of another system provided by El-Khateeb et al., [18]. The air to packing material ratio as well as the aeration arms is chosen to enhance the passive aeration inside the unit. The material used for manufacturing of PS, CSF and MTF units is made of polyvinyl chloride (PVC) pipes.

The slope of the base for the two systems was fixed at 45° to enhance the settleability and withdrawing of sludge in PS unit and to collect the treated effluent in CSF and MTF units. All the statistical analyzes were carried out by using descriptive test method.

Samples were collected from Al Jouf. Al Jouf produces about 67% of the olive oil in the Kingdom. The produced oil is highly competitive because of the appropriate conditions of the region.

3. Results and discussion

3.1. Characteristics of the generated wastes

Production of olive oil always accompanied with the generation of olive mill solid waste as well as olive mill wastewater. Three phase and two phase centrifuge decanters techniques are used for the production of olive oil in Al Jouf region. Olive mill wastewater is characterized by, black brownish colour, extremely high content of organic compounds with high values of chemical oxygen demand (COD), high level of phenolic compounds (most of which are refractory or toxic to micro-organisms), BOD/COD ratio which shows the biodegradability of the organic contents of olive mill wastewater in most cases ranged from 0.2 to 0.5. The concentration of phenolic compounds was found to be more than 2.5 g l^{-1} [7–9].

3.2. Characteristics of wastes

Table 1 shows the main characteristics of the wastewater effluents from two and three phase olive mills in Al Jouf. Actually, the workers in the two phase olive mills add water to the waste produced during oil production to discharge the waste easily to the sewerage system.

The BOD/COD ratio was found to be very low and ranged from 0.2155 to 0.3002. This low ratio could be attributed to the presence of poor biodegradable organic compounds (phenol) and/or toxic once [7–9,19,20]. The obtained results were found to be in a good agreement with that obtained by Khdair and Ghaida [21].

The production of olive oil produces huge amounts of different wastes that may have a great impact on the environments (water, plant and soil) because of its toxic effect. Phytotoxic wastes (olive mill waste and olive mill wastewater) are considered the most pollutant by-product of this industry. The major part (more than 95%) of the highly toxic phenolic compounds was discharged with the waste of olive mill [22,23]. Pavlidou et al. [23] investigated the impacts of olive mill wastewater that discharged to top soil, surface

Table 1
Characteristics of olive mill wastewater[a]

Parameter	Three phase olive mill			Two phase olive mill		
	Gameia	Al Shamal	Al Jadid	Namaa	Al Hamad	Al Shallan
pH	3.6–4.3	3.7–4.5	3.6–4.4	3.5–4.3	3.8–4.6	3.7–4.5
COD	72,150	64,950	55,350	1,50,562	1,28,550	1,35,000
	±15,000	±13,550	±11,365	±27,490	±22,300	±23,550
BOD	21,660	13,998	1,2795	17,955	21,977	1,7482
	±4,650	±2,350	±2,460	±3,010	±4,250	±3,330
BOD/COD	0.3002	0.2155	0.2312	0.1193	0.1710	0.1295
	±0.08	±0.06	±0.06	±0.04	±0.04	±0.06
TKN	13,450	13,254	10,565	15,235	17,898	16,525
	±2,350	±2,470	±1,790	±2,450	±2,650	±2,480
TP	11,233	11,253	12,330	14,523	15,425	14,333
	±2,480	±2,333	±2,600	±2,710	±2,750	±2,365
TSS	10,256	10,365	10,222	15,325	19,253	16,356
	±2,150	±2,010	±2,200	±3,000	±3,250	±2,950
Oil & Grease	36,430	22,153	23,500	19,658	25,000	23,650
	±1,036	±6,800	±7,310	±5,470	±7,910	±7,450
Phenols	1,470	1,235	1,950	2,153	1,560	2,152
	±250	±125	±235	±310	±255	±310

[a]Average of 13 samples (standard deviation between brackets), Concentration in mg/l.

water and marine environments. The researcher directly correlated the enrichment of surface water and the coastal zone in Greece with phenols, total organic carbon (TOC), nitrite, ammonia, copper, nickel and manganese to the discharge of olive mill waste and olive mill wastewater. These wastes were found to possess toxic effect to marine living organisms (Palaemonidae shrimp).

3.3. Reducing the impact of olive mill waste and olive mill wastewater

The use of two phase centrifuge decanter in KSA is of utmost important. This process will reduce the environmental impact because of the lower amount of water uses and consequently, the produced waste [24].

On the contrary, olive mill waste and olive mill wastewater have a valuable source of economic compounds for valorisation and recovery purposes [25,26]. Reusing of olive mill waste and olive mill wastewater can eliminate to great extant, the impact of olive waste. Ayoub et al. [27] recommended the application of olive mill wastewater (in appropriate dilutions) to enhance soil fertility and olive plant yields. The high contents of phenols in OMWW hindered the application of the treatment of such wastewater biologically. Annab et al. [28] explored and proposed a complete cycle of olive mill wastes treatment. Two sorbents prepared from olive pomace, granular (GAC) and powdered (PAC) activated carbons were successfully synthetized and encapsulated in calcium alginate. The obtained sorbents (GAC-CA and PAC-CA) were tested for gallic acid removal. But PAC-Ca were found to be efficient than GAC-CA. Consequently, PAC-CA beads were used for the treatment OMWW to adsorb polyphenols. The outcomes of this study certify proofs of great potential of PAC-CA beads for polyphenols removal.

3.4. Performance of the treatment units

The PS step allows the sedimentation of some of suspended solids as well as separation of residual oil. Composite samples from Namaa and Gameia olive mill as example of two and three phase technologies was treated using PS technique.

Table 2 reflects the performance of improving the quality of olive mill wastewater. The organic loads represented by COD, BOD, TSS and oil & grease were reduced greatly. The level of phenolic compounds was reduced by 86% in three the phase and the two phase mill for PS treatment step. Consequently, the biodegradability of the primary treated effluent was enhanced. The BOD/COD ratio increased from 0.3 and 0.12 to 0.49 and 0.46 the tree and two phase mill, respectively. The effluent of PS step was chemically treated using AO process. The results existing in Table 2 reveal that the performance of AO treatment process is quite satisfactory.

The efficiency of AO was found to be more that the treatment of OMWW by using other technique. Azzam [29] employed simple natural locally available materials as adsorbents to decrease the levels COD and phenolic compounds in OMWW. Specifically, volcanic tuff (VT), natural clay and charcoal were investigated as possible adsorbents of OMWW's organic matter. Synergistic effects on COD and phenols removal were noticed. Reductions in COD and phenols concentrations reached 14% and 21%, respectively.

Remaining concentrations of COD, BOD, TSS, and oil and grease and phenols are 2,930, 1,910, 35, 20 and 4 mg l^{-1} for three phased olive mill and 3,010, 2,050, 32, 17 and 3.5 mg l^{-1} for two phase olive mill, respectively. El-Gohary et al. [7] diluted olive mill wastewater to 1:1 ratio with tap

Table 2
Performance of the combined PS/AO/CFS treatment system for the improving the quality of olive mill wastewater[a]

Parameter	Three phase olive mill									Two phase olive mill								
	Gameia OMWW	PS Effl.	%*R*	AO	%*R*	CSF	%*R*	MTF	%*R*	Namaa OMWW	PS Effl.	%*R*	AO	%*R*	CSF	%*R*	MTF	%*R*
pH	3.6–4.3	3.5–4.3		7.8–8		7.7–8.1		7.7–8.2		3.5–4.3	3.4–4.3		7.9–8.2		7.8–8.1		7.7–8.1	
COD	72150 ±15000	25230 ±2500	65	2930 ±350	88	980 ±159	67	1020 ±165	65	150562 ±27490	33450 ±4510	78	3010 ±685	91	1020 ±450	66	1050 ±171	65
BOD	21660 ±4650	12350 ±1480	43	1910 ±210	85	421 ±68	78	430 ±66	77	17955 ±3010	15390 ±2500	14	2050 ±318	87	590 ±120	71	570 ±68	72
BOD/COD	0.3 ±0.08	0.49 ±0.05		0.65 ±0.1		0.43 ±0.08		0.42 ±0.1		0.12 ±0.04	0.46 ±0.06		0.68 ±0.1		0.58 ±0.08	15		
TKN	13450 ±2350	2450 ±540	82	89 ±25	96	39 ±9	56	50 ±12	44	15235 ±2450	3330 ±680	78	102 ±20	97	52 ±8	49	60 ±13	41
TP	11233 ±2480	1840 ±350	84	40 ±6	98	21 ±5	48	28 ±8	30	14523 ±2,710	2650 ±410	82	44 ±7	98	25 ±5	43	28 ±9	36
TSS	10256 ±2,150	2,375 ±350	77	35 ±8	99	58 ±11	20	42 ±9	3	15325 ±3000	3,330 ±650	78	32 ±6	99	65 ±7	6	55 ±12	6
Oil & Grease	36430 ±10,360	570 ±105	98	20 ±2.5	96	0.65 ±0.1	97	0.9 ±0.2	96	19658 ±5470	350 ±95	98	17 ±3	95	0.75 ±0.07	96	0.85 ±0.2	95
Phenols	1470 ±250	210 ±52	86	4 ±1	98	0.35 ±0.07	91	0.58 ±0.5	86	2153 ±310	290 ±66	87	3.5 ±0.8	99	0.29 ±0.05	92	0.45 ±0.1	87

[a]Average of 13 samples (standard deviation between brackets), Concentration in mg l^{-1}.

water to decrease the COD level to be suitable for the AO process. In this study, the using of sedimentation process was chosen to decrease the COD concentration as well as to decrease the volume of olive mill wastewater and as a result to reduce the consumed chemicals. In an attempt to enhance the quality of the olive mill wastewater the effluent from the AO was feed to CSF as well as MTF units. It could be noted that the concentration of COD, BOD and TSS was reduced greatly using both systems. The obtained results were found to be in a good correlation with that obtained by Achak et al. [30]. In that study a diluted olive mill wastewater with domestic wastewater (1:1 ratio) was treated using sand filter. The sand filter treatment also led to important reductions in organic matter (90% of total COD, 83% of dissolved COD and 92% of phenolic compounds) and nutrients (91% of TKN and 99% of phosphates). In the contrary Zorpas and Costa [31] found that COD was removed by only 70%.

Wastewater enters the MTF, trickles downward over the biofilm developed on the surface of the packing materials (electric hoses pipes) and air moves upward or downward. The rough surface of the packing material supports the attachment of biofilm. Wastewater treatment using the MTF results in a net reduction of organic loads (COD and BOD). On the other hand, liquid-solids separation is required and therefore the bottom of the reactor has settling zone. The high porosity of the MTF was to avoid clogging and promote ventilation [32]. The motion of the wastewater inside the MTF enhancing the sucking air and increase the ventilation and accelerate the aerobic treatment [33,34].

Table 2 summarizes the performance of the treatment units. The CSF shows slight efficient removal of organic loads (COD and BOD) and TSS compared with MTF. This may be attributed to the extensive surface area of sand as well as the physical filtration and entrapment of suspended solids remains in the influent water [35].

4. Conclusions

The obtained results showed that, the combination of sedimentation, advanced oxidation, complex sand filtration and modified trickling filter efficiently treat the olive mill wastewater. By applying such technique a considerable amount of olive mill wastewater could be reduced which may protect the environment due to the contamination of the environment with such waste. The level of COD, BOD, TSS, oil & grease and phenol were reduced from 72,150, 21,660, 10,256, 36,430 and 1,470 mg l^{-1} to 980, 421, 58, 0.65 and 0.35 mg l^{-1} for CSF unit and 1,020, 430, 42, 0.9 and 0.58 mg l^{-1} for MTF unit for treating the three phase OMWW, respectively. When moving to the two phase OMWW effluent the level reduced to 1,020, 590, 65, 0.75 and 0.9 mg l^{-1} for CSF unit and 1,050, 570, 55, 0.85 and 0.45 mg l^{-1} for MTF unit, respectively. Consequently, it is recommended to reuse the treated effluent for the irrigation of trees around the olive mill plants.

Also, research work should be carried out to maximize the benefits of olive mill solid waste and olive mill wastewater residues because it contains materials of great economic value such as anti-oxidants as well as anti-bactericidal compounds.

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