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PURIFICATION OF OLIVE MILL WASTE: CIRCULAR ECONOMY MODEL FOR THE MEDITERRANEAN REGION

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Received 15 February 2024; accepted 30 May 2024; published 30 June 2024

Abstract. When olive mill wastewater is given directly to nature without being treated, natural waters become coloured, aquatic life is adversely affected, surface and underground waters are polluted, bad odours occur, and soil quality deteriorates. For these reasons, waste water is not allowed to be poured into soil and water without being discharged. This problem has become a problem that needs to be solved for the Mediterranean countries. With this study, acid cracking and chemical treatability studies achieved the conversion of the physicochemical pretreatment process of olive black water to an automation system. With the applied processes, removal efficiencies of 85% for Chemical Oxygen Demand, 99% for suspended solids, 97% for oil grease and 92% for phenol were obtained. Since the mixing processes of the tanks in the chemical additions are long, the heating chamber in the acid cracking process is designed larger than the tanks. Thus, when there is a black water density in the system, the heating tank in this system can be used as a heating and resting tank in sudden densities in this system. This waste separation process includes producing raw materials, protecting natural resources, and increasing sustainability. In particular, separating the phenol material and making it usable have been essential gains.

Keywords: life cycle practice; sustainability; olive processing; olive mill wastewater; Mediterranean countries

Reference to this paper should be made as follows: Feyzioglu, A., Ersoy, S., Omoruyi, T., Santoro, D., Piccinetti, L. 2024. Purification of olive mill waste: a circular economy model for the Mediterranean region. *Insights Into Regional Development*, 6(2), 92-102. [http://doi.org/10.9770/IRD.2024.6.2\(6\)](http://doi.org/10.9770/IRD.2024.6.2(6))

JEL Classifications: Q1, Q2

Additional disciplines: Environmental Economics, Life Cycle Models and Saving, Industrialization, Environment

1. Introduction

According to archaeological excavations, the olive tree can be traced to 12000 BC. Since this known date, olive trees were planted, olive oil was produced and stored, and press and storage containers used in these processes were found. Olives may differ according to the region and type of olive production and may have aroma, colour, and taste (Arvanitoyannis, Kassaveti & Stefanatos, 2007). While 80-84% of the total olive oil production in the world is met by EU countries, 97% of this rate is met only by the countries in the Mediterranean region. According to the 2008 - 2009 harvest period data of the International Olive Oil Council, Spain ranks first with 40.12% in world olive oil production, Italy ranks second with 19.54%, Greece ranks third with 12.91%, and 5%, Tunisia is at the fourth place with a rate of 0.58, and Turkey is in the fifth place with a rate of 5.55% (International Olive Council, 2011). Portugal, Morocco, and Algeria are other producing countries that follow

these five major producing countries. Except for the Mediterranean Region, olives are produced in the Middle East, America, Argentina, and Australia (Paraskeva & Diamadopoulou, 2006).

The common methods employed in olive oil production are the conventional pressing two-phase production process and the three-phase production process. In the two-phase production process, black water comes out with pomace. Depending on the production method, there is less volumetric but more heavily polluted black water formation in the traditional method (Yalılı Kılıç, Kestioğlu & Kaya, 2014; Dahdouh et al., 2023). While 50 kg of water is released per 100 kg of olives in traditional production facilities, 110 kg of water is produced in continuous production facilities (Vitolo, Petarca & Bresci, 1999).

When black water is released directly into nature without treatment, natural waters become coloured, adversely affecting aquatic life, surface and groundwater pollution, bad odours, and soil quality deterioration. For these reasons, black water cannot be poured into soil and water without discharge. This problem has become an issue that must be resolved for the Mediterranean countries. Olive black water varies for the area where each olive oil producer is located. Blackwater is dark brown in colour. It has typical characteristics such as acidic pH (3 – 5.9), 220 g/L, COD value, 80 g/L phenolic content, 20 g/L total solids content and low biodegradability (Azbar et al., 2004)

This study aims to investigate the ecological effect of mill wastewater that is disposed of during olive oil production on some chemical and biological properties in the soil. Olive production is an important agricultural activity in the Aegean and Marmara Regions of Turkey. There are approximately 750 million olive trees worldwide, 98% located in regions close to the Mediterranean. The countries with the highest olive production are Spain, Italy, Greece, and Turkey. The wastewater formed during olive oil production contains many harmful substances that threaten soil and plant productivity. The damages caused by these harmful substances to the environment cause ecological and economic losses (Doğan, Sarioğlu & Ağca, 2016; Fleyfel et al., 2022).

Olive mill wastewater is an important problem in countries that can produce and process olives (Piotrowska et al., 2005; Klisovic et al., 2021). Disposal of the formed composition is hazardous (Tunalıoğlu & Bektaş, 2011; Rapa & Ciano, 2022), chemical (Uzun & Seferoğlu, 2017) and biological deterioration (Tunalıoğlu & Bektaş, 2010), adversely affects agricultural areas (Manavoğlu, Güneri & Yokaş, 2021) and can be harmful to human health (Kılıç, Kaya, & Kestioğlu, 2009). To reduce the harmful effects of wastewater, which is very difficult to dispose of due to the important pollutants in its content, it is generally used in suitable collection pools for a few months and evaporation. However, the methods used in the treatment of olive wastewater can be counted as aerobic treatment, anaerobic treatment, aerobic treatment + Fenton oxidation, chemical treatment, chemical + biological treatment, field treatment, adsorption, advanced oxidation processes, membrane processes, electro Fenton + anaerobic treatment, composting (Galloni et al., 2022; Roila et al., 2022; Mouzakitis & Adamides, 2022).

There are different processes in the processing of olive fruit. Batch (Traditional Pressing) Production Process, Continuous Production Processes, 2-Phase Production Process and Phase Production Process can be counted as the main methods used in olive oil production. The generally used production scheme is shown in Figure 1.

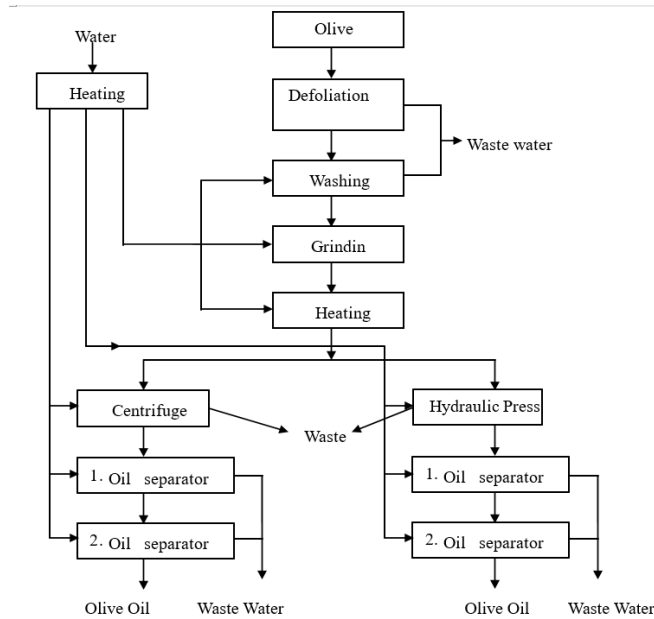


Figure 1. Olive Processing Production Flow Chart (Source?)

Although the olive harvest varies according to the geographical and climatic variables, the oil extraction process begins with the olives collected in February. The beginning of these processes is the separation of olive leaves; this process is done not to damage the branches of the machines but to obtain higher quality oil. After defoliation, the olives are crushed, kneaded, and separated from the liquid phase, and the oil is separated from the water and residue, respectively (Figure 2.)



Figure 2. Olive processing process: Olive formation and growth, pouring into the hopper, taking it into the tank, squeezing, automation of separation, obtaining the residue

The olives from the crushers are heated with hot water (max 25-30 C) in the malaxer and turned into a paste, so the oils in the dough can be combined and separated more easily. Mixers mostly consist of blades rotating slowly (20 rpm) in a metallic cylinder (Kiliç, Yonar & Kestioğlu, 2013). The liquid phase separation process aims to remove the oil from the dough. In horizontal decanters operating at a speed of 3,000 – 4,000 revolutions per minute, centrifugal force is applied to the slurry and oil, olive juice and pomace are separated by performing a centrifugation process to clean the substances such as pulp and black water that may remain in the oil. Olive oil takes its final form. The most important feature of applying these processes one after another is the reduction of fatty acid. The methods used in olive oil production are Batch (Traditional Pressing) Production Process, Continuous Production Processes, Two-Phase Production Process and Three-Phase Production Process. It is observed that the characteristics of the processes used in olive oil production have changed (Arvanitoyannis, 2008).

2. Methods

2.1. System Design

In this study, the electrical diagram of the olive mill wastewater treatment project, which was made from the E-plan application, was created. Macro was created by using the product codes and pins of the materials used, and the electrical connection of these products was made. The electrical scheme continues with the main supply and then the heaters and power supply fed from there. Then step drivers and plc modules fed from this power source are shown. CPU and analog heat module are used as PLC module and the connection of these modules with drivers and heaters is shown. Then, the PLC output and the control connections are shown, and finally, the HMI screen connected to the CPU is shown. This display order has been chosen for the technicians who will make the panel connections to work more easily. The most basic operation when drawing with the e-plan program is to clearly show the connection and interruption points, which clearly show the connections and their distribution. Next comes the accuracy of the macros used and the accuracy of the macro pins. Because of connection problems on the panel, the faults in the main supply connections cause the main damage to the panel. Then faults in the power supply feeds are important. The connections are checked by eliminating the contradictions in the product data sheets and the interruption points.

A suitable floor sheet was arranged for the automation panel to be made. Cable ducts and rails were hammered into the floor sheet. Then the PLC group and other materials were assembled. The terminals to be used were determined and arranged on the rails. The supply voltage of the panel was passed through a 220-volt fuse, and the 24-volt power supply was connected to the input supply. The input supply to the winding transformer for 48 volts, which is the supply of the cartridge resistance, which will receive another supply from 220 volts, was connected through the fuse. The output voltage of the winding transformer is connected to the 1st contact of the 48-volt SSR; the 2nd contact is connected to the resistors, whose output contact is the output contact. The resistance start signal coming from the CPU is given by passing it over the Relay. The start signal from the CPU is connected to the A1 terminal of the relay, 0 volts to the A2 terminal, and 24 volts to the 11th terminal, that is, to the COM terminal. The connection from the 14 terminals of the relay is made to the SSR A1 terminal. 0 volt is connected to terminal A2. The parts used in the development of the designed system are:

1. Siemens S7-1200 CPU
2. Siemens KTP-700 operator panel
3. Siemens S7-1200 analog input module
4. 2 pcs Nema 17 Stepper motor
5. 2pcs Stepper Driver
6. 220/48 Volt 50 VA Isolation Transformer
7. 24 Volt power supply
8. SSR (Solid State Relay)
9. Relay
10. Thermocouple

11. 2 pieces of cartridge resistance
12. Heating Chamber
13. Mixer Hopper
14. 2 pcs Mixer
15. Insurance
16. Terminal

Since it is done over the standard process in the project, the process is followed over the HMI panel. Siemens KTP-700 basic panel is used as HMI panel. Panel programming was done through TIA PORTAL program. The amount of wastewater can be viewed on the screen used. The number of chemicals used, which varies according to the amount of wastewater used in the applied process, can be monitored from the panel.

The process consists of 3 stages and is divided into 3 sections on the panel. You can follow the addition of sulfuric acid in 1 stage, the working status and speed of the stepper motors, the pH value, which is the last part of the 1 stage, is measured and the measured pH value is seen at Figure 3.

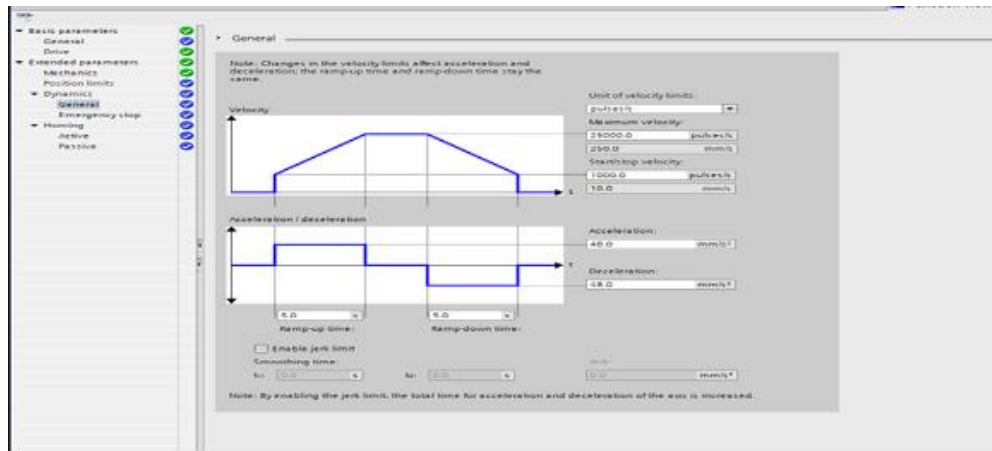


Figure 3. PH value

In the second stage, the temperature of the heating tank is measured with a thermocouple and shown on the panel as Heating Tak(C). The working status of the resistors used to heat the heating tank and the working status of the scrapers can be checked. It is the 3rd stage and the last stage of the process. (Figure 2.) The use of sodium hydroxide in the 3rd stage, the amount of ferric chloride and polyelectrolyte usage, the working status and speed of the stepper motors, the pH value of the tank, which is the last application of the 3rd stage, can be measured from the panel (Figure 4).

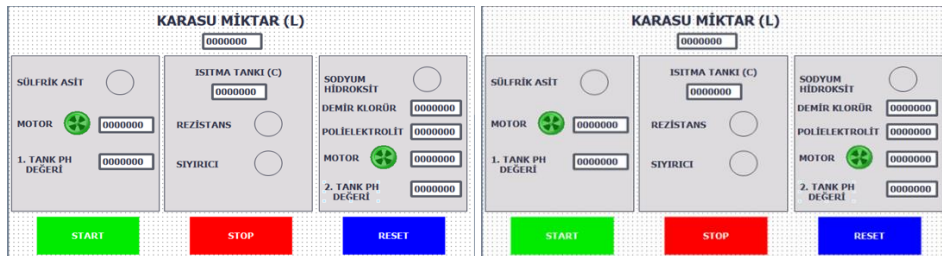
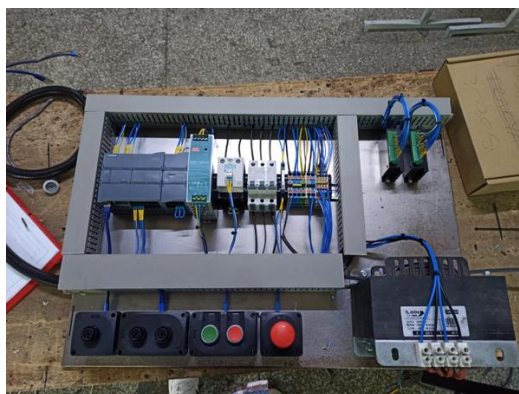


Figure 4. Process control preface

There are Start-Stop and Reset buttons on the panel. The system as Figure 5 can be managed with buttons that act as START button to start the system, STOP button to stop the system, and RESET button to start the system from scratch.

**Figure 5.** Treatment System control panel

2.2. Materials

The characterization of olive, black water, which we used as a reference in the treatment process, is given in Table 1.

Table 1. Content of black water waste

Parameter	Unit	Value
COG	mg/L	128000 ± 1000
ACM	mg/L	36300 ± 492
Oil-Press	mg/L	8920 ± 199
Fenol	mg/L	3440 ± 151
TOK	mg/L	26400 ± 400
pH	-	4,91
Electrical Conductivity	mS/cm	7,84

2.2.1. Chemical Materials

For chemical pretreatment, Sulfuric acid (H_2SO_4) is used to separate oil and black water and lower the pH to Iron (III) chloride ($FeCl_3 \cdot 6H_2O$). To combine the particles in Blackwater, Polyelectrolyte ($(NH_4)_2S_2O_8$) The polyelectrolyte is used as the last step, which binds the particles that start to combine with iron (III) chloride much faster and more effectively (Figure 6).

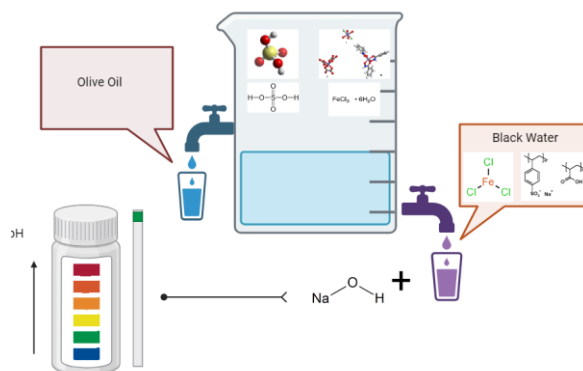


Figure 6. Chemical decomposition processes

Particles of remaining harmful substances are captured by filtration and the remaining water is sent for further chemical treatment. Sodium Hydroxide (NaOH) was used to increase the pH level.

3. Results

The physico-chemical treatment processes of the black water produced due to the processing of Memecik Olives grown in Aydın province of Turkey were automated and the purification processes were carried out. Memecik olive is the first Turkish olive registered by the EU, and its homeland is the Southern Aegean slopes. It has the highest quality and taste with plenty of sunlight, ideal heat-rainfall environment and special soil conditions. Memecik olive oil contains high levels of polyphenol and α -tocopherol (Vitamin E) and its nutritional properties. In the analyzes made, it was determined that Memecik olive oil has superior properties in terms of polyphenols. [sources of polyphenols used in the production of chemicals]. The automation system we have developed for the purification of the black water produced in the processing of olive oil consists of acid cracking tank, slow mixing tank, oil skimming unit, chemical treatment and filtering units. The results obtained from the created system are explained in detail below.

Since black water contains oil, acid cracking was performed as the first process. By adding H₂SO₄ to the raw black water, the pH value of the black water was reduced from 5 to 2 (Figure 7)

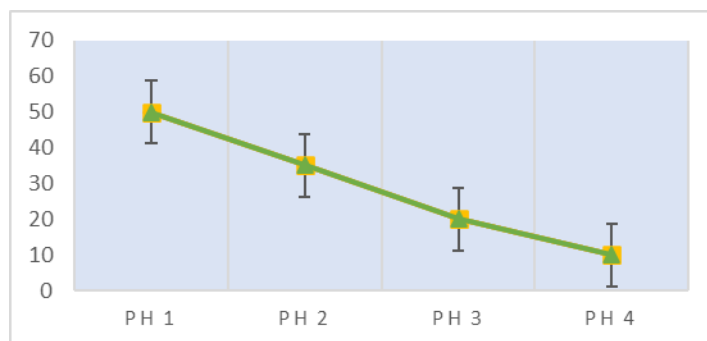


Figure 7. The effect of the number of filtration processes on the pH value

The slow mixed process starts (30-45 rpm); when the oil in the black water is easily removed from the upper phase, the tank is heated up to 60 - 70°C, and the temperature is measured with the help of a thermocouple (Figure 8). After the oils are stripped from the surface, the water is expected to cool while passing to the following process. 150 ml of oil came out of 1L black water; this rate may vary in black water coming from each facility.

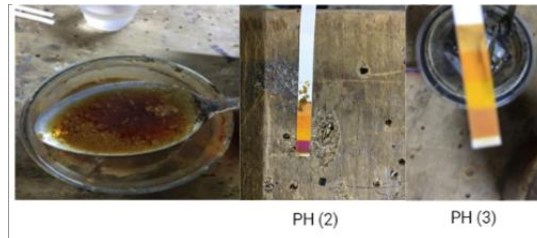


Figure 8. The oil coming out of the olive black water treatment tank and its PH values

Iron (III) chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) and Sodium Hydroxide (NaOH): By adding sodium hydroxide, black water at 2 pH value coming out of acid krakinh was increased to 3 pH value. By adding 10% ferric chloride to the amount of olive black water, rapid mixing is performed for 5 minutes and the attraction to each other is initiated before the residues in it combine with the polyelectrolyte.

Polyelectrolyte: After the rapid mixing process, 10% polyelectrolyte was added, and slow mixing was carried out for 30 minutes to increase the composition of the residues. After the slow mixing process, it was observed that the residues in the olive black water were visibly combined. In this way, the decoration procedure will work more healthily.



Figure 9. Residual material from the chemical treatability study of olive black water

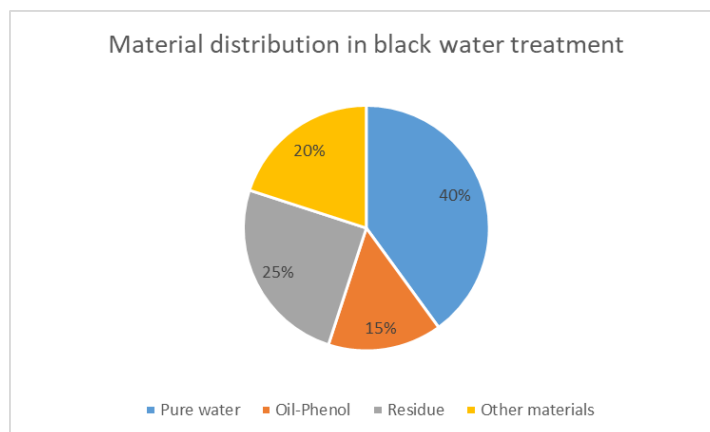


Figure 10. Components of material that come out as waste with the production process, separated by the automation system

Filtering the black water: To separate the residues formed in the olive black water, which is the last stage of the established system, the filtering process was started, and a cloth filter was used to filter the water. In the filtration

procedure process, the separated residues with visible sediment separation can be used as fertilizer because they are separated from oil and grease. The residues released during the filtration process are shown in Figure 9 and Figure 10.

4. Conclusions

A prototype of Olive Mill Waste Water System has been developed. The automation system has been used for the purification of the waste water produced in the processing of olive oil. This consists of acid cracking tank, slow mixing tank, oil skimming unit, chemical treatment and filtering units for chemical pretreatment in the system.

Sulfuric acid (H_2SO_4) is used to separate oil and wastewater from each other and to lower the pH to 2. Iron (III) chloride ($FeCl_3 \cdot 6H_2O$) To combine the particles in Blackwater. Polyelectrolyte ($(NH_4)_2S_2O_8$) The polyelectrolyte is used as the last step, which binds the particles that start to combine with iron (III) chloride much faster and more effectively. It captures the particles by filtration and sends the remaining water to advanced chemical treatment. Sodium Hydroxide (NaOH) was used to increase the pH level.

In conclusion, the system can increase in the form of oil, wastewater and sediment from the wastewater to a large extent.

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Author Contributions:

Ahmet FEYZIOGLU:

Ahmet Feyzioglu led the conceptualization and design of the study. He was instrumental in developing the overall framework of the circular economy model applied to the purification of olive mill waste. His expertise in mechanical engineering provided the foundational principles and methodologies utilized in the research.

Sezgin ERSOY:

Sezgin Ersoy contributed significantly to the experimental design and laboratory analysis. He conducted the primary experiments related to the purification processes and analyzed the data obtained from these experiments. His technical skills ensured the accuracy and reliability of the experimental results.

Trevor Uyi OMORUYI:

Trevor Uyi Omoruyi was responsible for the literature review and theoretical framework. He gathered and synthesized existing research on circular economy models and waste management practices in the Mediterranean region, providing the necessary background and context for the study.

Donatella SANTORO:

Donatella Santoro played a crucial role in the data interpretation and discussion of results. She provided insights into the implications of the findings within the broader context of environmental sustainability and policy. Her input was vital in framing the study's contributions to the field and potential impact.

Leonardo PICCINETTI:

Leonardo Piccinetti coordinated the project and managed communication among the team members. He also facilitated the collaboration with external stakeholders and ensured that the project milestones were met. His organizational skills and strategic oversight were key to the successful completion of the research.

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