Chapter 6
Physicochemical Characterization and Estimation of the Pollution Degree of Olive Oil Mill Wastewaters from the Cold Extraction System and the Traditional System

Zakia Gueboudji and Kenza Kadi

Abstract Olive oil production is widespread in Mediterranean countries such as Algeria. One of the main by-products from olive oil extraction is olive oil mill wastewater (OMW). It considers as an environmental problem for the producing countries. This study aimed to compare the physicochemical characterization and the pollution degree of OMW obtained from two different extraction systems, cold extraction system and traditional system, in Khenchela, eastern Algeria. The results of the physicochemical analyzes have shown that olive mill wastewaters from the two systems were very acidic and very rich in organic and mineral matter. There was a significant effect on electrical conductivity (EC), biological oxygen demand (BOD_5), chemical oxygen demand (COD), fatty matter (FM), organic matter (OM), and polyphenols (PP). While there was no significant difference for the parameters pH, humidity (H%), dry matter (DM), total suspended solids (TSS%), and mineral matter (MM). For the two pollution indices studied, BOD_5/COD and BI (biodegradability index), there was not a significant effect. It was recorded approximately similar values. In conclusion, the cold extraction system was the least polluting compared to the traditional extraction system. In addition, the pollution indicators clearly demonstrated the biodegradable nature of these wastewaters, for which biological remediation is appropriate.

Keywords Cold extraction system · Olive oil · Olive oil mill wastewater · Physicochemical characterization · Pollution · Traditional system

Z. Gueboudji (✉) · K. Kadi
Biotechnology, Water, Environment and Health Laboratory, Department of Molecular and Cellular Biology, Faculty of Nature and Life Sciences, Abbes Laghrour University, BP 1252 Road of Batna Khenchela, 40004 Khenchela, Algeria
e-mail: gueboudji.zakia@gmail.com; gueboudji.zakia@univ-khenchela.dz

K. Kadi
e-mail: kadi.kenza@univ-khenchela.dz

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6.1 Introduction

Mediterranean countries have become the largest consumers, producers, and exporters of olives and olive oil in the world. The olive oil production of the countries of the Mediterranean basin represents approximately 98% of the world's production. The production is concentrated in Spain, followed by Italy, Greece, Turkey, Tunisia, Morocco, Portugal and Algeria (Manzanares et al. 2020). Nowadays, with the promotion of the beneficial virtues of olive oil on human health, Olive oil production has risen in recent decades as a great source of antioxidants and vital fatty acids in the human diet, and it is now one of the world's most powerful diet trends (Souilem et al. 2017). The demand of olive oil does not cease increasing and consequently the production increases. The olive industry generates, in addition to oil as the main product, large amounts of solid by-products called olive mill pomace (OMP) and the other liquid called vegetable water or olive oil mill wastewater (OMW) (Gueboudji et al. 2021a). The world production of olive oil mill wastewater represents a volume of more than 30 million m^3/year (Nunes et al. 2018). For the production of olive oil, the fruits are harvested from the tree from the month of November, crushed and then kneaded to increase the yield of released oil. The extraction of the oil from the olive paste can be carried out according to different processes: (i) batch press, (ii) three-phase continuous and (iii) two-phase continuous (Klen and Vodopivec 2012). Three different products and by-products are generated for the first two processes (olive oil, pomace and vegetable water) and only two (olive oil and wet pomace) with the two-phase system. Even if they are less environmentally friendly and generate large quantities of olive oil mill wastewater, traditional pressing processes and three-phase centrifugation processes are still used in Algerian oil mills (Yakhlef 2019). Olive oil mill wastewaters or vegetable waters are water from vegetation that is generated during the extraction of virgin olive oil. These are effluents rich in organic matter (phenolic compounds, lipids, sugars, proteins, etc.) and in mineral salts (potassium, sodium, magnesium, etc.). These vegetable waters are often spread uncontrollably on agricultural soils or stored in evaporation ponds near oil mills, thus exposing water–soil–plant systems to inevitable pollution. The physicochemical and biological treatments of olive oil mill wastewaters, which consist in reducing their impact on water resources, are still insufficient and costly (La Scalia et al. 2017; Cedolaa et al. 2020; Gueboudji et al. 2022c). Algeria, a country with approximately 6.2×10^6 olive trees spread over an area of 471,657 ha according to provisional figures from the Directorate for the Regulation of Agricultural Production, is among the countries of the Mediterranean basin where the Olivier finds its area of extension. It is the ninth olive oil producer country in the world with a production of 80,000 tonnes in 2017/2018 of Mediterranean production. Thus, like all Mediterranean olive oil-producing countries, Algeria is faced with the problem of eliminating OMW with a production of 200,000 tonnes of OMW per year. In order to reduce the costs of the various treatments applied to OMW and to rationalize the management of their waste, research is focused on their recovery in various fields: composting, agriculture, cosmetics and even in the pharmaceutical industry (Senani-Oularbi 2018; Gueboudji 2022).

The objective of this study is to compare the physicochemical characterization and pollution degree of OMW obtained from two different extraction systems, which was the cold extraction system and the traditional system, in Khenchela, east of Algeria.

6.2 Olive Oil Extraction Processes

The processing of olives for extraction of oil can be done mechanically (by pressure or by centrifugation). Various extraction systems are used to extract olive oil (Baccioni and Peri 2014).

6.2.1 Basic Process or Press System (Most Common System)

Press system is the most common and well-known traditional olive oil extraction system in ancient times. Milling takes place with granite stone stones, which are converted into a tank whose floor is also made of stone. The grinders used for grinding are then slightly unbalanced with respect to the axis of rotation. So they slide a little on the surface when you turn, allowing you to knead the dough. The dough is obtained in about 30 min. The kneaded dough is placed in a thin 2 cm thick layer on a nylon fiberboard's called scourt. They are stacked on top of each other around a center pin mounted on a small cart. The set is placed on a hydraulic grass piston that allows a dough pressure of 400 kg/cm^2. The liquid phase flows into the receiving tank. This operation takes 45 min. Finally, centrifuges allow the separation of olive oil from vegetable waters. The solid and liquid phases are separated by simple pressure, while the oil is separated from the plant water by natural decantation or centrifugation in vertical centrifuges. The productivity of the extract is 86–90% compared to the oil in the fruit (El-Abbassi et al. 2012; Ben-Hassine et al. 2013).

6.2.2 Continuous Process (Centrifugation System)

This modern extraction design replaces the traditional pressing. It uses horizontal centrifuges called decanters, which improve the yield and productivity of the oil mills. There are two types: the two-phase system and the three-phase system.

6.2.2.1 Three-Phase Centrifugation System

This system allows the separation of three substances: olive oil, press residues and vegetable water (Zbakh and El Abbassi 2012). It requires the addition of hot water to the olive paste from the kneading before entering the centrifugal edge. So the different steps are separated according to their density by means of high centrifugal forces in machines called horizontal centrifuges to separate solid–liquid phases or vertical centrifuges to separate liquid–liquid phases that rotate at high speeds of about 3500 rpm (El-Hajjouji 2007; Zbakh and El Abbassi 2012).

6.2.2.2 Two-Phase Centrifugal System

This two-phase system (oil and ore), also called the ecological system, allows you to produce olive oil without the need for water in the carafe. So that the latter separates the oil and pomace-water, mixture from the vegetation into a single phase of pasty consistency called wet vinegar or two-phase vinegar, which limits the production of vegetable water (Rahmanian et al. 2014).

6.2.3 Refining

There is also another industrial process, which is refining. Refining is a relatively recent technology; it is the set of operations that transform crude oil into an industrial product by removing impurities that make it unfit for consumption. The oil contains desirable and useful elements (vitamins, unsaponifiable, etc.) and other undesirable elements such as (waxes, peroxide, free fatty acids, etc.). The purpose of refining is to maintain or improve organoleptic characteristics: (taste, neutral odor, clarity, light youthful color), nutritional characteristics and the stability of fatty substances in general. To do this, it implements several steps to eliminate, for example; waxes, free fatty acids, pigments, metallic traces, etc.), which are compounds that are harmful to the quality (Peri 2014).

6.3 Olive Oil Mill Wastewater (OMW)

The olive oil extraction industries generate a large quantity of by-products and residues (pomace and wastewater) calling for specific management, in order to minimize or attenuate its nuisances, and thus enhance and exploit their wealth. The pomace represents the solid fraction, coming from the pulp and the stone of the olive. This waste is generally recycled in different areas, including composting, energy production, animal feed, etc. Olive oil mill wastewater represent the liquid fraction from vegetation water and water added to extraction processes. These waters remain difficult to treat, given their acidity and their high pollution load. Olive oil mill wastewater (OMW) is a reddish-brown colored liquid, which turns black, foul smelling, cloudy in appearance and has a specific smell of olive oil (Rahmanian et al. 2014; La Scalia et al. 2017).

6.4 Physicochemical Properties of OMW

The amount and physicochemical properties of waste created are determined by the utilized oil extraction technology, processed fruits, and operation circumstances (added water, temperature, etc.). During the washing of the olives prior to milling, a minor quantity of solid waste (leaves and tiny twigs) is created. Nonetheless, these by-products do not provide a management challenge. The primary waste from three-phase extraction systems and typical mills is OMW. It is made up of fruit vegetable water and water utilized at various phases of oil extraction. Furthermore, the three-phase extraction techniques produce solid waste, which is utilized to extract olive kern. Table 6.1 summarizes the physicochemical properties of OMW as reported by various publications. The composition of OMW varies greatly depending on the olive type, the maturity of the fruit, the volume of added water, and the extraction technique (press or centrifuge). The typical weight content of OMW is 83–94% water, 4–16% organic molecules, and 0.4–2.5% mineral salts. Among other things, the organic fraction contains 2–15% phenolic compounds, which are divided into low molecular weight (tyrosol, hydroxytyrosol, p-coumaric acid, ferulic acid, syringic acid, proto-catechuic acid, etc.), and high-molecular-weight compounds (tannins, anthocyanins, etc.) and catechol-melaninic polymers. It has a dark hue (due to lignin polymerization with phenolic chemicals), a high acidity (pH about 5), and strong electrical conductivity. The most recent characteristic varies according to the salt content of OMW, which is determined by the procedures utilized for olive fruit conservation before to milling (Souilem et al. 2017).

Table 6.1 Physicochemical characteristics of olive mill wastewaters (Souilem et al. 2017)

Parameters	Unit	Range of values
pH	–	4.8–5.7
Conductivity	mS/cm	5–81
COD	g/L	16.5–156
BOD	g/L	13.4–37.5
Dry residue	g/L	11.5–90
Lipids	g/L	7
Phenols	g/L	0.8–8.9
Sugars	g/L	1.3–4.3
Total nitrogen	g/L	0.06–0.9

6.5 Comparison Between Physicochemical Characterizations of Olive Oil Mill Wastewaters from the Cold Extraction System and the Traditional System

The treatment of wastewater is a thorny problem because it is rich in organic matter but also in pollutants and dangerous substances. Several parameters are indicators of the presence of pollutants in the water and their quantity. Among these parameters, four are very often used: pH, TSS, COD, and BOD_5. In this study, 13 characteristics are measured. OMW samples were taken from two different mill, traditional and cold extraction system located in Khenchela eastern Algeria. Potential of Hydrogen (pH) and electrical conductivity (EC) are measured directly in samples using pH meter and conductivity meter. Fatty matter (FM) was determined by the chloroform/methanol method. Total suspended solid (TSS) is obtained by filtration after centrifuging samples. Dry matter content (DM) and humidity (H%) was measured by drying at 105 °C for 24 h. Organic matter (OM) was calculated by the difference between the dry weight of the OMW and its weight after the calcination. Mineral matter (MM) was determined by weighing after ignition in a muffle furnace type (Nabertherm) at 550 °C, for 24 h. The chemical oxygen demand (COD) was determined using potassium dichromate, as described by BOD_5 (biological oxygen demand) is determined by the respirometric method. Analyzes were carried out in triplicate (Gueboudji 2022). Polyphenols (PP) were determined by the reagent method of Folin Ciocalteau (Müller et al. 2010), after an extraction with maceration according to the method described by Gueboudji et al. (2022d). BOD_5/COD is an indicator, which determines the degree of pollutants produced by liquid effluents. If, BOD/COD > 0.5, effluent treatable by biological processes. If, 0.2 < BOD/COD < 0.6, feasibility of treatment using selected microbial strains. If, BOD/COD < 0.2, biological treatment impossible (Radoux and Cadelli 1995). Biodegradability Index (BI) is found by calculating the COD/BOD_5 ratio. It indicates the significance of pollutants with little or no biological degradation (Rodier 1996), if, BI > 6, hardly biodegradable substrate. If, 3 < BI < 6, partially (or less easily) biodegradable substrates. If, BI < 3, Very easily biodegradable substrate.

The results obtained of physicochemical parameters are showed in Table 6.2.

The results of the physicochemical analyzes have shown that olive mill wastewaters from the two systems were very acidic and very rich in organic and mineral matter. There was a significant effect on electrical conductivity (EC), biological oxygen demand (BOD_5), chemical oxygen demand (COD), fatty matter (FM), organic matter (OM), and polyphenols (PP). While there was no significant difference for the parameters pH, humidity (H%), dry matter (DM), total suspended solids (TSS%), and mineral matter (MM). For the two pollution indices studied, BOD_5/COD and BI (biodegradability index), there was not a significant effect. It was recorded approximately similar values.

Table 6.2 Physicochemical characterizations

Parameters	Unit	Traditional system	Cold extraction system
pH	–	5.06 ± 0.02	5.11 ± 0.02
EC	mS/cm	4.10 ± 0.07	13.55 ± 0.05
FM	%	3.87 ± 0.10	1.98 ± 0.12
TSS	%	0.89 ± 0.03	0.98 ± 0.04
H%	%	94 ± 1.15	83.5 ± 1.02
DM	%	06 ± 0.22	16.5 ± 0.51
OM	g/L	13.05 ± 0.71	34.94 ± 1.25
MM	g/L	12.2 ± 0.08	24.6 ± 0.05
PP	g/L	7.3 ± 0.15	0.98 ± 0.09
BOD	g/L	51.1 ± 2.25	77.4 ± 4.15
COD	g/L	220 ± 4.00	250 ± 4.5
BOD_5/COD	–	0.23 ± 0.01	0.31 ± 0.01
BI	–	4.31 ± 0.03	3.23 ± 0.03

The comparative analysis of the physicochemical characteristics of the vegetable waters in the two different methods studied showed that most of the time, OMW are characterized by an acid pH (between 5.06 and 5.11), in due to the presence of organic acids (phenolic acids, fatty acids, etc.) (Zaier et al. 2017; Gueboudji et al. 2021b). Thus, the values recorded in our study are within the limit of the range quoted in the literature (4.2–5.9). However, the acidity of the vegetable waters is linked to the duration of their storage in the storage basins. This can also be explained by auto-oxidation and polymerization reactions that transform phenolic alcohols into phenolic acids. These reactions are manifested by a change in the initial coloring of the vegetable waters towards a very dark black color. Indeed, OMW samples studied are characterized by a very dark brown color. The mineral composition of the vegetable waters studied has shown that these wastewaters have a high saline load due particularly to sodium chlorides, probably linked to the salting practiced to preserve the olives until they are crushed, in addition to the natural richness of the olives in salts minerals. This could be explained by the high values of the electrical conductivity found in the various oil mills studied (between 4.10 and 13.55 mS/cm). The average contents in dry matter is of the order of 6 g/L and 16.50 g/L respectively. OMW studied are very rich in suspended solids, their average content is between 0.89 and 0.98 g/L. However, the average values recorded during this study were high, this could be explained by the fact that in the basins, the suspended solids of OMW drop under the effect of settling and this is probably due to the effect of the wind and/or the commotion caused when unloading the vegetable waters. In the same way, vegetable waters are very loaded with organic matter expressed in terms of BOD_5 and COD. Thus, according to Table 6.2, the values obtained can reach 51.1 g/L (BOD_5), 220 g/L (COD) and 13.05 g/L organic matter in OMW obtained from the traditional extraction system. For the cold extraction system, it found 77.4 g/L (BOD_5), 250 g/L

(COD) and 34.94 g/L organic matter. This shows the high oxygen demand for the complete oxidation of the organic matter contained in these effluents, which reflects their very important polluting powers. Thus, this COD content is very high compared to that recorded in other types of discharges: These values are 200–400 times higher than those of municipal waters are. Indeed, COD does not exceed 4.02 g of O_2/L in slaughterhouse effluents, which are considered the main discharges of a dominant organic nature (Aissam 2003; Gueboudji et al. 2022a).

The biodegradability index (BI) expressed by the COD/BOD_5 ratio is important for the definition of the purification chain of an effluent. The results of this report are an indication of the importance of pollutants with little or no biodegradability. Indeed, a low value of the COD/BOD_5 ratio implies the presence of a large proportion of biodegradable materials and makes it possible to consider biological treatment. Conversely, a high value of this ratio indicates that a large part of the organic matter is not biodegradable and, in this case, it is preferable to consider a physicochemical treatment. Thus, the average values of the COD/BOD_5 ratio of OMW studied is greater than 3, which is the limit threshold for biodegradability. Therefore, it can conclude that even if OMW studied have a high organic load, they are partially biodegradable. Examination of this report underlines the biodegradable nature of OMW of the two oil mills for which a biological treatment seems quite suitable. These discharges are also characterized by the predominance of toxic substances, in particular phenolic compounds. Values of 0.98 g/L in that of cold extraction system and can reach 7.3 g/L have been measured in OMW of traditional system. This high concentration could limit any natural biodegradation, and therefore could lead to a more or less profound disturbance of the entire ecosystem. Thus, phenolic compounds are very varied cyclic organic substances (Babić et al. 2019). According to Jail et al. (2010), their solubilization in oil is lower than that in vegetable water, which explains their high concentration in vegetable waters. According to Justino et al. (2012), these compounds have a variable structure, they come from the enzymatic hydrolysis of glucosides and esters from the olive pulp during the extraction process. According to the results, in OMW of traditional system, the highest concentrations of phenolic compounds were noted compared to the cold extraction system, this could limit any natural biodegradation, and therefore could lead to more disturbance, or shallower of the entire ecosystem. This variability in the phenolic composition of OMW between the two extraction methods depends not only on the technological processes used to separate the phase aqueous (OMW) of the oily phase, but on the variety, the maturity of the fruit and the climatic conditions, but also on the duration of storage of OMW and (Gueboudji et al. 2022b).

6.6 Conclusion

The discharge of OMW from olive oil producing industries is a major problem, especially for the countries of the Mediterranean basin, because they contain a large organic fraction and cause several types of pollution. The variability of the results obtained from the physicochemical properties of OMW samples is mainly due to a difference in the olive oil extraction process. They play an important role in the process of biological treatment of these effluents, the richness of their impact on the growth and development of microbes, as well as its treatment capacity. This study showed that the cold extraction system was the least polluting compared to the traditional extraction system, and made it possible to show that OMW studied have a high organic load and they are partially biodegradable according to the COD/BOD_5 ratio and subsequently the biological treatment processes for these OMW are more effective than the physicochemical ones.

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