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# Treatment of Olive Mill Effluent Wastewater Using Some Advanced Processes and Reuse of the Treated Wastewater

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**Abstract**

*In this study it was aimed to treat the Olive mill effluent wastewater using different sequential treatment processes namely anaerobic and aerobic biological processes, sonication and photo degradation with Nano SiO2 under sun ligth power. The maximum total COD yields were 60 % and 66 % in the anaerobic and aerobic reactor, respectively, while the total COD yield in the sequential biologic reactor was 86 %.The maximum COD yields in the sonicator was 53 % after 45 min retention time. No removal of COD was observed via photolysis and adsorption. The maximum photo catalytic removal of COD was 40 % at a nano SO2 concentration of 0,5 mg/l after 10-60 min. After RO the COD yield was approximately 100% while to yields of total phenol, TN, TP, dissolved COD and DSS were 96.6%, 99.3%, 99.98%, and 99.97%, respectively. The cost to treat 1 m3 of OMW was 1.033 €. The effluent of RO can be used as irrigation water.*

**Keywords:** Olive mill, Photodegradation, Reverse osmosis, Sonication.

## Introduction

The olive mill wastewater (OMW) has a high relation in the Mediterranean countries. Although olive oil is a useful product its production gave high several adverse effects to the ecosystem, primarily due to its high COD, color and polyphenol content. Approximately 28 million m3 of OMW effluents are produced per year in the Mediterranean countries. OMW is defined by a high suspended solids content (e.g. TSS between 6 and 70 g/L), dark brown color, characteristic unpleasant odor, low pH, high turbidity and high organic load (e.g. COD between 30 and 318 g/L) [1, 2]. In addition, OMW include many complex organic substances (i.e. phenolic compounds (TPh between 0.5 and 24 g/L), polysaccharides, tannins, pectin, organic acids, etc.), which are generally resistant to biodegradation. Also, these permanent organic matters can lead to various negative adverse effects on the environment, such as foul odors, threat to the aquatic life, discoloring of natural waters, eutrophication of surface waters, pollution of superficial groundwater and toxicity (i.e. eco- and phytotoxicity) [2-4].

The most common practice for the management of OMW contains the use of evaporation ponds and the subsequent discharge of solids in landfills and/or on soil. While evaporation ponds offer a good way of reducing the liquid portion of this effluent, they do not contribute to the reduction of its toxicity, while they simultaneously impart an odor problem to the areas where such waste is stored [5]. Moreover, the direct spreading of raw OMW for agricultural aims could be considered as:

1. an economic mean to resolve the OMW pollution-related problems, particularly in replacing chemical fertilization,
2. a low-cost source for water, particularly in the countries of the Mediterranean zone that are facing serious and extended water scarcity events [6].

The uncontrolled discharge of OMW can pose a serious environmental risk, and as a result suitable treatment is required. A number of OMW treatment methods have been utilized until now and these can be divided into four general categories: (i) physicochemical methods (e.g. sedimentation, coagulation/flocculation, etc.), (ii) biological processes (e.g. aerobic activated sludge, anaerobic digestion, etc.), (iii) membrane filtration and separation processes (e.g. microfiltration (MF), UF, NF, reverse osmosis (RO)), and (iv) advanced chemical oxidation processes (e.g. heterogeneous photocatalysis, ozonation, photo-Fenton oxidation, etc.) [7-9]. It is well known that the simple physical processes (i.e. dilution, evaporation, sedimentation, coagulation/flocculation, filtration and centrifugation) are not able, if applied alone, to reduce the organic load and the toxicity of OMW to acceptable limits [7]. On the other hand, conventional biological processes have shown satisfactory efficiencies in terms of OMW purification, especially regarding the biodegradable organic content [10]. More specifically, the aerobic biological treatment systems could become an interesting alternative due to their fast process kinetic and high removal rates [8]. Furthermore, the use of advanced membrane filtration and separation processes, such as MF, UF, NF and RO, have been proposed to get effluent streams from OMW of acceptable quality for safe discharge in the environment, tree or land irrigation, or even for recycling and

reuse in the olive mill facilities [11-14].

The aim of this study was to investigate some sequential treatment processes to determine the optimum operational conditions for the treatment of OMW using sequential anaerobic-aerobic reactors, sonacation, sunligth photocatalysıs and RO in an economical and feasible way. Furthermore the recovery of treated wastewater as irrigation purpose was evaluated.

## Materials and Methods

### OMW Characteristics

The characterisation of OMW samples taken by a three-phase olive mill factory located around İzmir was given in Table 1.

**Table 1. Main characteristics of raw OMW samples (mean values).**

| Parameter | Unit | Concentrations |
|---|---|---|
| pH | | 4.8 |
| EC | (mS/cm) | 14.1 |
| COD | (g/L) | 83.4 |
| BOD5 | (g/L) | 60.5 |
| DOC | (g/L) | 19.6 |
| TSS | (g/L) | 36.3 |
| TP | (g/L) | 0.13 |
| TN | (g/L) | 3.2 |
| T Phenol | (g/L) | 7.9 |
| TSS | (g/L) | 8 |
| Poliphenol | (g/L) | 5.6 |

### Experimental set-up and Procedures

### Sequential Anaerobic and Aerobic Reactors

Dark colored glass reactors sealed with rubber lids having volumes of 1,5 liter was used for the anaerobic treatment 300 ml of granulated anaerobic sludge taken from a yeast factory anaerobic digestor was used as seed and put to the glass reactor (Fig 1a). Then, the anaerobic reactor was filled with 900 ml raw textile wastewater. It was shaken in an shaker at 37°C. The effluent of this reactor was used as feed of the aerobic reactor. The aerobic glass reactor has a volume of 3 liter and it was filled with 800 ml aerobic sludge taken from the aerobic stage of the municipal sewage sludge. Then this reactor was filled with 500 ml anaerobic reactor effluent. The aerobic reactor was stirred with oxygenated with an oygen pump continously with a flow rate of 1,2 l /min. With this pump the oxygen level of the aerobic reactor was 3-4 mg/l.

### Treatment with Sonication

The effluent of the aerobic reactor was given to a sonicator ( Bandelin sonorex) at a frequency of 5 Hz at a power of 18 W/m$^2$. The samples was placed during 15, 30, 45, 60, 75, 90, 105 and 120 min( Fig 1b).

### Treatment with photolysis, adsorption, and photocatalysis

The effluent of the sonication was taken under sun ligth at a power of 8 W/m2 during 4, 6 and 8 hours at hours between 10.30 am-17.30 pm to detect the removals of pollutans via sun ligth. The effluent of the sonicator was taken in dark for 24 hours to detect the removal with adsorption. Then the effluent of sonication was photodegraded using nano-SiO2 under sun ligth. The effects of increasing nano-$SiO_2$ concentrations (1, 3 and 6 mg/L) and photodegradation times (5 min., 10, 15 and 30 min.)on the photocatalytic removal of pollutants were investigated (Figure 1c).

### Treatment with Reverse Osmosis (RO)

RO membrane filter bioreactor sistem consist of 3 pre-stage compartment. In the first stage a sediment filter with a pore size of 5 µm was used to bloke the fine sands. The second compartment is a carbon filter with a pore size of 10 µm and remove florur and organic chemicals. In the 3th stage provides the filtration of wastewater and the pore size of this filter is 1 µm. The RO is fed with a flow rate of 500 ml/day and at a power of 30 bar. The pore size of the RO membrane is 0,11 µm.This stage remove all the microoorganism, salinity, magnesium, calsium and all metals and heavy metals ( Figure 1d).

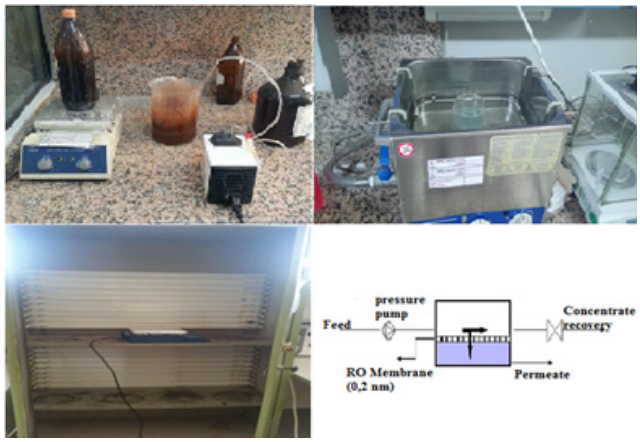


**Figure 1**. a) Anaerobic and aerobic reactors in laboratory, b) sonication reactor system, c) Photocatalitic reactors, d) Reverse Osmosis flow rate

## Operational Conditions

### Anaerobic / Aerobic Sequential reactors

The flow rates in both reactors was 200 mL/day. The sludge ages (θc )in both reactors were choosen as 1 day, 6 days, 11 and 13 days. The operational conditions for all reactor were given in Table 2.

**Table 2.Operational conditions for anaerobic/ aerobic reactors**

| Parameter | Unit | Value (Anerobic R.) | Value (Aerobic R.) |
|---|---|---|---|
| Volume (V) | mL | 200 | 200 |
| Flow (Q) | mL/day | 33 | 33 |
| Retention time (θH) | day | 1,6,11,13 | 1,6,11,13 |
| Sludge age (θc) | day | 1,6,11,13 | 1,6,11,13 |
| MLSS(reactor inside) | mg/L | 7.064 | 4,951 |
| MLVSS(reactor inside) | mg/L | 5.65 | 3,962 |
| F/M | mg COD/ mg MLVSS*day | 0.69 | 0.43 |
| pH | - | Set to 8 | Set to 8 |

### Operational Parameters in Sonication System

The volume in the sonication reactor is 150 mL. The flow rate was 0.08 mL / h. Retention time in sonication reactors is 30 minutes (Table 3.)

**Table 3.Operational conditions in the sonication reactor**

| Parameter | Unit | Value |
|---|---|---|
| Volume (V) | mL | 150 |
| Flow (Q) | mL/h | 0.08 |
| Retention Time (θc) | Min | 30 |

### Operational Parameters in Photolysis

The volume in the photolysis reactors is 110 mL. The flow rate is calculated as 0.37 mL / h in photolysis reactors (Table 4.).

**Table 4. Operational conditions in the Photolysis reactor**

| Parameter | Unit | Value |
|---|---|---|
| Volume (V) | mL | 110 |
| Flow (Q) | mL/h | 0.37 |
| Retention Time (θc) | min | 5 |

### Operation Parameter in Photolysis+ nano-$SiO_2$

In photolysis reactors, the volume is 100 mL. The flow rate is calculated as 0.02 mL / h in photolysis reactors. No proper yield was obtained in the photolysis + nano-SiO2 operation (Table 5.).

**Table 5.Operational Conditions in Photolysis+ nano-SiO2**

| Parameter | Unit | Value |
|---|---|---|
| Volume (V) | mL | 110 |
| Flow (Q) | mL/h | 0.03 |
| Retention Time (θc) | min | 60 |

### Operational Parameter in Reverse Osmosis

The RO system has a volume of 5 liters. Table 6 shows the values for flux, pressure and pH operating parameters for the RO system.

**Table 6. Operational Conditions in RO**

| Parameter | Unit | Value |
|---|---|---|
| Volume (V) | L | 5 |
| RO Flow (Q) | ml/h | 0.3-0.5-1.0 |
| RO Pressure | bar | 10-20-30 |
| pH | - | 4-7-8 |

### Analytical Methods

**Total COD** was measured according to Standard Methods (2012), 5220 D according to closed reflux method. **Dissolved COD** was measured by filtering the wastewater through a membrane with a diameter of 0.45 microns. Water from the membrane filter was taken and COD was measured according to the Standard Methods (2012) 5220 D.**Total Nitrogen** was measured by Spectroquant Merck kits numbered 1.14537 in a spectrophotometer (Standard Methods, (2012)).**Total Phosphorus** was measured based on Standard Methods (2012) in a Merck photometer with the spectroquant phosphate phosphorus kits numbered 1.14729.01.**MLSS and MLVSS** were measured according to Standard methods(2012) 2540 D. and 2540 E, respectively. The **Heavy Metals** were measured following the Standard Methods (2012) -on the Chrome ICP device according to the Standard Methods 3500 -Cr. Sulfate ($SO_4$), was measured according to Standard Methods 4500 $SO_4$-C. Boron (B), was measured according to Standart Methods 4500-B. Potassium (P), Calcium (C) and Magnesium (Mg) was measured according to Standard Methods 3120-B.Carbonate ($CO_3$) was measured according to Standard Methods 2320 B.

## Results and Discussion

### Anaerobic / Aerobic System Efficiencies

The performance of the biological system was tabulated in Tables 7 and 8 for anaerobic and aerobic reactors..

**Table 7.Anaerobic Reactor**

| | ANAEROBIC REACTOR | | | | |
|---|---|---|---|---|---|
| **SRT(Days)** | **Imput COD (mg/L)** | **Output COD(mg/L)** | **Yield (%)** | **TS in Effluent (mg/L)** | **Total Gas (ml/day)** |
| 5 | 19300 | 11580 | 40 | 1300 | 1.5 |
| 10 | 19300 | 10422 | 46 | 900 | 0.3 |
| 15 | 19300 | 9843 | 49 | 600 | 0.14 |
| 20 | 19300 | 7720 | 60 | 500 | 0.12 |

**Table8.Aerobic Reactor**

| | AEROBIC REACTOR | | | |
|---|---|---|---|---|
| **Days** | **Imput COD (mg/L)** | **Output COD(mg/L)** | **Yield (%)** | **TS in effluent mg/L)** |
| 5 | 7720 | 4246 | 45 | 950 |
| 10 | 7720 | 3860 | 50 | 562 |
| 15 | 7720 | 3397 | 56 | 400 |
| 20 | 7720 | 2625 | 66 | 300 |

The maximum COD removal in anaerobic reactor was 60% while the maximum COD removal in aerobic reactor was 66%. The total yield of sequential biological reactor was found to be as 86%.

### Yields obtained in the sonication reactor

The maximum sonication yield was observed as 53% for a 45 min retention time in the sonicator (Table 9).

**Table9. Removal efficiencies in Sonicator**

| **Time** | **Output aerobic R. COD; input sonication COD (mg/L)** | **Output Sonication COD value (mg/L)** | **Yield (%)** | **Total Yield (%)** |
|---|---|---|---|---|
| 10 | 2625 | 1785 | 32 | 76 |
| 20 | 2625 | 1523 | 42 | 80 |
| 45 | 2625 | 1234 | 53 | 84 |
| 70 | 2625 | 1995 | 24 | 74 |

### Performance of photolizis and adsorption processes

The effluent of sonication reactor was taken under sunligth between 10 and 70 minutes. No significand COD losses was detected(data not shown). In the same time the effluent of the sonication reactor

was taken in dark conditions for 24 and 48 hours. No COD removal was observed.

### Performance of photocatayitic removal with Nano $SiO_2$

The effluent of photolisis process was processed with 0,5 mg/l Nano SiO2 under sunligth in times between 12.00 a.m morning and 15.30 p.m afternoon. The maximum COD yield was obtained after 45 min photodegradation time (Table 10).

**Table 10. Measured Yield from Photocatalytic Process**

| Time | Output of sonicator; input of photocatalytic R. (mg/L) | Output Photocatalyitic COD value (mg/L) | Yield (%) |
|---|---|---|---|
| 10 | 1234 | 815 | 34 |
| 20 | 1234 | 766 | 38 |
| 45 | 1234 | 741 | 40 |
| 70 | 1234 | 741 | 40 |

### Performance of Revers Osmosis ( RO) process

In the effluent of the RO; the COD yield reached 100%. The yields of Phenol, TN, TP Dissolved COD reached approximately to 99,7%( Table 11). Table 12 exhibited some removals for the some metals and cations. The effluent yields achieved in this Table were compared with the Irrigation water standards given in Table 13.

**Table 11. Values from Reverse osmosis and Total Yields in the Raw OMW**

| Parameter | Unit | Value | | Yield % |
|---|---|---|---|---|
| | | Raw Wastewater | Output RO | |
| COD | mg/L | 1234 | 0 | 100,00 |
| Phenol | mg/L | 107,7 | 1,34 | 96,9 |
| Total N | mg/L | 70 | <0,4 | 99,3 |
| Total P | mg/L | 168,5 | <0,5 | 99,7 |
| Dissolved COD | mg/L | 3000 | 0,4 | 99,98 |
| Dissolved SS | mg/L | 4500 | 1 | 99,97 |

**Table 12. Chemicals in RO Output**

| Parameter | Unıt | Value |
|---|---|---|
| Boron | mg/l | <0,183 |
| Calcium | mg/l | 16,8 |
| Magnesium | mg/l | 2,23 |
| Iron | mg/l | 0,207 |
| Sodium | mg/l | 3,57 |
| Aluminum | mg/l | 0,033 |
| Zinc | mg/l | 1,36 |
| Bullet | mg/l | <0,01 |
| Potasium | mg/l | 3,24 |
| Elektrical Conductivity | mikrosiemens/cm | 182 |
| E.Coli | colony/100ml | - |

The effluent of RO was correlated with the Turkish Water Pollution Control Regulation given in Table 13. According this evaluation the RO effluent of the textile industry wastewater quality was recorded as first and second class irrigation wastewaters.

**Table 13. RO output water according to SKKY water class (Water Pollution Control Regulation –WPCR (2010)**

| Parameter | Measured Concentrations | Technical Procedures Communique Of SKKY Table.E.7.2 Irrigation Water Quality Parameters Based On Classification Of Irrigation Water (2010) | Water Class |
|---|---|---|---|
| Boron (B) | <0,183 (mg/L) | <0,5 (mg/L) | 1. Class |
| Na | 3,57 (mg/L) | 3-9 | 2. Class |
| Conductivity | 182 mikrosiemens / cm | >700 | 1. Class |
| Ca | 16,8 (mg/L) | - | - |
| Mg | 2,23 (mg/L) | - | - |
| E.coli | 0 colony/100mL | - | - |

### Cost Analysis

The sum of the cost of reactors were 0,271€. The nanocomposite cost was calculated as 0,6 €. The pumps, electricity and operational cost were 0,145€. The cost analysis was illustrated in Table 14. In order to treat 1 $m^3$ OMW wastewater the total cost was calculated as 1.033€. The quality of this wastewater is high and it can be used in the recreational purposes to irrigate the grass and to growth the herbes or to irrigate some nutritional foods in the pedosphere.

**Table 14. Cost Analysis**

| | Features | Cost € | Total Cost € |
|---|---|---|---|
| Cost Of Both Anaerobic And Aerobic Reactors | Reactor Cost | 0,011 | 0,271 |
| | Oven Cost | 0.26 | |
| Photocatalytic Reactor Cost | Photo Reactor Cost | 0.017 | 0,617 |
| | Nanoparticcle Cost | 0.6 | |
| Total Cost | | | 0,888 |
| Ro Cost | Pump Cost | 0.016 | 0,145 |
| | Electric Cost | 0,061 | |
| | Operating Cost | 0,068 | |

## Conclusions

The textile industry wastewater can be treated successively and recovered as irrigation water by using sequential biolagical, sonication, photocatalyzis and RO processes. The maximum COD removal in anaerobic reactor was 60% while the maximum COD removal in aerobic reactor was 66%.The total yield of sequential biological reactor was found to be 86%. In the sonication reactor THE yield was observed as 53% for a 45 min retention time in the sonicator. No removal of COD was observed via photolysis and adsorption. The maximum photo catalytic removal of COD was 40 % at a nano SO2 concentration of 0,5 mg/l after 10-60 min. In the effluent of the RO; the COD yield reached 100%. The yields of Phenol, TN, TP Dissolved COD reached approximately to 99,7%. In order to treat 1 m3 OMW wastewater the total cost was calculated as 1.033€.

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