

# Applying solar distillation for the sustainable management of olive mill wastewater

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**Abstract** Olive mill wastewater (OMWW) is a major by-product of olive oil production industry. Its chemical characteristics (high concentrations of COD and TSS, low pH value, existence of total phenols at the range of some ppm) does not allow its efficient treatment using conventional physicochemical or biological wastewater treatment methods. On the other hand, an important number of compounds with bioactive properties have been detected in OMWW increasing the scientific and economical interest for their recovery. In the current study, lab experiments were initially conducted using conventional distillation to study the role of temperature and initial OMWW characteristics to the composition of the distillate. Chromatographic analysis revealed the presence of different bioactive compounds in the distillates as well as the positive role of temperature's increase on their recovery. A pilot-scale solar still system was afterwards constructed to investigate the simultaneous solar drying of OMWW and the recovery of bioactive compounds with antioxidant properties in the distillate. The system operated in different experimental cycles and the produced distillates and solid residues were analyzed for the presence of bioactive compounds and the energy content, respectively.

**Keywords:** olive mill wastewater, solar distillation, polyphenolic compounds, antioxidants, recovery

## 1. Introduction

Agro-industry wastewater are produced in major quantities in Greece creating an important ecological problem for the local communities. They are characterized by seasonality on their production as well as high organic load. In various types of agro industrial wastewater, an important number of phenolic compounds has been detected. Many of these compounds are known for their anti-oxidative properties and they can be used as food additives or in cosmetic products (El-Abbassi et al., 2012).

Among different agro industrial wastewater, olive mill wastewater (OMWW) are characterized by high organic load, low pH values and high phenol content. Several

physicochemical methods, such as extraction with organic solvents, adsorption with resins or use of membranes, have been so far tested for the recovery of phenols from OMWW (Kalogerakis et al., 2013). These methods have significant drawbacks, either due to their environmental footprint or due to their increased operational cost. As a result, they cannot be applied in large-scale. An alternative, low cost and eco-friendly method for the treatment of OMWW is solar distillation. This method is well known for its ability to dewater rapidly wastewater and sludge. In a previous, we also showed that the solar distillation of OMWW could produce distillates containing phenolic compounds (Sklavos et al., 2015). In this study, lab scale experiments were initially conducted using rotary evaporator for investigating the effect of temperature on the phenolic profile of extracts. A pilot scale solar distillation unit was also constructed and used to investigate the simultaneous solar drying of OMWW and the production of distillates.

## 2. Methods

### 2.1. Sampling

For the lab experiments, OMWW samples were collected from a 3-phase olive mill located at Lesbos Island. For the solar distillation experiments, OMWW was collected from an olive pomace factory that receives wastewater from different 2-phase olive mills of Lesbos Island. The average concentration of total phenols at the samples for the lab experiments was equal to  $2.06 \pm 0.43$  g/L. The chemical characteristics of OMWW originated from the olive pomace factory are presented to Table 1.

**Table 1** Chemical characteristics of OMWW collected from the olive pomace factory

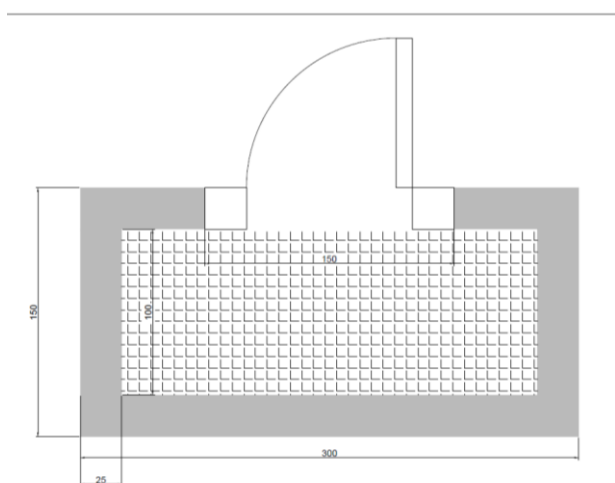
Parameter	Value ( $\pm$ std)	Parameter	Value ( $\pm$ std)
pH	45	Total Phenols	$4.7 \pm 0.21$ g/L
Conductivity	13.860	TSS	$29.8 \pm 0.8$ g/L
COD	$69.3 \pm 2.0$ g/L	VSS	$21.7 \pm 0.6$ g/L

## 2.2. Lab experiments

For the rotary evaporator's experiments, OMWW were centrifuged at 4000 rpm for 10 min, the supernatants were collected and distilled at different temperature (70, 80, 90° C). All experiments were conducted in five replicates and the collected extracts were examined for their total phenolic content as well as for the existence of specific phenolic compounds using liquid chromatography.

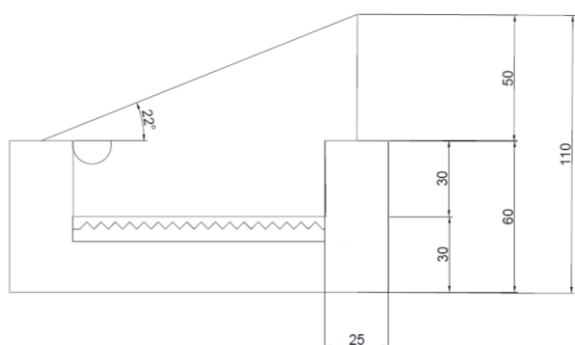
## 2.3. Solar distillation experiment

A pilot-scale solar distillatory unit was constructed. The inside size of the basin was 2.50 x 1.50 x 0.30 m as illustrated to Figure 1.



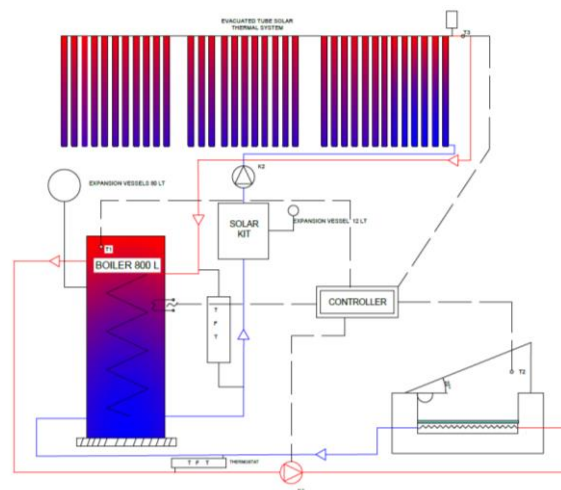
**Figure 1** Floor plan of solar distillation unit basin (all the dimensions are in cm)

The upper part of the unit was made by a frame of galvanized steel. The frame was supporting a 3 mm glass with inclination of 22°. A side view of the solar distillatory is shown in Figure 2.



**Figure 2** Side view of the solar distillation unit (all of the dimensions are in cm)

The unit was equipped with underfloor heating system supported by a solar heating system. The relevant info is shown in Figure 3.



**Figure 3** Mechanical sketch of the solar distillation unit with the plumbing and sensors' connection.

For its operation, OMWW was collected in March 2020 and April 2021 and two experimental cycles were conducted. At the first experimental cycle, no adjustment of the solar still temperature was applied while a volume of OMWW equal to 60 L was added. At the second cycle, the underfloor heating system was adjusted at 55 °C and 100 L of OMWW was added in the still.

Distillates were collected every 24 h and were measured for pH, conductivity, COD, total phenols and antioxidant activity with DPPH assay. The climate conditions as well as the temperature in the still was also recorded every 2 min using appropriate sensors and a data logger device.

## 3. Results

### 3.1 Lab experiments

In the rotary evaporator experiments it was observed that the concentrations of total phenols in OMWW extracts increased with the increase of temperature from 70°C to 90°C. HPLC analysis revealed the existence of hydroxytyrosol and tyrosol at 70°C, while caffeic acid was also detected at 80 and 90 °C. The highest concentrations of these compounds were found at 90 °C.

### 3.2 Solar distillation experiments

The first notable difference that was observed between the two experimental cycles was the average temperature that was achieved at the OMWW found in the solar still. In the first cycle, it was equal to  $24 \pm 6$  °C, while in the second, where the underfloor heating system was used, it was equal to  $40 \pm 6$  °C. The increased temperature enhanced distillate production. As a result, at the first experimental cycle, the average distillate volume was equal to  $2.65 \pm 1.35$  L/day and the whole volume of OMWW was evaporated in 27 days. At the second experimental cycle the average volume of the distillate was  $6.11 \pm 1.79$  L/day and 14 days were required for the evaporation of the 100 L of OMWW. The pH and conductivity values of the distillates were equal to

4.92 ± 0.19 and 1217 ± 390 µS/cm, respectively, for the first run and 3.87 ± 0.13 and 654 ± 150 µS/cm for the second run. The COD of the distillate was 4525 ± 3383 mg/L (1<sup>st</sup> run) and 6027 ± 2726 mg/L (2<sup>nd</sup> run). Concerning the phenolic content, the concentration of total phenols to the distillates were equal to 10 ± 12 mg/L (1<sup>st</sup> run) and 35 ± 11 mg/L (2<sup>nd</sup> run).

## Conclusions

The rotary evaporation experiments showed that the temperature increase from 70 to 90 °C enhanced recovery of polyphenolic compounds from OMWW. The application of underfloor heating system in the solar still improved its performance regarding the produced daily distillate volume and the found concentrations of phenolic compounds.

## References

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