Energy and geo-environmental applications for Olive Mill Wastes. A review*

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ABSTRACT: Olive oil production wastes are one of the major environmental problems to all olive producing countries, especially at the Mediterranean basin. Several attempts have been proposed over the years for the reduction of the wastes' polluting load before they reach the natural waters. The majority of the methods require a centralized olive oil production which is not the case in most of the countries. Moreover the lack of economic motive for the management of wastes has set their treatment in half-measures, like evaporating ponds or neutralization. The content of the wastes in several inorganic substances but mostly their high organic load consisting from compounds with pleiotropic actions has led the research community in utilizing treatment methods for the valorization of the wastes. In this mini-review are presented a) the potential of energy production from the wastes as bio-gas or fuel blocks, b) the recovery of organic compounds with high biological activity using raw materials with low cost, abundant in the oil producing countries and c) in the utilization of the wastes as water substitutes in industry, like brick manufacturing. These methods and the possible combinations of them can transform the wastes from a by-product and polluting agent to a useful starting material for various applications, rendering their management cost-effective.
Key-words: *Olive mill wastes, olive pomace, adsorption, bentonite, zeolite, sepiolite, heavy metal biosorption, biologically active compounds*.

ΠΕΡΙΛΗΨΗ: Τα απόβλητα που δημιουργούνται κατά τη διαδικασία παραγωγής του ελαιολάδου αποτελούν σημαντικότατο περιβαλλοντολογικό πρόβλημα σε όλες τις ελαιοπαραγωγούς χώρες, ειδικότερα στις Μεσογειακές. Τα τελευταία χρόνια έχουν προταθεί αρκετές μέθοδοι για τη μείωση του ρυπαντικού φορτίου των αποβλήτων, πριν αυτά καταλήξουν στους υδάτινους πόρους. Η πλειονότητα των μεθόδων απαιτεί επικεντρωμένη παραγωγή του ελαιολάδου, το οποίο δεν είναι κανόνας στις περισσότερες εκ των ελαιοπαραγωγικών χωρών. Επιπλέον η έλλειψη οικονομικών κινήτρων για τη διαχείριση των αποβλήτων έχει οδηγήσει σε χρήση ημίμετρων, όπως οι δεξαμενές εξάτμισης ή την απλή εξουδετέρωση των αποβλήτων. Η περιεκτικότητα των αποβλήτων σε αρκετά ανόργανα συστατικά αλλά κυρίως το υψηλό οργανικό τους φορτίο σε ενώσεις που εμφανίζουν πλειοτροπικές δράσεις έχουν οδηγήσει την επιστημονική κοινότητα στην αναζήτηση μεθόδων αξιοποίησης τους. Σε αυτό το άρθρο παρουσιάζεται μια ανασκόπηση: α) της δυνατότητας παραγωγής ενέργειας από τα απόβλητα με τη μορφή βιοκαυσίμων και καύσιμων πλίνθων, β) της ανάκτησης οργανικών μορίων με υψηλή βιολογική δραστικότητα με τη χρήση φθηνών προσροφητικών υλικών που υπάρχουν σε αφθονία σε όλες τις ελαιοπαραγωγούς χώρες και γ) της χρήσης των αποβλήτων σαν υποκατάστατο του νερού στη βιομηχανία, όπως στη παραγωγή κεραμευτικών προϊόντων. Αυτές οι μέθοδοι και κυρίως οι πιθανοί συνδυασμοί τους μπορούν να μετατρέψουν τα απόβλητα από ένα ρυπαντή σε μια χρησιμότατη πρώτη ύλη για διάφορες εφαρμογές, καθιστώντας έτσι τη διαχείρισή τους οικονομικώς βιώσιμη.
Λέξεις-κλειδιά: *Απόβλητα ελαιουργίας, ελαιοπυρήνας, προσρόφηση, μπεντονίτες, ζεόλιθοι, σεπιόλιθοι, βιο-προσρόφηση βαρέων μετάλλων, βιολογικά δραστικές ενώσεις.*

INTRODUCTION

Olive oil production is traditionally associated with the Mediterranean countries, as indicated by the statistical data. The countries around the Mediterranean Basin hold the 98% of the Olive trees globally, covering an area of 5,163,000 ha, while deriving over 93% of the total olive oil produced. The global olive oil production for 2010 is estimated to be 2,881,500 metric tons. The European Union countries produce the 78.5% of the total olive oil which stands for an average production of 2,136,000 tons. The major olive producing country is Spain with 1,200,000 tons followed by Italy with 540,000 tons, Greece with 348,000 tons, Portugal with 50,000 tons and finally France and Cyprus with 5,000 tons. Other non EU Mediterranean olive producing countries are Tunisia with 185,000 tons, Syria with 128,000 tons, Turkey with 117,000 tons and Morocco with 78,300 tons (Niaounakis & Halvadakis, 2006; IOC, 2010).

During the olive fruit harvesting large quantities of solid wastes are produced. It is estimated that 25 Kg of leaves and branches are removed from each olive tree annually, in order to ameliorate the next year's production by removing the non-productive parts of the plant. These parts of the plant are usually burned by the producers due to the fact that there is no other cost effective and environmental friendly management.

Through the ages the olive oil extraction procedures have evolved from the traditional pressing system to the more effective continuous three phase centrifugal system (3PCS), which in turn is currently being substituted by the modern two phase centrifugal system (2PCS).

The pressing system is the extraction system used through the ages from the ancient times with minimal modifications

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and though its technology is long gone by, it is still used in some areas.

The 3PCS is named after the "three phases" that are generated during the extraction, the first being the olive oil, the second the olive pomace, also known as orujo, and the third the Olive Mill Waste Waters (OMWW) also known as alpechin. The 3PCS has the advantage of quintupling the daily olive oil production compared to the traditional pressing system, although it also uses 50% more water during the process consequently producing as much as twice more OMWW. The OMWW is consisted of i) the Vegetable Water (VW), meaning the water present in the olive fruit, ii) the olive fruit washing water before its crushing, iii) the processing hot water added to the various stages of the olive oil extraction, which constitutes the largest portion of the OMWW and iv) the water from the washing of equipment and facilities.

The OMWW traditionally were dumped from the olive mills to the nearest creek or river, disrupting the local and the shoreline at the river's end, even from the ancient times (Hadjisavvas, 1992). As a result many rivers in Spain (Gualdalquivir), Italy (Vomano, Saline, Foro) and Morocco (Sebu, Fez) have become anoxic (Di Giovacchino *et al.*, 1976; Cabrera *et al.*, 1984; Zenjari & Nejmeddine, 2001). It is estimated that in Mediterranean basin alone end up 10-12×10^6 m^3 of wastes every year (Cabrera *et al.*, 1996).

The olive pomace which is also a by-product of the extraction is usually submitted to a second oil extraction with organic solvents, mostly hexane, producing crude seed oil. After this second extraction the exhausted olive cake can be used as fuel from the extraction facility for the drying of the cake before its subsequent extraction (Vlyssides *et al.*, 1998).

The annotation OMWW will be used for the rest of this paper to indicate the wastes produced from three phase centrifugal system whereas the two phase wastes will be mentioned as Olive Mill Wastes (OMW), while Olive Oil Wastes (OOW) will be used when mentioned to both waste types.

The 2PCS was introduced in the early 90's and is considered more "environmental friendly" in comparison to the 3PCS because it doesn't need the addition of water for the oil extraction reducing the water consumption by 75%. Through the use of a more effective centrifugation system it produces only two phases the first being the olive oil, while the second being a mixture of the pomace and the water contained in the olive fruit, producing a semi-solid very wet olive cake (Niaounakis & Halvadakis, 2006; Roig *et al.*, 2006). Based on this fact all EU olive oil producers are obliged to replace the 3PCS with the 2PCS.

Despite the environmental benefits of OMW, the physical and chemical properties of the wastes are the major drawback for the subsequent seed oil extraction method. The high humidity of the OMW in conjunction with its high concentration in carbohydrates makes the waste to stick to the drying furnaces blocking the hot air stream, increasing pressure and temperature subsequently increasing the possibility of an explosion or a fire in the seed oil extraction plant.

Several drying procedures have been tried for the reduction of this high humidity but failed. Their major problem was the high temperature and time needed for the drying which increased the production cost and reduced the nutritional value of the seed oil. The produced seed oil lost many of the volatile substances present in olive oil, while other substances were degraded producing hazardous or carcinogenetic substances like benzopyrenes (Arjona *et al.*, 1999). One can understand that the use of 2PCS is not a panacea because in fact it simply transports the problem from the olive mills to the seed oil extraction plants that also have trouble managing it.

Aside the composition and the physicochemical attribute of the wastes one of the most potent drawbacks for their efficient and environmental friendly disposal is the fact that olive mills are usually small family business widely scattered in the olive producing countries. It is estimated that in Greece are operating more than 2,000 mills (Manios, 2004). The scattering of the mills and the short harvesting season makes the creation of a centralized collection and processing plant impossible while the small scale of the mills is a negative for the installation of autonomous processing plant, which requires initially a high investment and operational cost.

EVERY DAY PRACTICE FOR THE WASTES MANAGEMENT IN THE MAJOR OIL PRODUCING COUNTRIES

Spain

Spain has forbidden by law from 1982 the river disposal of OMWW, so 100 evaporation ponds were constructed in order to reduce the volume of the wastes during the summer months. The evaporation ponds improved water quality but the mal odor from the oxidation products of the organic matter annoyed the ambient population. The replacement of the 3PCS by the 2PCS reduced the waste's volume so many ponds shut down (Alburquerque *et al.*, 2004). The management of OMW, as mentioned above, is also troubled and a lot of effort has been put on finding a solution.

Italy

In Italy there are over 5,000 olive mills that operate using the traditional pressing system. Italy is the only oil producing country with a special legislation for the disposal and recycling of the wastes but with the flaw that the inspections must be conducted by the regional and provisional authorities that do not keep exact records concerning the dates and places of the disposal.

Greece

In Greece in 2002 there were 2786 olive mills, the 70% of them being 3PCS, while the rest used the pressing system or a combination of the two methods. The olive mill owners hesitated to upgrade to the 2PCS because the seed oil factories refused to take the complex mixture of OMW for extraction (Vlyssides *et al.*, 2003). Although this situation has changed over the past few years and following the EU direction all olive mills have to change to 2PCS and almost all have complied.

Aside this direction the management of the wastes is not subjected to a specific regulation. The oil producing municipalities regulate autonomously the waste management, based on the experience from the past years or whether it is participating to an environmental program or not. In general, at a percentage of 58%, the wastes are neutralized using $Ca(OH)_2$ at the end point of the mills pipeline, before reaching the natural waters. Only in the Prefecture of Chios Island evaporation ponds similar to the Spanish have been constructed and are currently collecting the wastes from twelve of the fourteen mills on the island. On Samos island a real scale waste water management technique is being evaluated. The technique includes an initial pretreatment and fractionization of the wastes by natural sedimentation and separate management of the fractions formed (Georgacakis & Christopoulou, 2002).

GENERAL DATA FOR THE OOW

The composition of olive wastes is extremely variable and depends mostly from the olive oil extraction procedure and the VW which in turn depends from the a) olive tree variety, b) the olive fruit's maturity and harvesting time, c) the climatic conditions, d) the cultivation soil and e) the presence of pesticides and fertilizers. Specifically for the phenolic content of the wastes intercultivar and harvesting time variation accounted for a 2-5-fold change in the total phenol and antioxidant capacity, while levels of individual biophenols experienced up to 50-fold change. The phenol content and antioxidant capacity of OMW significantly changed between seasons with different variation patterns for different cultivars (Obied *et al.*, 2008).

The olive oil extraction procedure contains many different parameters, the major being whether the olive mill uses pressing system, 2PCS or 3PCS. The 2PCS as mentioned above produces a watery cake, a mixture of solid and liquid wastes. The other two extraction systems, namely the pressing system and the 3PCS, separate the solids from the liquid wastes.

The extraction of 1000 Kg olives by 3PCS yields 210 Kg olive oil, 550 Kg olive pomace and 1-1.6 m^3 OMWW, whereas the same amount of olives with the 2PCS yields 200 Kg of oil, 800 Kg of OMW and 0.2 m^3 of waste water for the washing of oil (Alburquerque *et al.*, 2004).

The extensive use of water in the 3PCS compared to the pressing system and 2PCS leads to a deceptive dilution of its substances and its pollutant load, a phenomenon which is negated by the huge volume of the wastes. The composition of the wastes can vary even between olive mills from the same region because of a difference in the water's temperature during the extraction or an insufficient separation of the olive leaves from the olive fruits. The general characteristics of OMWW are 1) the intense violet-black color, 2) the strong oily smell, 3) high values of COD and BOD_5 (can reach up to 220 g/L and 100 g/L respectively, rendering them among the "heaviest" biomechanical wastes), 4) the slightly acidic pH (3-6), 5) high phenol/polyphenol content, 6) high content of solid matter (Vasquez-Roncero *et al.*, 1974; Capasso, 1997).

The aforementioned facts make the exact characterization of the OOW's composition almost impossible. The only reliable results can only be collected from the statistical analysis of the numerous bibliographical data. The results from such meta-analysis studies for pressing system and 3PCS are presented in Table 1 (Di Giovacchino *et al.*, 1988) along with original data for 2PCS presented in Table 2 (Alburquerque *et al.*, 2004).

But even this type of statistical meta-analysis has limited usefulness because in most of the cases the research papers are missing several significant data like the type and origin of the wastes or the olive plant species that were extracted from (Tsonis *et al.*, 1989).

ENVIRONMENTAL EFFECTS OF OOW

Effects on soil

The application of two different OMWW doses on clay soil (high dose 600 mL/m^2, low dose 300 mL/m^2) for three years resulted in an increased organic carbon content of the soils and a reduction in the soils porosity, confirmed by mercury intrusion porosimetry and scanning electron microscopy (Cox *et al.*, 1997). The reduction in porosity is due to the reduction of the larger pores (>1 μm) even though the small pores (<0.1 μm) increased (Cox *et al.*, 1996; Zenjari & Nejmeddine, 2001). The reduction in porosity was attributed to the combined effect of the suspended and soluble organic matter and salts in OMWW and the solubilisation –insolubilisation of the soils carbonate minerals.

The application of OMWW on soils contributes to their stability through the binding actions of the polysaccharides present in the wastes, a stabilizing effect that lasts until the degradation of the compounds. Thus the OMWW can decrease soil erosion while improving oxygenation and hydraulic retention reducing evaporation losses due to the increased micro-porosity of the soil (Mellouli *et al.*, 1998; Colucci *et al.*, 2002).

Several research groups have studied the effect to the acidity of alkaline soils after direct OMWW application and have concluded that the mildly acidic character of the wastes temporarily acidifies the soil (the soil recovers its initial acidity after 15 days) (Della Monica *et al.*, 1978; Morisot & Tournier, 1986). This temporary acidification, especially from organic acids, is considered beneficial for alkaline soils because it increases the availability/absorption of phosphorous and other elements from the plants (Otani *et al.*, 1996;

Kirk *et al.*, 1999). Although a recent study contradicts the aforementioned results, suggesting that the application of OMW only temporarily increases extractable P from the soil, while reducing it over time (2 years) compared to control group (Mechri *et al.*, 2007). The writers list several possible explanations for the experimental results, the more probable being the reduction of the biomass of microorganisms by the phenolic compounds who exert phytotoxic and antibacterial effects (Ramos-Cormenzana *et al.*, 1996).

The application of OMWW on acidic grounds causes a minimum acidification of the soil for the first 100 days but a distinct increase in pH after that period (Marsilio *et al.*, 1989). A possible solution for this adverse effect is neutralizing the wastes with lime before their application on the soil.

The effect of OMWW on the sediments of rivers has also been studied by several researches, because is the commonest but also illegal way of disposing the wastes. The researchers studied the solubilisation of various heavy metals from river sediments after their treatment with various concentrations of OMWW at different pH values (Bejarano & Madrid, 1992). The results indicated that increased OMWW concentration and the lowest tested pH increased Pb solubility while Fe and Cu had the optimum solubility at the highest pH tested. The same research group in a later study showed that the solubility of Mn and Zn was unaffected by the wastes and there was also the possibility of Mn deposition from the wastes to the sediment. They concluded that with the exception of Fe and Mn all the other metal solubilisation is pH depended. Most metals with the exception of Cd and Zn solubilised at pH 5, at pH 4 solubilisation was detected for Ni, Cu, Mn and Pb, and at pH 3 only noticeable for Ni and Mn. (Bejarano & Madrid, 1996b; Bejarano & Madrid, 1996d; Bejarano & Madrid, 1996a; Bejarano & Madrid, 1996c).

Effects on Water

The disposal of OMWW to rivers has been forbidden by law first in Spain and later to all the other Mediterranean countries but even today a lot of oil farms are still disposing the wastes in natural waters, the sea or even the sewage system. In the case of dumping them into sewage system, the acidic nature of the wastes corrodes the pipelines creating extensive damages (Rozzi & Malpei, 1996).

The effects of the wastes on the water bodies are closely related to their concentration, composition and the seasonal emergence of the wastes.

The huge amount of wastes produced during the olive fruit harvesting and oil extraction period it has been reported to be 5-10 or even up to 25-80 times larger than the domestic sewage (Schmidt & Knobloch, 2000; Balice *et al.*, 1990).

The most visible effect is the discoloration of natural waters from the presence of tannins and polyphenols and their oxidation products which give a dark color and are very difficult to remove from the effluent (Hamdi, 1992).

The wastes as pointed out in Tables 1 & 2 contain large amounts of reductive sugars and phosphorous, both organic and inorganic, encouraging and accelerating the growth of microorganisms and algae. The uncontrollable proliferation of these organisms leads to the manifestation of eutrophication destroying the whole ecological balance, while depleting the soluble oxygen (huge BOD_5) leading to anoxic conditions. Furthermore it is an excellent substrate for the several pathogenic microorganisms that can affect the aquatic life but also humans that come in contact with them.

The phenols present in OMWW, except their oxidation products, can also have a direct effect on several fish species. The river fish *Gambusia affinis* and the crustacean *Daphnia magna* are severely intoxicated by phenol derivatives (40 mg/L) after 15 min exposure. This concentration can be achieved by the addition of 1 L OMWW in 100,000 L of water. Having in mind that some olive mills dump up to 5000 L/h the flow rate of the river should 100,000 L/s, in order to avoid the aforementioned toxic effects (Gonzalez *et al.*, 1994). The fractionation of OMWW samples using reverse osmosis techniques and the subsequent toxicity testing of the fractions collected, in algae and crustacean, attributed the

TABLE 1

Average results for characteristic parameters of pressing system.

OMWW.

Parameter	Pressing System	OMWW
pH	5.27	5.23
Dry matter (g/L)	129.7	61.1
Specific weight	1.049	1.020
Oil (g/L)	2.26	5.78
Reducing Sugars (g/L)	35.8	15.9
Total Phenol (g/L)	6.2	2.7
o-Diphenols (g/L)	4.8	2.0
Hydroxytyrosol (mg/L)	353	127
Ash (g/L)	20	6.4
COD (g O_2/L)	146.0	85.7
Organic N (mg/L)	544	404
Phosphorous (mg/L)	485	185
Na (mg/L)	110	36
K (mg/L)	2470	950
Ca (mg/L)	162	69
Mg (mg/L)	194	90
Fe (mg/L)	32.9	14.0
Cu (mg/L)	3.12	1.59
Zn (mg/L)	3.57	2.06
Mn (mg/L)	5.32	1,55
Ni (mg/L)	0.78	0.57
Co (mg/L)	0.43	0.18
Pb (mg/L)	1.05	0.42

TABLE 2
Average results for characteristic parameters of OMW (dry weight samples).

Parameter	Mean	Range	CV(%)
Moisture (% fresh weigth)	64	55.6-74.5	7.6
pH[a]	5.32	4.86-6.45	6.6
Electrical Conductivity [a] (dS/m)	3.42	0.88-4.76	33.9
Ash (g/Kg)	67.4	24.0-151.1	42.5
TOC (g/Kg)	519.8	495.0-539.2	2.8
C/N (ratio)	47.8	28.2-72.9	22.1
Total N (g/Kg)	11.4	7.0-18.4	4.5
P (g/Kg)	1.2	0.7-2.2	29.7
K (g/Kg)	19.8	7.7-29.7	34.2
Ca (g/Kg)	4.5	1.7-9.2	57.3
Mg (g/Kg)	1.7	0.7-3.8	58.7
Na (g/Kg)	0.8	0.5-1.6	36.6
Fe (mg/Kg)	614	78-1462	74.9
Cu (mg/Kg)	17	12-29	28.8
Mn (mg/Kg)	16	5-39	70.2
Zn (mg/Kg)	21	10-37	36.3

toxic effects to the low molecular weight compounds and especially to catechol and hydroxytyrosol (FIORENTINO *et al.*, 2003). Another research group has also studied the effect of 13 OOW samples from different regions of Portugal in microorganisms including *Daphnia magna* and has concluded that polyphenols have low toxicity but low biodegrability whereas tannins exhibit high toxicity and biodegrability (PAIXAO *et al.*, 1999).

The oil and lipids contained in the OOW can also have adverse effects in rivers/riverbanks and the surrounding lands by creating a thin film that blocks sunlight and oxygen transportation.

POSSIBLE USES OF OOW

Up to today many different methods for the management and reduction of the OOW polluting effect have been used. The majority of them concerning the co-composting with other wastes, chemical and biological-aerobic oxidation, anaerobic treatment, adsorption, or as substrates for edible microorganisms or microorganisms producing substances with high added value and several combinations of them (CROCE *et al.*, 1994; LOPEZ & RAMOS-CORMENZANA, 1996; ZOUARI, 1998; HOYOS *et al.*, 2002; AGGELIS *et al.*, 2003; FIORENTINO *et al.*, 2004; MANIOS, 2004; SABBAH *et al.*, 2005).

An excellent review on the environmental effects of the wastes as well as their possible treatment procedures has been extensively reviewed by (NIAOUNAKIS & HALVADAKIS, 2006). The focus of the present paper will be, as the title suggests, on the Energy and Geological applications of the wastes.

FUEL BLOCKS AND FUEL ADDITIVE

Solid olive residues have small compression strength and shattering index even when milled, attributes that deteriorate by the humidity of the residues, the optimum values being 200 mPa for pressure at 7.5% humidity. The addition of fibrous materials like paper wastes increases the shattering index without affecting the burning rate of the block, creating a good alternative to coal (YAMAN *et al.*, 2000; DALLY & MULLINGER, 2002). The addition of solid olive wastes in coal-paper wastes bricks increased water resistance of the fuel bricks (YAMAN *et al.*, 2001).

Olive cake is also a good alternative for coal. It has a caloric value of 12,500-21,000 KJ/Kg, with a sulfur content of about 0.05-0.1% (ATIMTAY & TOPAL, 2004). Olive cake is used for years at seed oil plants for heating and drying, as mentioned previously in text.

The OMW also have been tested in mixtures with other fuels, like coal, by several research groups. The addition of OMW at concentration of 10% in the fuel mixture reduced carbon monoxide emissions by 33%, even though the temperature dropped 25 °C (CLIFFE & PATUMSAWAD, 2001). The increment of OMW in the fuel mixture also reduces SO_2 and NO_x emissions, due to the dilution of the fuel-sulfur and higher volatile content respectively, but increases N_2O emissions by decreasing the flame temperature (ARMESTO *et al.*, 2003; SUKSANKRAISORN *et al.*, 2003).

The disadvantages of using the wastes as fuel are their high ash content, so wastes can only be used in plants that can handle ash and are equipped with dust collectors, and a post-treatment of the exhaust gases, increasing the cost of their use.

BIOGAS PRODUCTION

A lot of research groups have investigated the possibility of methane production from OMWW (BOARI *et al.*, 1984; ROZZI *et al.*, 1989; BORJA *et al.*, 1995; DALIS *et al.*, 1996; TEKIN & DALGIC, 2000). The two major problems that the researchers should overcome in order to utilize the methane production from OOW are i) the toxicity of the wastes that reduces the efficiency of the method and increases the investment and working cost, and ii) the need of large digesters in order to maintain a viable plant, thus excluding the small and medium scale mills from the project unless supported from the state (BONFANTI & LAZZARI, 1999).

The difficulties met in the anaerobic treatment can be lifted through the physicochemical pretreatment of the wastes for the removal of biorecalcitrant and/or inhibiting toxic substances (essentially lipids and polyphenols) as selectively as possible before anaerobic digestion. The experiments conducted showed that ultrafiltration, removed the majority of lipids and polyphenols but with poor selectivity, also removing large amounts of biodegradable COD. Centrifugation turned out to be preferable to sedimentation owing to smaller volumes of separated phase. The most interesting result of the study was obtained after the addition of $Ca(OH)_2$ (up to pH 6.5) and 15 g/L of bentonite, and then feeding the mixture to the biological treatment without providing an intermediate phase separation. The biodegradable matter adsorbed on the surface of bentonite and gradually released during the biotreatability test, thus allowing the same methane yield both in scarcely diluted (1:1.5) pretreated OMWW and in very diluted (1:12) untreated OMWW. Thus the authors suggest a continuous process application combining pretreatment (with $Ca(OH)_2$ and bentonite) and anaerobic digestion without intermediate phase separation (BECCARI *et al.*, 1999).

The Biochemical Methane Potential (BMP) of the wastes has been studied along with other agro-industrial wastes, namely poultry breeding and cheese making (cheese whey) wastes after anaerobic treatment. Through the BMP experiments the researchers tried to correlate the methane produced after microbial treatment with the initial Chemical Oxygen Demand (COD) of the samples. The experiments showed that methane was the 77±6% of the total biogas produced by the microbes and also that each litre of OMW produces 57.5 L of methane, rendering OMW the best substrate, from the three studied, for methane production (DEMIRER *et al.*, 2000).

However experiments from several research groups studying the co-digestion of the OOW with several other agro-industrial wastes including manure from several animal breeding facilities have been able to produce methane with adequate reduction of COD. The results showed that the high buffering capacity contained in manure, together with the content of several essential nutrients (like nitrogen that the OOW lack of), make it possible to degrade OOW without previous dilution, without addition of external alkalinity and without addition of an external nitrogen source (ANGELIDAKI & AHRING, 1997; GELEGENIS *et al.*, 2007; AZBAR *et al.*, 2008; DAREIOTI *et al.*, 2010).

The co-digestion of OMWW with wine grapes residues (WGR) has also been studied in another original paper and the results indicated an improvement of 23-36% in the methane yield of the 1:1 mixture from the two agro-industrial wastes when compared to that obtained from the digestion of pure OMWW and WGR. The ultimate methane yield of co-digesting OMWW:WGR under mesophilic conditions was estimated to be 214 L CH4/Kg COD added, while at thermophilic conditions there was an increase of 28.9% (FOUNTOULAKIS *et al.*, 2008).

The same research group has also observed increased methane and hydrogen production from the co-digestion of municipal wastes, OMW and slaughterhouse wastewater by the addition of 1% crude glycerol, a major by-product of the biodiesel production (FOUNTOULAKIS & MANIOS, 2009).

The olive pomace has also been tested for methane production. The researchers used a culture obtained from a local landfill area, which was left to adapt to the wastes for 10 d at 37 ^{o}C. They used anaerobic digesters of 1 L working volume at 37 ^{o}C, investigating the biogas generation rates at varying concentrations of total solids (TS), and hydraulic retention times (HRT). The maximum rate was found to be 0.70 L of biogas/L of digester volume/d, corresponding to a HRT of 20 days and 10% TS with a yield of 0.08 L of biogas/g COD added to the digester. The methane content of the biogas was in the range of 75–80% the remaining being mostly carbon dioxide (TEKIN & DALGIC, 2000).

LIQUID FUELS

The possibility of creating liquid fuel from used olive oil and olive cake has also been studied. The by-products were either pyrolised or submitted to alkali catalyzed transesterification using methanolic solutions of NaOH, KOH, Na_2CO_3, or K_2CO_3. The alkali catalyzed processes could yield esters up to 94%, creating a fuel with specifications similar to diesel (DEMIRBAS *et al.*, 2000).

HYDROGEN PRODUCTION FROM OOW

Aside from methane production OOW can be used for Hydrogen production, which is considered a renewable and pollution free fuel (MCKINLAY & HARWOOD, 2010).

The most commonly used microorganisms are the photosynthetic bacteria that have high conversion yields of organic compounds to Hydrogen, due to their ability to trap multiple wavelengths and their versatility in utilizing multiple organic substrates.

Hydrogen production experiments were conducted using diluted OMWW in the range of 20% to 1%. The maximum Hydrogen production potential (HPP) was achieved at 2% with a production of 13.91 L H_2/L of OMWW. During the process COD decreased from 1100 mg/L to 720 mg/L, BOD_5 from 475 mg/L to 200 mg/L and phenol content from 2.32 mg/L to 0.93 mg/L. Furthermore some valuable byproducts were produced, like carotenoids (40 mg/L OMWW) and polyhydroxybutyrate (60 mg/L OMWW), making OMWW a promising substrate for biohydrogen formation (EROGLU *et al.*, 2004). Pre-treatment of the wastes with clay doubled the hydrogen production compared to the untreated wastes, possibly due to removal of hardly biodegradable compounds like phenols (EROGLU *et al.*, 2008). The same research group in a recent paper studied four different OOW and observed a positive correlation between the organic load (mainly acetic acid, aspartic acid and glutamic acids) and C/N ratio of the wastes with the biohydrogen production. The wastes from a traditional olive mill had the maximum hydrogen yield with a HPP of 20 m^3/m^3 of wastes and a C/N ratio of 73.8 M/M (EROGLU *et al.*, 2009).

An innovative treatment is the two-stage anaerobic digestion where hydrogen and methane production can take place in two separate bioreactors in series. The two-stage anaerobic treatment process has several advantages over the conventional single-stage process, since it permits the selection and the enrichment of different bacteria in each anaerobic digester and increases the stability of the whole process by controlling the acidification phase in the first digester and hence preventing the overloading and/or the inhibition of the methanogenic population in the second digester (Ueno *et al.*, 2007; Koutrouli *et al.*, 2009).

RECOVERY OF PHENOLIC-ANTIOXIDANT SUBSTANCES AND OTHER BIOLOGICALLY ACTIVE COMPOUNDS

Another possible application for the OOW is the extraction and recovery of the biologically active constituents that exist in the wastes. The OOW contain high concentrations of several phenolic compounds as presented in Tables 1 & 2. The phenolic compounds are amphiphil but with a better solubility towards water. Olive oil contains 50-100 mg/Kg of phenols representing only the 1-2% of the total phenolic contain of the fruit, the rest of them being "lost" to the wastes (Rodis *et al.*, 2002).

The phenolic compounds are a large family of molecules sharing one common feature, at least one phenolic group, substituted in most of the cases with another functional group. OOW contain phenolic alcohols, phenolic acids, secoiroids (elenolic acid derivatives like oleuropein), flavonoids lignin and lignanes.

The phenolic compounds have the ability to capture and neutralize the free radicals and reactive oxygen species (ROS) that are created in organisms during several metabolic pathways. The phenols can also inhibit the oxidative actions of hydrogen peroxide in contrast to other natural antioxidants like vitamin E. Phenols can also a) protect the Low Density Lipoproteins (LDL, informally called "bad" cholesterol) from oxidative modifications (Scaccini *et al.*, 1992; Andrikopoulos *et al.*, 2002), b) inhibit platelet aggregation (Petroni *et al.*, 1995; Fragopoulou *et al.*, 2007), c) inhibit the cytotoxic effects from ROS (Manna *et al.*, 1997), d) inhibit the enzymes that produce several inflammatory molecules (Petroni *et al.*, 1994; Kohyama *et al.*, 1997) and finally e) act as antibacterial and antifungal on microorganisms like *Salmonella, Cholerae, Staphylococcus, Pseudomonas, Cryptococcus* (Juven & Henis, 1970; Mahmoud, 1994; Sousa *et al.*, 2006). OOW along with the phenolic compounds contain several more biologically active lipids, like glycolipids that have the ability to inhibit the action of the most potent inflammatory mediator known, the Platelet Activating Factor (PAF) (Demopoulos *et al.*, 1979; Karantonis *et al.*, 2002; Karantonis *et al.*, 2008). PAF acts through several pathways to initiate and sustain inflammatory responses in organisms and is directly implicated in the pathogenesis of atherosclerosis which is an inflammatory disease and the first cause of death in western world (Demopoulos *et al.*, 2003). The polar lipid fraction of olive oil and pomace that contains the glycolipids acting as PAF inhibitors, when administered to hypercholesterolemic inhibited the formation of atheromatous plaques but also reduced the thickness of the ones already formed (Karantonis *et al.*, 2006; Tsantila *et al.*, 2008).

Many process have been proposed for the recovery of the biologically active compounds many of them covered by patents (Niaounakis & Halvadakis, 2006). Almost all contain a separation step using resins, filters of activated carbon or the use of membrane technology (ultrafiltration, reverse osmosis) and a final step of purification through chromatographic separation (Agalias *et al.*, 2007).

Bentonitic rocks and other activated clays are sometimes used as low-cost adsorbents. The clayey adsorbents achieved a maximum phenol removal from the wastes of about 81% and 71% removal of the total organic matter, within 4 hours (Al-Malah *et al.*, 2000). In another study the HPLC comparative analysis of the initial OMWW with the one filtered through clayey diatomite and the organic extract of the filter pointed the selective adsorption of specific phenolic molecules by the filter. An increment of their relative concentration was observed in the filter's extract that came in agreement with the reduction of the same compounds' relative concentrations in the filtered OMWW, when compared to the initial unfiltered sample. The glycolipids, which are the most potent inhibitors of PAF on the other hand were concentrated after the filtering of the sample or the substances that were co-eluted in the HPLC were removed, increasing their biological activity (Stamatakis *et al.*, 2009). The use of this kind of pretreatment has the benefit of initially separating the two major biologically active components present in OMWW facilitating the subsequent separation steps. The aforementioned purified biologically active molecules can be used as food additives creating new functional foods enriched in substances that promote the beneficial effects of the Mediterranean Diet.

The adsorption of phenolic molecules from bentonites has been extensively studied by (Al-Asheh *et al.*, 2003). Sodium-treated bentonite underwent several activation methods before its exposure to the phenol solution. It was treated with cetyltrimethyl ammonium bromide (CTAB) as a cationic surfactant, with aluminum-hydroxypolycation as a pillaring agent and a combination of the two (CATB/Al-Bentonite). The Na-bentonite was also physically treated in an oven operated at 850 °C. Batch adsorption tests were carried out to remove phenol from aqueous solution using the abovementioned bentonites. It was found that the amount of phenol removal was seriated in the following order: CTAB/Al-Bentonite>Al-Bentonite>CTAB bentonite>thermal-treated bentonite>cyclohexane-treated bentonite>natural bentonite. X-ray diffraction analysis showed that the reason behind the highest phenol uptake was an increase in the microscopic platelets of bentonite treated with CTAB. The increase in sorbent concentration or initial pH values of the solutions also increased phenol removal from the solution. The increase in temperature decreased phenol uptake by the bentonites used in this work. The Freundlich isotherm model was employed and well represented the experimental data.

The improved absorption of organic compounds from chemically modified montmorillonite was observed from other research groups (Freitas *et al.*, 2009). The absorption of organic acids positively correlated with the surface area, pore volume and distribution, as expected but was also affected from the adsorbent's surface polarity. Polar adsorbents (hydrophilic) have higher affinity for polar substances, and nonpolar adsorbents (hydrophobic) have a larger affinity for nonpolar adsorbates. In the case of the modified montmorillonite clay, the adsorption capacity (polar adsorbent) is favored due to the presence of the organic cation hexadecyltrimethylammonium ($HDTMA^+$) among its layers, which makes the surface more hydrophobic and organophilic than the unmodified montmorillonite. In all adsorption processes studied, considering the adsorbent and adsorbate type, initial concentration of the solutions, and temperature, the negative values of DH and DG showed that the adsorption of organic acids on unmodified montmorillonite and HDTMA-montmorillonite clays is a exothermic and spontaneous process. The increase in the values of DG with the increase in temperature demonstrated that the adsorption process was unfavorable at high temperatures. The equilibrium experimental data of acetic acid adsorption on unmodified montmorillonite and HDTMA-montmorillonite were well represented by Langmuir and Freundlich models, and the same observation can be made for the adsorption of butyric acid on montmorillonite.

Zeolites have an affinity in absorbing phenolic molecules (Crini, 2006). When tested against OMWW treatment zeolite removed phenolic/polyphenolic molecules better than the bentonite but it appeared less effective than the clay mineral in reducing the COD of the samples. Furthermore the regeneration and reuse of the minerals was tested after treating them with Low Temperature Ashing (LTA) technique, which allows the application of a controlled oxidation of organic matter at low temperature (80–100 °C), transforming organic materials into CO_2 and water vapor, without affecting the inorganic constituents (D'Acqui *et al.*, 1999). The zeolite turned out to be more thoroughly regenerated than the bentonite, able to remove phenolic/polyphenolic molecules and reduce COD after multiple uses. One interesting observation is that the regenerated zeolite had even better adsorption properties than the raw material and it was attributed to the better drying of the mineral during LTA process (80-100 ^{o}C under vacuum) compared to raw that had some humidity left. These experimental results are rendering zeolite a useful mineral in reducing the organic load from the OMW even thought it wasn't the best adsorbent tested.

The best phenol/polyphenol and COD removals were achieved by a clay soil (typic xerorthent from Val d'Orcia, Siena, Italy; 45% clay (mostly kaolinite), 52% silt and 3% sand). Even though it had good adsorption properties, it allowed extremely low OMWW percolation rates and it was impossible to regenerate it (Santi *et al.*, 2008).

LIME TREATMENT

Lime (CaO) except the neutralization of the wastes' mild acidic pH has also been suggested by researchers for their pretreatment. Lime can easily be purchased anywhere and it is cheaper than other chemicals such as aluminium sulfate, ferric chloride, magnesium sulfate, etc., that have been suggested by other researchers for the same purpose (Curi *et al.*, 1980; Tsonis *et al.*, 1989). After the lime treatment process, COD values of the samples reduced by 41.5% for pressing system wastes and 46.2% for OMWW. The average removal percentage of the other parameters obtained from the samples of nine classical pressing and eight centrifugal systems are 29.3–46.9% for total solids, 41.2–53.2% for volatile solids, 74.4–37.0% for reduced sugar, 94.9–95.8% for oil-grease, 73.5–62.5% for polyphenols, 38.4–32.0% for volatile phenols and 60.5–80.1% for nitrogenous compounds, respectively. The removal of the TS and the oil-grease phase renders the OMWW more susceptible to further treatment like filtration and evaporation. Another really interesting observation of this study is that the lime treatment of a mixture from standard phenolic compounds present in OMWW showed a total removal of the *o*-diphenolic compounds, a partial removal for the compounds containing both phenolic and carboxylic groups, while the substances with one phenolic or one carboxylic group are unaffected (Aktas *et al.*, 2001). The same research group proposed a lime treatment procedure for the recovery of the residual oil. The researchers optimized the oil recovery by adding lime at a concentration of 8g/L while passing air through the mixture. This recovered oil is not edible but can be used for the production of soaps and cosmetics (Saglik *et al.*, 2002).

An improvement to lime treatment has been proposed by (Ugurlu & Hazirbulan, 2007), combining lime and clay mineral treatment. In this study, OMWW was treated with lime to a pH of 7, followed by 30% H_2O_2 solution. Phenol and lignin removal rates were found to be 99.5% and 35%, respectively. Then, adsorption experiments were carried out by using different sepiolite samples, i.e. heat activated sepiolite (AS), thermal- acid-activated sepiolite (AAS) and thermal-base-activated sepiolite (BAS). Their surface modification was examined by SEM, XRD and FT-IR techniques. BAS samples were shown to provide more phenol and lignin removal than AAS and AS. However, it was evidenced that adsorption on sepiolite is a complex process, so it cannot be sufficiently described by a single kinetic model throughout the whole process. For example, intra-particle diffusion played a significant role, but it was not the main rate-determining step during adsorption. The adsorption data for AS were also fitted to Langmuir and Freundlich isotherms and exhibited a better fit to Langmuir. Gibbs-free energy for lignin and phenol were calculated, and ΔGo values of lignin and phenol adsorption process were -5.718 and -2.272 kJ/mol, respectively, which indicated chemical forces between the adsorbed molecules and AS.

WATER SUBSTITUTE IN BRICK MANUFACTURE

The utilization of the OMWW in brick manufacturing as a water substitute is another possible environmental and cost effective management of the wastes. The substitution of water had no detrimental effect on extrusion performance when compared with the standard (control) product, despite the acidity and solids present in the waste. The bricks produced using OMWW instead of water (OMWW bricks) required approximately 20% (v/v) less amount of water to achieve optimum extrusion compared to control, there as less energy input normally required for the essential pre-drying of the freshly extruded brick product prior to its firing. Drying shrinkage values of the OMWW bricks are comparable to control consequently there would be no need of changing the brick making process apart from the shorter drying cycle. The firing of the bricks showed similar levels of contraction between the control and the OMWW bricks. The burn-out of the solid organic matter present in OMWW, the OMWW bricks had higher open porosity and subsequently lower density than the control, resulting in lightweight bricks with improved thermal insulation properties. This, in turn, gives rise to increased water absorption and consequential lower strengths but still falling within the specifications and therefore acceptable for constructions. Moreover, it is believed that the calorific value of OMWW's organic matter (21-23 MJ kg^{-1}) and consequent heat liberation into the kiln during firing would offer a slight contribution towards the total fossil fuel energy input required by the brick kiln. To all this we must also mention the water that is saved when OMWW is used in its place (Mekki *et al.*, 2006). The authors scaled up these encouraging laboratory results and used OMWW instead of water in a project at a Tunisian brick company. The results were similar to the ones in the laboratory. Specifically the OMWW bricks were comparable in forming/extrusion performance to a control product that used fresh water. The OMWW bricks were tested for their comparative physical properties (volume shrinkage, water absorption, tensile strength of bricks, after firing at 920 °C and paste plasticity) in the unfired and fired states against a control representing the commercial product in the same factory. The results showed a significant increase in the volume shrinkage (10%) and the water absorption (12%), while the tensile strength remained constant. The maximum plasticity index value was found when incorporating 23% of OMWW. This rate either maintained the physical and mechanical properties of bricks or improved them. The incorporation of OMWW in bricks can represent a promising way to valorize this effluent, to rid the environment of a highly polluting wastewater and to save huge and precious amounts of water in a country where water shortage is a serious problem. This newly-prepared material has a double positive impact: it protects the environment and allows water economy (Mekki *et al.*, 2008).

Similar results were obtained when instead of OMWW the researchers used OMW as a water substitute. The OMW bricks had all the benefits of OMWW bricks mentioned above and also an improvement of 33% to their dry-bending strength when compared to control (De La Casa *et al.*, 2009).

BIOSORBENT FOR HEAVY METALS

Several researchers have proposed the utilization of the wastes as cheap metal biosorbents, treating heavy metal contaminated industrial wastes before their disposal. The composition of both OOW and olive pomace due to the presence of polyphenols, cellulosic fibres, lignins, and bio-polymers, render the wastes ideal materials for metal binding, through adsorption, chelation, complexation and ion exchange phenomena (Veglio & Beolchini, 1997). The preliminary results of olive pomace testing as metal biosorbent showed a correlation between time required for adsorption equilibrium and metal atomic weight-number. Specifically Cu, Zn and Cd reached equilibrium within 2 hours, while the heavier Hg and Pb needed 4 hours, their large ionic radius reducing diffusion. Moreover there was a correlation between the uptake of the metal from the waste and the acidic nature (pKi) of the metals, the higher the pKi the lower the uptake (Pagnanelli *et al.*, 2002). An exception to this observation is Cu and its ions that have the highest uptake but also high pKi, their affinity beside ion exchange also influenced by specific complexation with carboxylic and hydroxylic groups of the polyphenols and reducing sugars present in the water soluble organic fraction of the wastes (Pagnanelli *et al.*, 2003; Gondar & Bernal, 2009). The wastes can also be regenerated by a mildly acidic solution (HCl 0.1M) and re-used or burned after every application recovering the metals from the ashes (Pagnanelli *et al.*, 2002). Another research group studying the possibility of regenerating OMW for re-use has concluded that the 0.1 M HCl was not strong enough suggesting the use of at least 1 M HCl (Martinez-Garcia *et al.*, 2006). The authors have also optimized adsorption through agitation (150 rpm) and pH values (pH 7). The multi-metal biosorption experiments they conducted showed the ability of the wastes to simultaneously adsorb several metals, although the presence of Cu highly reduced the adsorption of the other metals present. Especially in the case of Pb and Cd they concluded that OMW is able to repeatedly sequester the metals from solution, releasing them later into an acid solution (regeneration) (Martinez-Garcia *et al.*, 2006). Cadmium has also been studied kinetically in single ion solution using as adsorbent olive pomace, achieving maximum removal from the solution at pH 11 due to the lack of competition between Cd^{2+} and H^+ at the binding sites but also the synergistic effect of $Cd(OH)_2$ precipitation at pH>9 (Blázquez *et al.*, 2005). The dramatic decrease in metal uptake from the wastes has also been observed for other small cations like Na^+ which compete the metal cations' binding sites the way H^+ does (Fiol *et al.*, 2006).

CONCLUSIONS

The olive oil production industry creates a lot of by-products with different consistency and physicochemical abilities which in conjunction with the wide spread locations of the olive mills and the seasonal nature of the wastes make difficult their management in an environmental friendly and cost-effective way.

The utilization of some of the aforementioned waste management methods alone or in combinations can transform the different types of wastes in starting materials for energy production (fuel blocks, biogas and hydrogen), water economy (water substitute in brick manufacture) and treatment (heavy metal removal from industrial wastes) and, probably the most important, a raw material for the extraction of natural compounds (phenols, antioxidants and inhibitors of inflammation) with high biological activity through the use of low cost minerals (bentonite, lime, zeolites) abundant in all the olive oil producing countries.

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