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ORIGINAL PAPER

Effect of temperature and aeration rate on co-composting of olive mill wastewater with olive stone wooden residues

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Abstract Co-composting of the solid residues and wastewater from the olive oil production process was examined as a potential bioremediation treatment for these wastes. Experimental results from a semi batch laboratory pilot plant were reported. Composting was performed for 20 days under constant moisture 40% and the temperature ranged from 55 to 72°C and the oxygen partial pressure from 10 to 17%. An operational region of temperature and oxygen partial pressure was defined in order to achieve a ratio of total olive mill wastewater consumption to olive stone wooden residue stabilization equal or greater than 2.5, the typical ratio for an olive mill plant. Another critical parameter for the optimisation of the 20-day co-composting process that was examined was the biological efficiency of the process, as the carbon dioxide produced to the total carbon available to biomass. A strong sigmoid correlation of co-composting efficiency with temperature derived, reaching a maximum plateau of 0.50 at 68°C. The optimum conditions for a 20-day semi batch co-composting proved to be 68°C and 16–17% oxygen partial pressure, indicating that this process could be an integrated treatment scheme for olive mills.

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Introduction

Olive mill wastewater (OMWW) arises from the production of olive oil in olive mills. It is produced seasonally by a large number of small olive mills scattered throughout the olive oil-producing countries. OMWW has a very high organic load, recalcitrant in nature and with a high amount of toxicity/phytotoxicity-associated compounds (Di Serio et al. 2008; Jarboui et al. 2008). Several physicochemical, biological and combined processes have been examined for the treatment of OMWW, resulting in considerable organic load and toxicity abatement. Although the problem of OMWW treatment could be more or less solved technologically, it is still far from being solved realistically, mainly because of practical/economical reasons. Owing to the small scale and dispersed nature of the olive mills and the seasonality of the process, affordable solutions have not yet yielded the required quality to meet stringent environmental standards (Paraskeva and Diamadopoulos 2006).

Apart from high strength wastewater, during olive oil extraction process solid residues derive. The final by-product of the olive oil production process is the olive stone wooden residue (OSWR). For every kilogram of olive processed, 0.25 kg of OSWR are produced. The calorific value of OSWR is about

4,500 kcal/kg and it is usually used as a fuel in greenhouses, ceramic and lime production plants, bakeries as well as in the olive mills and seed oil mills (Taralas and Kontominas 2006). Recently, the demand of OSWR is decreasing despite the fact that it is cheaper compared to fuel oil. This can be attributed to the fact that OSWR is more difficult to handle, transport, store and burn (Vlyssides et al. 1999). Unpleasant odours are emitted during its storage due to possible anaerobic conditions, thus restricting its use. Furthermore, strict environmental legislation on biomass incineration sets another obstacle on OSWR burning. Consequently, at least 25% of the produced solid residues remain unused and hence new uses of this biomass are sought (Azbar et al. 2004; Vlyssides et al. 2008).

This paper proposes an integrated method for the treatment of olive mill wastewater and olive stone wooden residues by co-composting. Composting is an aerobic procedure for organic solid waste stabilization. During the composting the easy biodegradable organic matter, under exothermic aerobic bioreactions, is stabilized producing biomass, carbon dioxide and thermal energy (Kaiser 1996; Mason and Milke 2005; Paraskeva and Diamadopoulos 2006). In order to keep temperature around the optimum thermophilic range of 60–70°C, air is supplied as a cooling mean (Vlyssides et al. 1996). The applied air also transfers the proper amounts of oxygen for the biological respiration needs. So the air for cooling needs is the controlling parameter for oxygen supply. Other parameters that control the composting procedure are: the temperature of the process, the moisture of the bulking material and the nutrients (nitrogen and phosphorous) (Haug 1980). For a continuous and stable process, the removed carbon by degradation as well as the moisture removed by evaporation must be replaced (Kaiser 1996). In this paper, the OSWR were used as substrate (bulking material) and the OMWW as a supplier of moisture, carbon and nutrients. The concept of this approach is based on the continuous replacement of the aerobically degraded organic carbon and the evaporated moisture, that are lost during the composting of the organic solid material, by the organic carbon and water content of the wastewater respectively. In a typical olive mill plant the ratio of OMWW to OSWR produced is approximately 2.5 v/w (Niaounakis and Halvadakis 2004). Thus, the aim of this paper is to specify such operational conditions of co-composting process that would result in a ratio of total olive mill wastewater consumption to olive stone wooden residue used as bulking material equal or greater than 2.5. The biological efficiency of this process is also set under investigation.

Materials and methods

Apparatus

In order to examine the feasibility of the proposed method a fully computerised laboratory apparatus was designed and constructed (Fig. 1). The most important critical parameters for the growth of microorganisms and bioreactions during co-composting that will be taken into account during the experimental procedure are the oxygen demand, the moisture and the temperature.

The composter consists of a cylindrical double layered metal autoclave (10.058 l active volume) with a six wing anchor agitator. The gas phase homogenization is achieved by continuous gas recycling. A O_2/CO_2 gas analyzer is included in the gas-recycling line. During the bioreactions, oxygen is consumed and carbon dioxide is produced, thus the oxygen partial pressure tends to lower. The control of this parameter is performed by feeding dry atmospheric air according to the O_2/CO_2 gas analyzer's indications. Through a simple and fully handlable system the bioreactor may operate under constant pressure. The exhaust gasses pass through a Wet Gas Meter (WGM) and their moisture is removed by a water trap. Thus, the carbon dioxide and the moisture of the gas phase can be estimated.

The wastewater is stored in a refrigerator (4°C) in order to avoid any undesirable biological degradation. The wastewater is added in the bioreactor in order to maintain constant the moisture of the system. For each time interval, the PLC calculates the wastewater addition from the water mass balance. Water is lost with the evaporating gases and is produced from the biological reactions. The latter is directly proportional to the CO_2 produced during the process. It was considered that 1 mol H_2O is produced per mol CO_2 produced (Kaiser 1996). The solid residue during co-composting process is hydrolysed and consequently its total mass is reduced. Assuming that the hydrolysis of OSWR followed a first order kinetics, the hydrolysis constant was

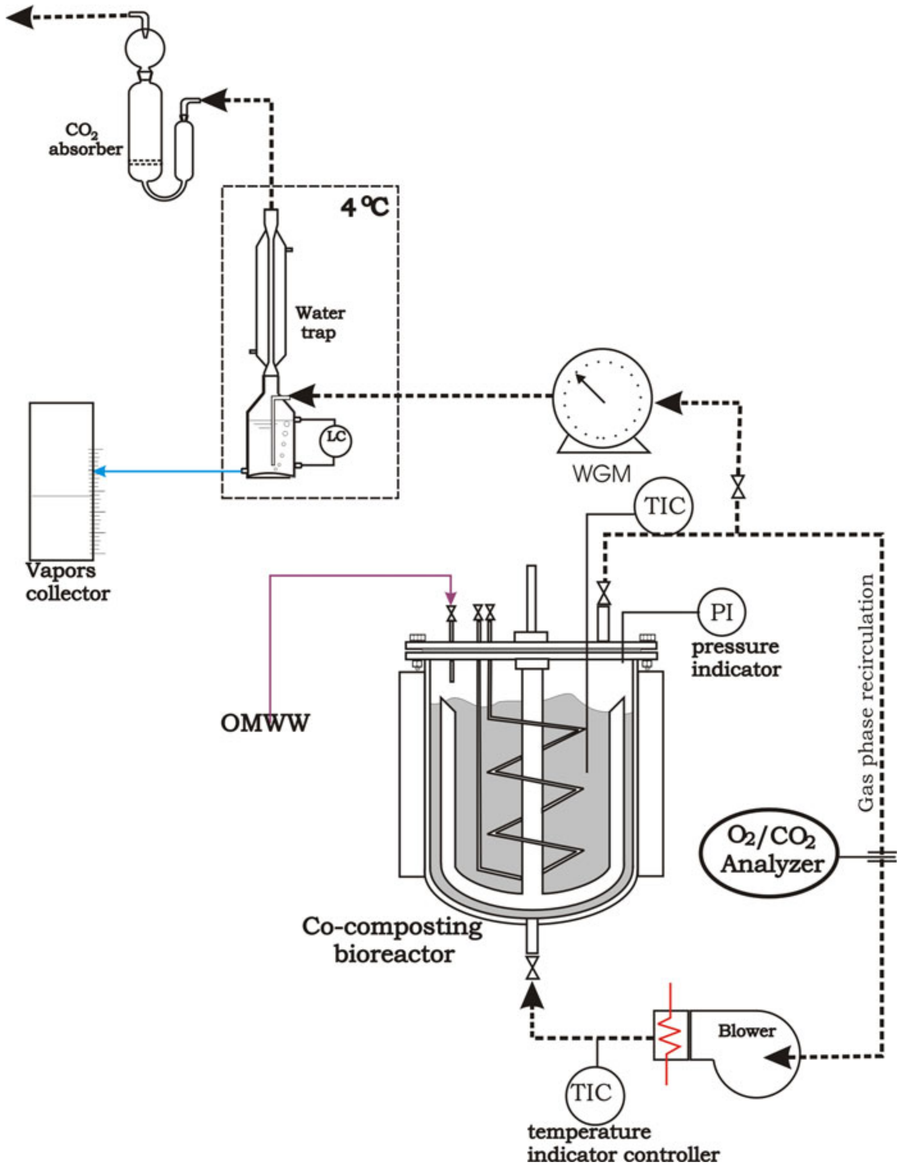


Fig. 1 Experimental apparatus of co-composting

estimated (Vlyssides et al. 2009). By this way, it is possible for the PLC to estimate at each time interval the mass of OSWR present in the composter and thus calculate the necessary wastewater addition in order to maintain the moisture of the system constant.

The temperature inside the bioreactor is fully controlled. The heating of the reactor is achieved by the heating of the recycled gas. The temperature of the recycled air is controlled under 80°C. The bioreactor's cooling is achieved by circulation of cool (4°C) water between the layers of the reactor.

Raw materials

Olive stone wooden residue

Olive stone wooden residue (OSWR) was used as bulking material for the co-composting process. The OSWR samples were taken from an olive mill plant in Crete, Greece. It is worth mentioning that the solid residues composition varies from area to area and from year to year depending on the climatological conditions, cultivation, etc. The main physical and

Table 1 Physical and chemical characteristics of OSWR

Parameter	
Moisture,%	13.50 ± 0.52
Fats and oils,% of TS (total solids)	1.85 ± 0.69
Nitrogen content substances,% of TS	7.39 ± 0.037
Total sugars, % of TS	2.13 ± 0.025
Cellulose, % of TS	37.39 ± 0.438
Hemicellulose, % of TS	17.04 ± 0.942
Ash, % of TS	3.66 ± 0.225
Ether extraction substances, % of TS	8.61 ± 0.035
Lignin, %of TS	21.97 ± 0.45
Kjendahl nitrogen content, % of TS	1.093 ± 0.015
Phosphorous content as P_2O_5, % of TS	0.113 ± 0.008
Potassium content as K_2O, % of TS	0.83 ± 0.07
Calcium content as CaO, % of TS	0.95 ± 0.092
Total carbon content, % of TS	56.13 ± 4.48
C/N ratio	51.34 ± 4.52
C/P ratio	1.137 ± 99.11

chemical characteristics of the OSWR used that were analysed according to standard methods (APHA 1985), are presented in Table 1. The solid residues were classified by sieving in order to estimate the initial particle size distribution.

Olive mill wastewater

The water needs of co-composting process were fulfilled by olive mill wastewater (OMWW) produced in the same olive mill plant in Crete, Greece. The composition of the OMWW, used for all experiments is shown in Table 2. All parameters were analysed according to standard methods (APHA 1985).

Experimental procedure

The experiments were conducted in a semi-batch mode; the OSWR (6 kg) was loaded in the beginning of each experiment and the addition of wastewater was performed according to the water needs. In each experiment, the temperature, oxygen partial pressure and moisture were kept constant to a predetermined set point. It was possible to fully control these parameters due to the appropriate design of the apparatus and its automations. During the experiments various parameters were continuously recorded. Parameters such as wastewater addition, effluent gas flow rate and

Table 2 Composition of Olive Mill Wastewater (OMW) used in experiments

Characteristics	Value (mg l^{-1})
pH	4.2
BOD_5	25,850
COD	80,250
Total suspended solids	4,580
Volatile suspended solids	4,024
Total phosphorus	870
Total Kjeldahl nitrogen	1,150
Total phenolic compounds (TPC)	14,250

composition may give useful information for the progress of co-composting process. The critical parameters for the applicability of the co-composting process in the scale of a single olive mill are the ratio of the total wastewater addition to the wooden residue consumption and the efficiency of the co-composting process. The term efficiency of co-composting process is defined as the carbon dioxide produced by the biological reactions to the total carbon available (both from wastewater and bulking material) to the microorganisms.

The composting of various feedstocks under controlled conditions has been studied thoroughly in the literature (Cronjé et al. 2003; Gomes and Pereira 2008; Liang et al. 2003; Petric and Selimbašić 2008; Richard et al. 2006; Wang et al. 2006). Form this literature survey, the parameters that were selected to be examined were temperature and oxygen partial pressure, given that the composting mixture was specified. In this context, an experimental plan was designed. The examined values range was selected ranging from 55 to 72°C for temperature and from 10 to 17% for oxygen partial pressure. The moisture was kept constant at 40%. 8 series of experiments were conducted under constant oxygen partial pressure (10–17%, step 1%) and temperatures 55, 58, 60, 62, 65, 68, 70, 72°C. The end of each experiment was set at 20 days (480 h). At the end of each experiment the bulking material (OSWR) was dried, weighed and classified.

Results and discussion

During the 20-days period of each experiment, the carbon dioxide produced and wastewater added was recorded on an hourly basis. For example, the results that derived during the experiment conducted under 65°C and 12% oxygen partial pressure are presented in Figs. 2 and 3. Figure 2 presents the CO_2 production during this experiment. The data of this Figure have derived from the recordings of the O_2/CO_2 gas analyzer and reflect the progress of the biological reactions. The peaks of the instant carbon dioxide production curve correspond to the maximum activity of the microorganisms. According to Hachicha et al. (2008), during the first stage of the process, microorganisms metabolized the soluble organic matter provided by easily biodegradable carbon. As a result, this aerobic degradation principally generated heat, biomass and carbon dioxide. The first peak of Fig. 2 can thus be attributed to the degradation of the soluble biodegradable carbon of the co-composting mixture. In this paper, the composting process is conducted under fully controllable conditions. Thus, the effect of crucial operational parameters such as oxygen supply and temperature, is eliminated. Consequently, this co-composting process cannot be directly compared to a conventional system with windrows (Hachicha et al. 2008; Haug 1980). After the consumption of the soluble biodegradable carbon, an adaptation phase of the mixed culture of the

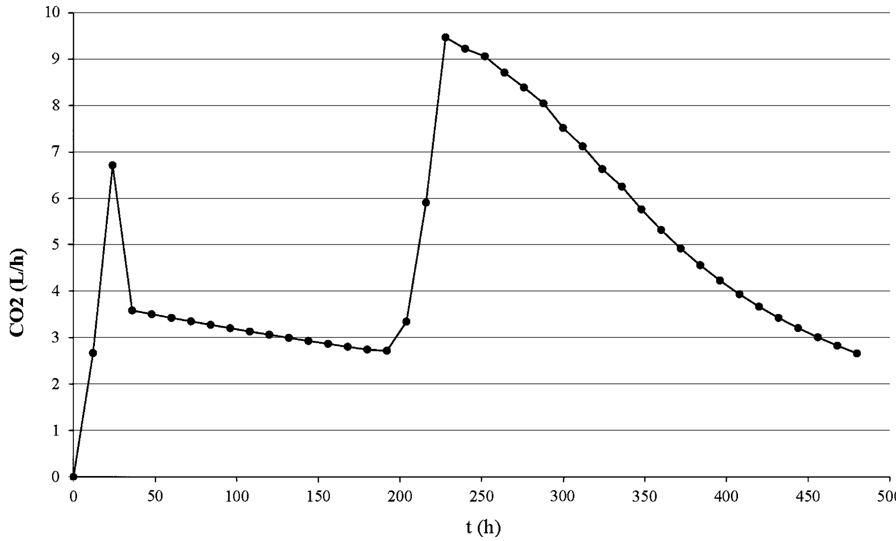


Fig. 2 Typical time variation of carbon dioxide production during co-composting at 65°C, 12% oxygen partial pressure and 40% moisture

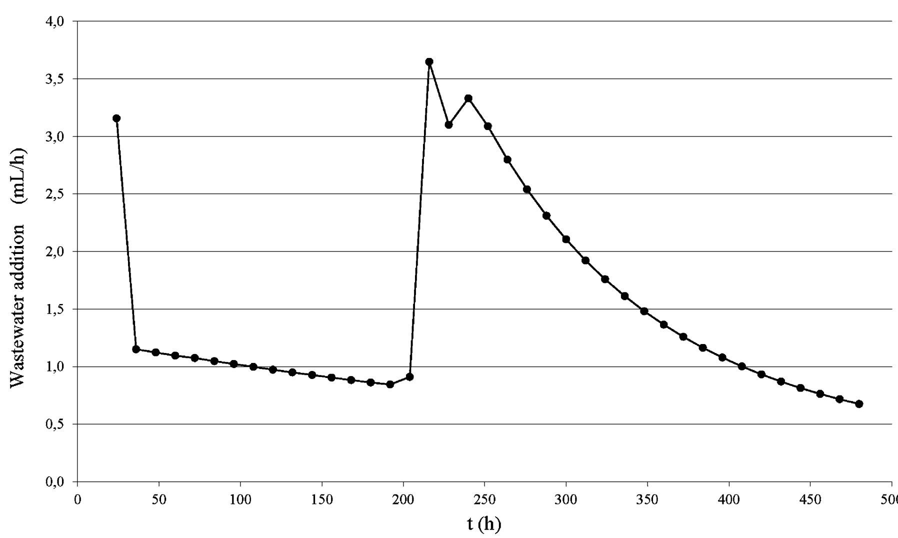


Fig. 3 Typical time variation of wastewater addition during co-composting at 65°C, 12% oxygen partial pressure and 40% moisture

microorganisms as well as a reduction of inhibitory factors such as phenolic compounds follows, resulting to the second peak of Fig. 2.

As it has been mentioned previously in the paper, the necessary wastewater addition was estimated by the water mass balance as well as the hydrolysis of OSWR. The wastewater finally added to the system was recorded by the PLC. The data of Fig. 3 are the results of the same experiment (65°C and 12% oxygen partial pressure). Figure 3 presents the instant water needs given that at the beginning of the experiment ($t = 0$) the co-composting mixture had the desired moisture (40%) by the appropriate wastewater addition. It is obvious that Fig. 3 follows the same pattern as Fig. 2. According to literature referring to composting models (Haug 1980; Kaiser 1996; Mason and Milke 2005), the bioreactive evolution of heat is directly proportional to the evolution of carbon dioxide. Thus, the water evaporation and the subsequent wastewater addition are proportional to CO_2 production, which is the reason why the patterns of Figs. 2 and 3 are the same.

Figure 4 illustrates the variation of the size distribution of the bulking material at the beginning and at the end of the experiment. The bulking material was sieved and thus classified at the end of the experiment as well. It is obvious that by the progress of co-composting the curve was displaced to lower diameters resulting in a total weight decrease. This was the case for all experiments. This can be attributed to the hydrolysis of OSWR.

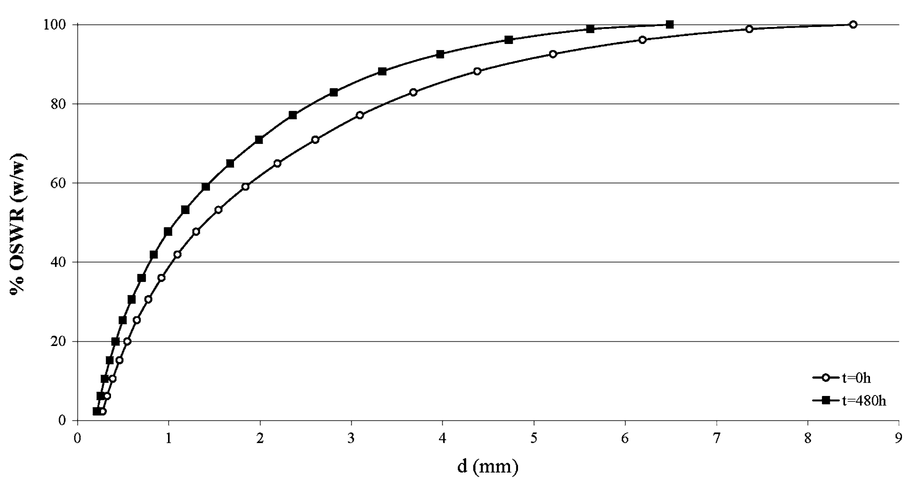
The final product—compost that was produced after a 2-month maturation period had the characteristics presented in Table 3. From the processing of the experimental results, in this case, the ratio of total wastewater added to the olive stone wooden residue loss at the end of the 20-day composting period was calculated equal to 1.47 and the corresponding co-composting efficiency 0.45.

After the completion of all experiments, a series of data had been collected. For each experimental condition examined (temperature and partial pressure), a ratio of total wastewater added to olive stone wooden residue loss at the end of the 20-day composting period as well as a co-composting efficiency had derived. Figure 5 presents the correlations of the OMWW/OSWR ratio at the end of the 20-day composting period with oxygen partial pressure under different constant temperatures. It is evident that there are strong exponential correlations between the parameters mentioned above. These correlations are presented in Table 4. From Fig. 5, it is obvious that under a constant temperature, the increase at the supply of air (increase of oxygen partial pressure) would result in an exponential increase of the wastewater consumption per kg

Table 3 Final product characteristics

Parameter	Value (% dry basis)
Organic matter	60–85
Nitrogen	1–2
Phosphorous	0.035–0.065
Humic substances	5–15
Ash	15–40
Moisture	30–40

Fig. 4 % weight distribution of fractions with different particle diameters



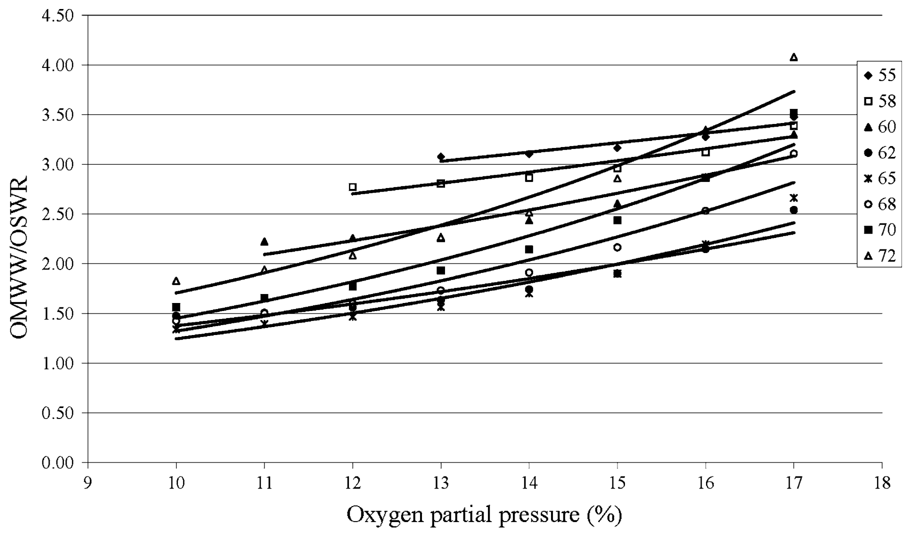


Fig. 5 Correlation of the OMWW/OSWR ratio after a 20-day composting with oxygen partial pressure under constant temperatures and 40% moisture

Table 4 Exponential equations correlating the OMWW/OSWR ratio with oxygen partial pressure (10–17%) under different constant temperatures and 40% moisture

Temperature (°C)	Equation	Correlation coefficient R^2
55	$\frac{OMWW}{OSWR} = 2.0578e^{0.0298 \cdot P}$	0.9027
58	$\frac{OMWW}{OSWR} = 1.698e^{0.0388 \cdot P}$	0.9352
60	$\frac{OMWW}{OSWR} = 1.0275e^{0.0646 \cdot P}$	0.9199
62	$\frac{OMWW}{OSWR} = 0.6559e^{0.0741 \cdot P}$	0.9292
65	$\frac{OMWW}{OSWR} = 0.4841e^{0.0945 \cdot P}$	0.9355
68	$\frac{OMWW}{OSWR} = 0.449e^{0.108 \cdot P}$	0.9502
70	$\frac{OMWW}{OSWR} = 0.4695e^{0.1129 \cdot P}$	0.9568
72	$\frac{OMWW}{OSWR} = 0.5575e^{0.1119 \cdot P}$	0.9607

of bulking material for a composting period of 20 days. This increase can be attributed to two factors:

1. the increase of biological activity due to the fulfilment of oxygen requirements of the microbial community.
2. the physical factor of water evaporation to the gas phase.

Figure 6 presents the influence of both temperature and oxygen partial pressure on the OMWW/OSWR ratio after 20 days of composting. Taking into consideration the fact that the ratio of olive mill wastewater to olive stone wooden residue produced in a typical olive mill is approximately 2.5, an operational region of the co-composting process could be defined (Fig. 6).

Figure 7 presents the correlation of the co-composting efficiency with temperature under different constant oxygen partial pressures for 20 days of composting. It is obvious that these curves follow a sigmoid pattern and that the co-composting efficiency is almost independent of pressure. The sigmoid correlation ($R^2 = 0.9959$) that best fitted these results is the following:

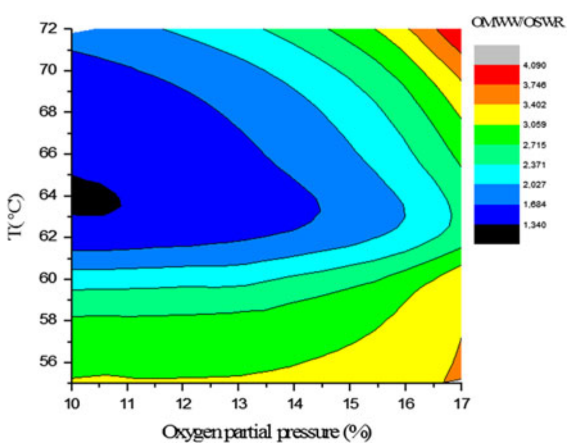


Fig. 6 Effect of temperature and oxygen partial pressure on the OMWW/OSWR ratio after a 20-day composting

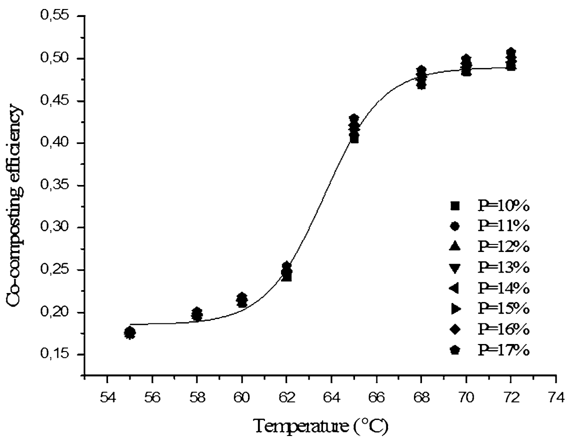


Fig. 7 Effect of temperature and oxygen partial pressure on the co-composting efficiency after a 20-day composting

$$\text{co-composting efficiency} = 0.18579 - \frac{0.30673}{1 + 10^{20.9411 - 0.32891 \cdot T}}$$

Based on the equation above as well as Fig. 7, the co-composting efficiency after 20 days of composting reaches a maximum plateau of approximately 0.50 at 68°C.

Conclusions

Taking into consideration all the experimental data mentioned above, it can be concluded that the method of co-composting can be successfully used in order to both consume the olive-mill wastewater as well as to stabilize the olive stone wooden residue. In other words the proposed technology achieves to treat successfully all wastes produced during olive oil production.

A sigmoid correlation of co-composting efficiency with temperature derived. This equation could be a useful tool for the estimation of an operational temperature for an efficient 20-day co-composting. It was proved that the efficiency of the co-composting process was independent of the oxygen partial pressure. This conclusion indicates that in the working system the oxygen was not the limiting factor for the biological reactions and thus the oxygen partial pressure does not influence the efficiency value. The supplied air apart from meeting, the oxygen needs for the biological reactions, is necessary for cooling purposes and the enhancement of the wastewater evaporation, which is one of the main goals of this system. Given the sigmoid pattern of the curve, co-composting efficiency reaches its maximum value at 68°C. Any increase above this temperature does not result in a subsequent increase of efficiency, but simply to unnecessary energy cost.

If the wastewater treatment is set as the first priority in this treatment scheme, then the ratio of the wastewater addition to the wooden residue consumption should be at least 2.5. Consequently, from Fig. 6 and considering a 20-day composting process, a broad operational region of temperature and oxygen partial pressure was defined.

In order to render co-composting an integrated treatment scheme for olive mill plants both parameters, OMWW/OSWR ratio and co-composting efficiency, should be taken under consideration. Thus, the optimum conditions proved to be 68°C and 16–17% oxygen partial pressure for 20 days co-composting.

Conclusively, the co-composting process could lead the olive oil production to sustainability, by producing a fertiliser ready to return to olive tree cultivations from the liquid and solid wastes of the production process. This study rendered encouraging results for the applicability of the proposed treatment scheme, but there are still many aspects such as economical, method up-scale, etc. that further investigated.

References

APHA (1985) Standard method for the examination of water and wastewater, 16th edn. American Public Health Association, Washington

Azbar N, Bayram A, Filibeli A, Muezzinoglu A, Sengul F, Ozer A (2004) A review of waste management options in olive oil production. Crit Rev Environ Sci Technol 34:209–247

Cronjé A, Turner C, Williams A, Barker A, Guy S (2003) Composting under controlled conditions. Environ Technol 24(10):1221–1234

Di Serio MG, Lanza B, Mucciarella MR, Russi F, Iannucci E, Marfisi P, Madeo A (2008) Effects of olive mill wastewater spreading on the physico-chemical and microbiological characteristics of soil. Int Biodeter Biodegr 62(4): 403–407

Gomes AP, Pereira AF (2008) Mathematical modelling of a composting process, and validation with experimental data. Waste Manag Res 26(3):276–287

Hachicha S, Sallemi F, Medhioub K, Hachicha R, Ammar E (2008) Quality assessment of composts prepared with olive mill wastewater and agricultural wastes. Waste Manag 28:2593–2603

Haug TR (1980) Compost engineering: principles and practice. Technomic Publishing Co, Inc., Lancaster

Jarboui R, Sellami F, Kharroubi A, Gharsallah N, Ammar E (2008) Olive mill wastewater stabilization in open-air ponds: impact on clay-sandy soil. Bioresour Technol 99(16):7699–7708

Kaiser J (1996) Modelling composting as a microbial ecosystem: a simulation approach. Ecol Model 91(1–3):25–37

Liang C, Das KC, McClendon RW (2003) The influence of temperature and moisture contents regimes on the aerobic microbial activity of a biosolids composting blend. Bioresour Technol 86(2):131–137

Mason IG, Milke MW (2005) Physical modelling of the composting environment: a review. Part 1: reactor systems. Waste Manag 25(5):481–500

Niaounakis M, Halvadakis CP (2004) Olive-mill waste management: literature review and patent survey. Typothito George Dardanos Publications, Athens

Paraskeva P, Diamadopoulos E (2006) Technologies for olive mill wastewater (OMW) treatment: a review. J Chem Technol Biotechnol 81(9):1475–1485

Petric I, Selimbašić V (2008) Development and validation of mathematical model for aerobic composting process. Chem Eng J 139(2):304–317

Richard TL, Walker LP, Gossett JM (2006) Effects of oxygen on aerobic solid-state biodegradation kinetics. Biotechnol Progress 22(1):60–69

Taralas G, Kontominas M (2006) Pyrolysis of solid residues commencing from the olive oil food industry for potential hydrogen production. J Anal Appl Pyrol 76(1–2):109–116

Vlyssides AG, Bouranis DL, Loizidou M, Karvouni G (1996) Study of a demonstration plant for the co-composting of olive-oil-processing wastewater and solid residue. Bioresour Technol 56(2–3):187–193

Vlyssides AG, Loizidou M, Zorpas AA (1999) Characteristics of solid residues from olive oil processing as bulking material for co-composting with industrial wastewaters. J Environ Sci Health A 34(3):737–748

Vlyssides A, Barampouti EM, Mai S (2008) Physical characteristics of olive stone wooden residues: possible bulking material for composting process. Biodegradation 19(2):209–214

Vlyssides A, Barampouti EM, Mai S (2009) An integrated mathematical model for co-composting of agricultural solid wastes with industrial wastewater. Bioresour Technol 100(20):4797–4806

Wang W-D, Wang X-F, Liu J-B, Gao L-J, Li X-M, Cui Z-J (2006) Effect of oxygen concentrations on process of composting. Huanjing Kexue/Environ Sci 27(3):594–598