

Existing and emerging technologies for the treatment of olive oil mill wastewaters

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ABSTRACT

Olive oil mill wastewaters (OOMW) represent a significant environmental problem especially in the Mediterranean, the main olive oil production region in the world. Characterization of OOMW is carried out by determination of various parameters, such as BOD₅, COD and content of total phenols. The aim of this paper is to evaluate existing (physical, biological etc.) and emerging technologies for an integrated treatment of OOMW. EU legislation regarding OOMW disposal practices is limited and only concerns the protection of the environment from the adverse effects in soils and water bodies.

1. INTRODUCTION

Olive oil mills generate large quantities of wastewaters which are slightly acidic and associated with high biochemical oxygen demand (BOD) and chemical oxygen demand (COD), up to 100 and 220 g/L, respectively. The phytotoxic effect of OOMW is partially attributed to organic fraction (phenolics and related substances) which is present in notable concentrations and inhibits the growth of certain microorganisms. The phenolic content (phenols, flavonoids or polyphenols) along with long-chain fatty acids produce methanogenic toxicity and therefore the option of discharging OOMW to land should be carefully considered (Paredes et al., 1986; Hamdi, 1992; D'Annibale et al., 2004; Mekki et al., 2007).

The characteristic dark brown colour of the effluents is due to polymerization of low molecular weight phenolics and is chemically related to lignin and tannin derivatives. Decolorization of OOMW cannot be effectively dealt using traditional biological waste treatment methods, such as those involving aerated lagoons or anaerobic digestion (Sayadi and Ellouz, 1992; Jarboui et al., 2008).

Problems arise mainly from the fact that olive oil production is intense and seasonal and therefore the treatment process should be flexible enough to operate in a non-continuous mode, otherwise storage of the wastewaters is required. Moreover, olive mills are family businesses and small-scale enterprises, mainly scattered around olive production areas, making thus individual on-site treatment options unaffordable (Paraskeva and Diamadopoulos, 2006).

Practically, all treatment processes developed for domestic and industrial wastewaters have been tested on OOMW but none of them appeared fully suitable to be generally adopted. OOMW management strategy should be therefore directed towards a combination of their detoxification and utilization for the production of valuable by-products (Roig et al., 2006; Oreopoulou and Russ, 2007).

The present paper presents briefly existing and emerging technologies for the treatment of OOMW generated mainly in the Mediterranean region. The legislative framework regarding OOMW management is also presented; however

no guidelines or specific disposal practices exist in the EU countries.

2. CHARACTERIZATION OF OOMW

Olive oil is produced in olive mills either by the discontinuous press method or by the continuous centrifugation method. In all stages of olive processing large quantities of clean water are consumed and wastewaters of various quality are produced depending on parameters, such as the olive variety, olive seed maturity, cultivation/processing method and geological-climatic conditions. However, OOMW production is strongly dependent on the processing system; in pressure systems the volume produced varies from 40-60 L/100 kg of olives, while in two-phase and three-phase centrifugation systems it is approximately 10 and 100 L/100 kg of olives, respectively (Kapellakis et al., 2008).

Mediterranean countries produce 97% of the total olive oil production, while European Union (EU) countries produce 80–84%. Spain is the biggest olive oil-producing country, followed by Italy and Greece. Table 1 shows an estimation of the total amount of OOMW produced annually ranging from 7×10^6 to over 30×10^6 m³. This large divergence of results can be partly explained by the fact that the provided data are only rough estimations, due to lack of clear information concerning the volume of the wastes actually produced (Kopsidas, 1992; Niaounakis and Halvadakis, 2006).

Characterization of OOMW is carried out using a number of chemical analyses, such as determination of BOD₅, COD, total solids, total phenols, total sugars, tannins and lignins, total fats, individual fatty acids, total organic carbon, total phosphorous, total nitrogen, metals and ash. These procedures are multistep and time-consuming; most of them require the use of toxic solvents and result in the production of laboratory wastes which should be also disposed of.

One of the most important analyte is the phenolic compound for which several methods have been described in the literature. Polyphenols which are not easily biodegradable, are toxic to most microorganisms and are seen

in high content in OOMW (up to 80 g/L) (Oreopoulou and Russ, 2007). OOMW phenolic content shows great variability depending on several factors, such as type of production process, type of olive and stage of maturity. The extraction system affects its concentration, with the two-phase decanter OOMW having the biggest volume (Lesage-Meesen et al., 2001).

Phenols are not found in their free forms in olives, but exist mainly as glucosides, tannins, antocianins and lignins. The 50 phenols that have already been identified in OOMW belong to three important categories of phenolic compounds: (a) cinnamic acid derivatives, (b) benzoic acid derivatives, and (c) compounds related to tyrosol (Miranda et al. 2001).

Table 1: Estimated OOMW volumes generated from olive oil processing (Niaounakis and Halvadakis, 2006)

Country	OOMW, m ³ /y	Olive cake, m ³ /y
Spain	2.8×10^6	1.6×10^6
Italy	2.4×10^6	1.6×10^6
Greece	1.4×10^6	0.8×10^6
Tunisia	0.55×10^6	0.3×10^6

3. LEGISLATIVE FRAMEWORK FOR OOMW MANAGEMENT

As OOMW are produced only in the Mediterranean region, EU policy has not brought into force any common guidelines for their management. EU Council Directive 91/271/EEC on “Urban Wastewater Treatment” concerns the protection of the environment from the adverse effects of the discharge of urban and agro-food industry wastewaters. Effective treatment should be therefore applied before wastewaters are discharged into receiving waters, while treated wastewaters should be reused whenever appropriate.

According to the above mentioned homogenized, unfiltered and undecanted wastewaters should not exceed BOD₅ and COD of 25 and 125 mg/L O₂. The maximum allowed concentration of phenols, total suspended solids, total phosphorous and total nitrogen is 1, 35, 2 and 15 mg/L, respectively.

The practices currently applied include land disposal, discharge into nearby rivers, lakes or seas and storage/evaporation in lagoons, causing thus serious environmental problems such as water resources contamination. The second significant problem to highlight has to do with the lack of a framework for separating soil from water receivers. The presence of organic matter as well as many inorganic compounds (nitrogen, phosphorus, potassium) causes severe pollution when OOMW are disposed of into water bodies, but when disposed of in soil erosion is prevented and can be beneficial to soil fertility (Rinaldi et al. 2003; Kapellakis et al., 2008).

In Greece there are no specific regulations regarding the discharge of OOMW. The main principles for OOMW management are based on the Greek Law 1650/86 "For the Protection of the Environment" according to which, olive mill owners are obliged to provide an environmental impact assessment study. The updated circular letter YM/5784/23-1-1992 (No 4419/23-10-1992) refers to the problems encountered due to OOMW disposal, the need for an efficient pre-treatment and the care required in order to avoid disposal to various water resources. Each Prefecture is responsible for adopting proper OOMW management practices encouraging different waste management approaches.

The issue of an olive-mill operation permit is subject to measures taken to treat OOMW, such as pre-treatment with lime before disposal, pre-treatment/fractionization by natural sedimentation and separate management of the individual fractions or disposal in constructed open ponds. However, there is no single technical solution that can ensure satisfactory treatment efficiency with an application cost within the economic means of each individual olive-mill owner, given its geographical distribution and the size of its olive-mill plants (Paraskeva and Diamadopoulos, 2006).

In the island of Crete south of Greece, OOMW were discharged until 1987 uncontrolled into the environment. About 80-90% of the total OOMW produced were disposed of in ephemeral rivers (Voreadou, 1994). Since then, local public authorities prohibited uncontrolled disposal in water bodies

and obliged mill owners to construct treatment units (involving mixing with $\text{Ca}(\text{OH})_2$ sedimentation and grease removal) and evaporation lagoons with low construction, operation and maintenance costs. To avoid potential downward OOMW leakage, a compacted clay liner should be placed at the bottom of the pond; after evaporation of OOMW, the remaining sludge may be landfilled (Kapellakis et al., 2002; 2006).

4. TREATMENT OF OOMW

Application of untreated OOMW on soils and crops as a fertiliser is a common practice, due to the organic matter and nutrients contained that could improve arid soils. However, the disposal problem of OOMW is not completely solved, since the effluents produced cause adverse impacts to aquatic life, change of the colour of natural waters, toxicity and odours. A balanced disposal method requires controlled land application of properly characterised wastewaters. Another important issue is that pre-treatment of OOMW should improve its quality as well as remove most of its toxicity (Saviozzi et al. 2001; López-Piñero et al., 2007; Saadi et al., 2007).

Several treatment options have been considered including physical, physico-chemical, biological, thermal technologies and combinations thereof, as well as other combined approaches that could improve decontamination efficiency.

4.1 Physical treatment

Physical processes involve the separation of different phases through mechanical means. When used, so far, for the treatment of OOMW are unable to reduce alone the organic load and toxicity of the wastes to acceptable limits. Dilution is very often used prior to biological treatment to reduce toxicity to the micro-organisms responsible for organic matter decomposition. Evaporation and sedimentation can concentrate OOMW by 70–75% mainly due to phase separation and dehydration; in both processes there are considerable odour problems

in open areas and the remaining paste requires further treatment.

Filtration and centrifugation increase the pH and conductivity of the effluents as well as remove organic matter (Paredes et al., 1998; Potoglou et al. 2004). The dissolved-air flotation method is a potential pre-treatment technique for the removal of suspended solids; the technique is quite inefficient though when the content of suspended solids is high (Mitrakas et al., 1996).

4.2 Physico-chemical treatment

Physico-chemical treatment of OOMW involves use of additional chemicals for their neutralization, flocculation, precipitation, adsorption, chemical oxidation and ion exchange. The neutralization technique can be used as a pre-treatment step for the removal of the suspended or colloidal matter from OOMW and it is performed either by reducing pH to the point of zero charge (pH~2–4) or by increasing it (pH~11). Flocculation induces aggregation of particles suspended in OOMW into larger particles. Precipitation is used to remove dissolved chemicals into an insoluble solid form.

Adsorption enables attachment of dissolved compounds to the surface of an adsorbent such as activated carbon, bentonite and other clays. Chemical oxidation is used for OOMW purification and involves agents like oxygen derivatives (hydrogen peroxide or ozone), chlorinated derivatives (chlorine dioxide, sodium hypochloride etc), potassium permanganate or a mixture of these. Ion exchange is ideal for removing heavy and alkali-earth metals as well as chloride, nitrate or sulfate ions (Niaounakis and Halvadakis, 2006).

4.3 Biological treatment

Biological treatment, employing the use of microorganisms to break down biodegradable chemical species present in OOMW, is considered environmentally friendly and cost effective. The actual type of microorganisms depends on the treatment option, i.e. anaerobic or aerobic.

Anaerobic process is used to remove organic matter in high concentration streams by converting organic compounds to methane and carbon dioxide; hydrolysis, acidogenesis and methanogenesis are the three major steps taking place. Aerobic process is used on lower concentration streams to further remove residual organic matter and nutrients from OOMW; it relies on microorganisms that thrive under aerobic conditions (availability of oxygen and nutrients).

Combined biological processes are used to meet treatment requirements, i.e. anaerobic digestion of OOMW is enhanced when an aerobic pre-treatment step is considered to reduce the amount of total phenolic compounds and associated toxicity. Composting (aerobic decomposition process) is used for the degradation of organic matter into a granular humus-like product, which can be used as a fertiliser or soil conditioner (Paredes et al., 1998; Paraskeva and Diamadopoulos, 2006).

4.4 Thermal treatment

Thermal processes involve the concentration of OOMW by reducing their water content and total volume. Three main treatment options are used: i) physico-thermal (evaporation - distillation of OOMW and drying of olive cake), ii) irreversible thermo-chemical (combustion and pyrolysis), which require expensive facilities and entail possible emission of toxic substances into the atmosphere and iii) combined physical and biological, such as lagooning where the sun's energy accelerates evaporation and drying of OOMW followed by partial degradation over long time periods.

Lagooning is used widely despite the fact that only reduces the volume of wastes without treating the pollutants and a black foul-smelling sludge, difficult to remove, is produced. Moreover, careful implementation should be considered due to serious threat of leakage of OOMW through the soil and into the groundwater (Caputo et al., 2003; Azbar et al., 2004; Niaounakis and Halvadakis, 2006).

4.5 Combined treatment

Complete abatement of OOMW pollutants can be hardly achieved by the adoption of a single process. The effective management of OOMW includes their pre-treatment before the application of the selected process. Experimental results show that prior to OOMW anaerobic degradation, ultrafiltration allows high removal of lipids and polyphenols but has poor selectivity, while centrifugation is preferable to sedimentation owing to smaller volumes of separated phase (Hamdi and Ellouz, 1993). Combining ozonation and aerobic treatment a total COD reduction of 82.5% can be achieved, which is higher than the percentage achieved when each of the above technologies is used alone. A similar COD reduction is seen when aerobic treatment precedes ozonation (Benitez et al., 1999).

Table 2 shows the investment cost for various combined treatments, the total cost/m³ of OOMW and the calculated cost/t of olive oil for a three-phase OOM generating 5000 m³ OOMW/year (olive oil:OOMW weight ratio of 1:5). The most expensive treatment [1] is beneficial due to the production of by-products. Combined treatments [2], [3] and [4] have low costs, but adverse environmental effects such as odours and water bodies' contamination. The calculated cost of treatments [5] and [6] is similar but is considered low when compared to the price of 1 t of olive oil which is approximately 3,000 €/t.

4.6 Other emerging treatment options

Integrated systems employing treatment of OOM effluents with sand filters (Achak et al., 2009), co-composting of olive mill sludge with poultry manure or sesame bark (Hachicha et al., 2009a; 2009b) or mixing them with calcareous soil and incubation under aerobic conditions (De la Fuente et al., 2008) are able to achieve a very high degree of treatment.

In the framework of LIFE07 ENV/GR/000280 project (duration: 01/01/2009-31/12/2012) entitled "Strategies to improve and protect soil quality from the disposal of olive oil mills' wastes in the Mediterranean region", protective/remedial technologies will be

developed to remove or significantly reduce the load of pollutants in soils and water bodies affected by the disposal of OOMW in the Municipality of Nikiforos Fokas, Rethymnon prefecture, Crete, Greece (PROSODOL project, 2009).

Table 2: Costs for various combined treatments, for a three-phase OOM (Azbar et al., 2004; EU INTERREG IIIA project report, 2008)

Combined treatment	Investment cost, €	Total cost, €/m ³ OOMW	Calculated cost, €/t of olive oil
[1]	1,150,000	59	295
[2]	72,600-138,000	2-3.6	~14
[3]	42,200	1.31	1.5
[4]	180,700	3.95	19.7
[5]	250,000	12	60
[6]	500,000-850,000	13.5-22.5	~90

[1]: Membranes for polyphenol recovery and composting

[2]: Phisico-chemical processing and evaporation

[3]: Forced natural evaporation

[4]: Forced mechanical evaporation and lagooning

[5]: Phisicochemical processing-ultrafiltration-reverse osmosis

[6]: Mechanical biological pre-treatment (biogas production)-sludge management (aerobic stabilization, solar drying)

One of the objectives of the project is the demonstration at a pilot scale of a low-cost OOM wastes pre-treatment technique in a tank or a lagoon containing reactive agents such as metallic iron and/or limestone and/or poor lignite to add alkalinity, remove some of the toxic load and degrade organic contaminants so that the main treatment in the following stages becomes easier. Limestone and lignite are redundant in Greece, while metallic iron is produced as by-product (waste) from a number of primary metallurgical and secondary metal finishing activities in all Med countries.

Initially, laboratory work will be carried out in plexiglas columns to assess the reactivity and longevity of the media used and establish the optimum residence time.

The issue of recovery of the final toxic load, by (acidic) washing and precipitation will be

studied; depending on its toxicity the small final toxic volume can be disposed of in engineered landfills or used in other secondary applications. Furthermore, regeneration of the reactive media used as well as toxicity of the precipitates will be studied in detail using standard EPA tests to define an overall pre-treatment management strategy. The use of an artificial wetland (active or passive) as a potential polishing step after pre-treatment may be also considered.

It is believed that such a pre-treatment will contribute to process optimization and will enable the establishment of an integrated strategy for the optimum management of OOMW.

Another important aspect that will be considered in the frame of PROSODOL is the implementation of a Life Cycle Analysis (LCA) for the entire olive oil production chain aiming at minimization of energy requirements and environmental impacts.

5. CONCLUSIONS

The existing treatments for the decontamination of OOMW which are characterised by high organic load and toxicity include physical, physico-chemical, biological and thermal technologies. These treatments when used individually suffer from drawbacks such as low efficiency or high cost. Combined or advanced alternative methods have been developed and show encouraging results, such as COD reduction up to 83% and very high degree of treatment. The legislative framework of the EU does not provide specific guidelines for OOMW management, due to their production only in the Med region. In most countries mill owners are responsible for the management of OOMW by using practices such as disposal into rivers or lagoons causing thus serious environmental problems.

In the framework of LIFE07 ENV/GR/000280 project entitled “Strategies to improve and protect soil quality from the disposal of olive oil mills’ wastes in the Mediterranean region”, protective/remedial technologies will be developed to remove or significantly reduce the load of contaminants in

soils and water resources affected by the disposal of OOMW. For this a low-cost OOMW pre-treatment technique will be developed in laboratory and evaluated in pilot scale using as reactive media metallic iron and/or limestone and/or poor lignite.

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