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Combined biological and chemical-physical process for olive mill wastewater treatment

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ABSTRACT

The aim of the present study was to investigate on the combined biological and chemical-physical process for the olive mill wastewater treatment for its ultimate disposal into surface waters and/or reuse. The chemical-physical process was used as a pre-treatment to the biological step. Tests on precipitation were performed using three coagulants, such as lime, alum and iron chloride salts, and varying their dosages under predetermined optimum pH conditions. At optimal pH of about 12, lime achieved 51% COD removal efficiency. As far as the alum and iron chloride salts ($FeCl_3 \times 6H_2O$) performances, the latter resulted in a 19% COD removal at a dosage of 3 g/l, while in the experiments using $Al_2(SO_4)_3 \times 18H_2O$, 20% COD removal with a dose of 4 g/l was observed. From the results obtained, lime was chosen as the optimal reagent. It was also shown that it is suitable to be used as influent to a subsequent biological step. A lab-scale Sequencing Batch Reactor (SBR) was used to carry the biological process. The plant was fed by the diluted OMWs as in the chemical coagulation tests. A final removal efficiency of about 60% was obtained at optimal operative conditions.

Keywords: Olive mill wastewater; Poly-phenol; Pre-treatment; Sequencing batch reactor

1. Introduction

The olive-oil extraction industry is an economically important activity for many countries of the Mediterranean Sea area, with Spain, Greece and Italy being the major producers [1,2]. This activity, however, may represent a serious environmental problem due to the discharge of highly polluted effluents, usually referred to as "olive mill wastewater" (OMW). It is estimated that every ton of milled olives corresponds to about 0.80 ton of OMW [3]. OMWs usually contain COD values as high as 80–300 g/l and also considerable amounts of suspended solids [4]. Furthermore, the organic matter mainly consists of polysaccharides, sugars, polyphenols, polyalcohols, proteins, organic acids and oil; some of these substances are difficult to biodegrade and may exert toxic and inhibitory effects on the microbial activity [5,6].

On the other hand, the polyphenols are characterised by antioxidant activity and are, therefore, of great interest to the cosmetic and pharmaceutical industries, in food processing and food products conservation. After filtration to eliminate the suspended solids, all compounds can be recovered by physico-chemical processes such as ultrafiltration, nanofiltration and reverse osmosis [7]. In recent ys, increasing attention has been devoted to the possibility of valorising the olive oil extraction residues. Several valorisation approaches have been attempted including, among others, secondary oil extraction, combustion, gasification, anaerobic digestion, composting and even the production of building bricks [8,9].

Traditional disposal on soil is still the typical solution adopted in Italy. However, the Italian law in force

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(L. 574/96) restricts the maximum amount of OMWs to be disposed off on soil to 50 m^3/ha and to 80 m^3/ha for wastewater arising from a traditional or a continuous mill, respectively.

In the recent ys some studies have investigated on alternative treatment solutions more environmentally compatible than soil disposal.

According to Ginos et al. [10], lime concentrations up to 40 g/l led to partial OME destabilisation with TSS removal being as low as about 40%, while the pH of the resulting liquid phase increased to about 7–8. However, increasing concentration to 60 g/l resulted in about 85% and 67% TSS and TP removal, respectively, with the solution pH increasing to about 11.5. COD removal was about 10% while the ratio of sludge generated resulted in over 80% sludge volume.

A thorough review on the use of biological and advanced oxidation technologies for OMW treatment was produced by Mantzavinos and Kalogerakis [11]. The authors refer that lime precipitation and water evaporation in ponds are commonly applied since relatively cheap; however, these processes alone can only marginally meet the more stringent requirements posed on discharge. A well-designed sequential treatment consisting of various chemical, physical and biological processes represent a better solution. Fenton's reagents applied on OMWs guaranteed total polyphenol and COD removal of 60% and 23%, respectively. In an aerobic batch reactor filled with the same OMWs, a biomass rich in fungi developed after about 30 d and was able to biodegrade phenolic compounds up to 70%. It was also observed that the advanced oxidation pre-treatment increased OMW biological treatability [12].

Coupling ozonation with anaerobic digestion caused stronger inhibition of methanogenic bacteria [13]. However, this effect was not present on acidogenic bacteria.

Results of great significance were obtained by adding $Ca(OH)_2$ (up to pH 6.5) and 15 g/l of bentonite, and then feeding the mixture to biological treatment without providing an intermediate phase separation [14]. Decolourization of 90% and nearly 85% removal of phenols were achieved by OMW electrochemical treatment in a modified Grignard reactor [15].

Despite the great effort spent up to now on the study of OMW treatment, still more research needs to be carried out directed towards the development of more economically and environmentally sustainable treatment solutions for OMW.

The above cited references highlight the optimal solution consists in the combination of different processes; however, the potentiality of some innovative systems has not been exploited completely yet. For instance, few studies can be found on the application of discontinuous processes for the biological step, despite it has been widely demonstrated their capability of biodegrading several recalcitrant compounds, such as phenol and chlorophenols [16–19]. In particular, a sequencing batch reactor (SBR) with its typical dynamic conditions, guarantees high flexibility, simple running, compact layout and is able to select the microbial species capable of degrading toxic compounds [20].

Therefore, the aim of the present paper was to investigate on the suitability of the SBR to carry out the biodegradation of the pre-treated OMWs. As preliminary stage, it was decided to use a chemical coagulation and to study the removal efficiency obtained with different coagulants. This paper will present the first results from the different chemical experiments and from a lab-scale SBR fed with either a diluted or a chemically pre-treated influent.

2. Materials and methods

2.1. Influent characterization

The raw OMW used for the experimental research was obtained from an olive oil continuous centrifuge processing plant located in the province of Lazio (Italy).

The OMW was preliminary sieved at 300 μm in order to reduce its content in suspended solids. Table 1 shows OMW characterization after sieving.

The experimentation was performed by using diluted OMW. This was prepared by mixing and diluting in distilled water known amounts of OMW (dilution ratio 1:25) to reduce the influent loading and to adjust the pH to the final value of 8.

2.2. Experimental setup

2.2.1. Coagulation tests

The physico-chemical treatment experiments were carried out using 500 ml batch reactors in a jar test equipment, in order to investigate the effects of different coagulants (such as lime, $Al_2(SO_4)_3 \times 18H_2O$ and $FeCl_3 \times 6H_2O$) at various dosages. Trials were performed at least in triplicate and the average results determined. For lime, the optimal pH was firstly determined based on the COD removal achieved and keeping fixed chemical

Table 1
Influent OMW composition

Parameters	Sieved OMW
pH	5.8
COD (g/l)	69.4
TSS (g/l)	26.6
VSS (g/l)	24
Total polyphenols (g/l)	3.4
Lipids (g/l)	2.9

Table 2
Operating conditions of the chemical coagulation experiments

Reagent	Dosage (g/l)	Coagulation pH
$Ca(OH)_2$	5	10, 11.5, 12, 12.3
$Ca(OH)_2$	5, 10, 15, 20	12
$Al_2(SO_4)_3 \times 18H_2O$	2, 3, 4, 6	8
$FeCl_3 \times 6H_2O$	2, 3, 4	8
Acid cracking	–	2
Acid cracking + $Al_2(SO_4)_3 \times 18H_2O$	2, 3, 4, 6	8
Acid cracking + $FeCl_3 \times 6H_2O$	2, 3, 4	8

dosage. In metal salts coagulation test the pH was adjusted to 8 according to Kestioglu et al. [21]. Then, the optimal dosage was determined for all the coagulants based on the COD removal efficiencies. For lime, the dosages were tested in the range of 5 ÷ 20 g/l; whereas for both $Al_2(SO_4)_3 \times 18H_2O$ and $FeCl_3 \times 6H_2O$ they were varied between 2 and 6 g/l. All the tests were performed at room temperature (20 ± 2 °C).

It was also investigated the effects of acid cracking on the COD removal, considered both alone and as a pre-treatment to chemical coagulation with $Al_2(SO_4)_3 \times 18H_2O$ and $FeCl_3 \times 6H_2O$. Acid cracking was applied by manually adjusting pH to less than 2 using sulphuric acid. All the operating conditions of the chemical coagulation experiments are listed in Table 2.

Jar tests for lime precipitation were carried out considering 3 min at 120 rpm, 20 min at 35 rpm and 2 h of sedimentation. For alum and iron chloride salts, one-h sedimentation was applied following coagulation time of 30 min (20 rpm) after flash mixing for a minute (120 rpm). The final pH was recorded in the sample taken from the supernatant phase above the precipitate; concentrations of total polyphenols (TP), total suspended solids (TSS) and COD in the same sample were also measured.

2.2.2. SBR plant and operation

The biological process was carried out in a lab-scale reactor of a total working volume of 10 l (V_{tot}). Each cycle of operation was composed of four phases: fill, react, settle and draw. Fill and react were both operated under mixed and aerated conditions.

During the first h of the aerobic phase, the influent volume (V_{fill}) was provided to the reactor, whereas the same volume of supernatant (V_{eff}) was withdrawn at the end of the settling phase which lasted 30 min. Two peristaltic pumps were used for the influent addition and the effluent draw, respectively. A mechanical mixer was used during the fill and the react phases. Besides, aerobic conditions were established by blowing air through a porous stone so as to obtain oxygen concentration in the mixed liquor always above 2 mgO_2/l. The duration of feed, aeration, mixing and draw was controlled by a timer. In order to maintain a high sludge retention time (Θc) and favour the biomass growth, the sludge waste was not applied during all the experimentation. The temperature was kept constantly equal to 20 ± 2 °C by means of a re-circulating water bath. The operating conditions of the SBR plant experiments are listed in Table 3.

The plant was seeded with a sample of activated sludge from a municipal wastewater treatment plant of the city of Rome. The biomass was gradually acclimated to the toxic and recalcitrant compounds present in the influent wastewater by using a batch mode of operation with a variable duration of the treatment cycle (Table 3). Each batch was started when residual COD concentration was low enough. This strategy allowed progressive selection and enrichment of the microbial species able to use the wastewater as energy and carbon source. Consequently, it was possible to gradually reduce the length of each cycle from 7 d to 1 d (see period I to period III). In period III operating conditions were significantly changed (VER was decreased up to 0.1 and the length of the cycle was reduced to 1 d) in order to evaluate biomass response to a stressing environment. In periods IV and V, previous operating conditions were progressively recovered: the length of the cycle raised to 3 d whereas the VER changed from 0.05 to 0.2 in order to avoid biomass-inhibiting loadings at the end of the fill phase.

2.2.3. Biological batch tests

Biological batch tests were also carried out resembling the lab-scale SBR in term of VER and Food/Microorganisms (F/M) ratio. The tests were performed in a 2 l reactor, under mixed and aerated conditions. Fill and draw

Table 3
Operating conditions of the SBR plant experiments

Parameters	Period I	Period II	Period III	Period IV	Period V
VER*	0.2	0.2	0.1	0.05	0.2
Cycle length (d)	7	3	1	3	3
Phase duration (d)	31	21	3	12	9

*Volumetric Exchange Ratio (V_{fill}/V_{tot}).

were manually operated. The biomass used for the tests was collected from the SBR lab-scale plant. As feed, it was used the supernatant from the chemical precipitation test which had given the best results in terms of COD removal.

Reactor and batch performances were monitored through periodical analyses on liquid samples from both the influent and the effluent streams. Besides, kinetic studies were also carried out by measuring the main parameters at regular intervals during operation.

2.3. Analytical methods

Temperature, pH (WTW, 330/SET-1) and dissolved oxygen (DO) (YSI 5739) were monitored throughout the process. Samples of the mixed liquor were periodically collected from the reactors. The samples were filtered at 1.2 μm to determine TSS and volatile suspended solids (VSS), by gravimetric method, and at 0.45 μm to determine COD in the filtered sample. TP content was determined spectrophotometrically according to the Folin-Ciocalteau method. Lipids were determined after petroleum ether extraction. The sample is acidified and extracted with a mixture containing n-hexan (80%) and MTBE (20%). The extracted liquid is evaporated and the residue is determined gravimetrically [22]. COD, TSS, VSS measurements were performed according to the Standard Methods for the Examination of Water and Wastewater [23].

3. Results and discussion

The experimental activity was divided into two parts: in one part, the chemical precipitation pre-treatment was investigated by testing different coagulants, while in the other part the biological process in a lab-scale SBR plant was studied. In both parts, diluted OMW was used. Besides, preliminary biological batch tests were performed, in order to verify suitability of the chemically pre-treated OMW to be used in the SBR plant in place of the diluted wastewater.

3.1. Chemical-precipitation process

Regarding lime precipitation, the COD, TP and lipids removal efficiencies at varying pH and with a low dosage of 5 g/l are reported in Fig. 1. The trials showed maximum removal efficiencies corresponding to the optimal pH value of 11.5–12. These results are in accordance with those found in the specialized literature [3,12,24]. Particularly, Aktas et al. [3] found removable percentages of about 64% for TS and VS, about 65% of TP and about 78%, 87% and 95% for sugar, nitrogen and oil-grease, respectively, when the lime was added at 10–25 g/l until the pH of the mixture reached 12. Among these results, only volatile phenols were adsorbed at a relatively low degree.

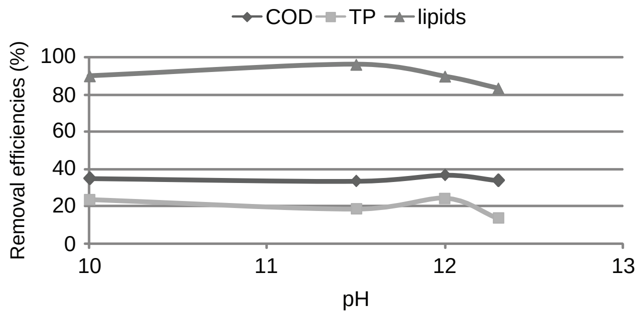

Fig. 1. COD, TSS, TP and lipids removal efficiencies at different pH with a lime dosage of 5 g/l.

Based on a comparison of the chromatograms of treated and untreated artificial phenolic mixture they found that some of the phenolic substances could be removed totally or partially and some of them were not affected. If the structures of these substances were considered, it was observed that the substances with two phenolic groups in the molecule, like catechin were totally removed; the substances which contain both phenolic and carboxyl groups, such as vanillic acid, syringic acid, were adsorbed partially and the substances which have only one phenolic or carboxyl group such as tyrosol and veratric acid were not affected by lime.

Comparison of the COD removal efficiency obtained at varying dosages of lime, $Al_2(SO_4)_3 \times 18H_2O$ and $FeCl_3 \times 6H_2O$ and at pH of 12 and 8, respectively, is represented in Fig. 2.

It can be noted that increasing the lime dosage from 5 up to 20 g/l caused COD removal efficiency to rise correspondingly, reaching a maximum value of 51% at 20 g/l.

The ratio of sludge generated following separation after lime precipitation over the total initial volume varied from about 66% sludge volume at 5 g/l to about 78% sludge volume at 20 g/l.

The TSS removal efficiency and the sludge generated volume were quite similar to those observed by Ginos et al. [10] at the same dosage using undiluted OMWs.

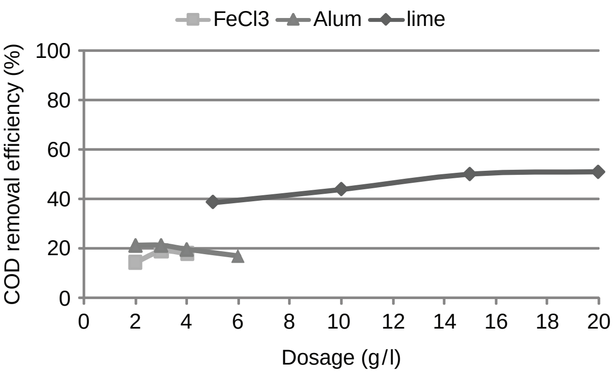

Fig. 2. COD removal efficiency of lime, alum and iron chloride salts at different dosages and pH.

Table 4
Characteristics of OMW after acid cracking and after lime precipitation (at dosage of 20 g/l)

Parameters	After acid cracking	After lime precipitation
pH	< 2	12
COD (g/l)	40.6	44
TSS (g/l)	1.4	2.8
VSS (g/l)	1.1	2.2
Total polyphenols (g/l)	4.1	2.6
Lipids (g/l)	0.3	0.3

Between alum and iron chloride salts, $FeCl_3 \times 6H_2O$ resulted in an average 19% COD removal at a dosage of 3 g/l, while in the experiments using $Al_2(SO_4)_3 \times 18H_2O$, 20% COD removal with an optimal dose of 4 g/l was observed.

Sarika et al. [25] achieved similar COD removal efficiencies using $FeCl_3 \times 6H_2O$ at the same dosage (1 g/l) but at a different pH (4.5 units).

Characteristics of OMWs after acid cracking and after lime precipitation are given in Table 4. It can be noted that acid cracking resulted in an average 41% COD, 28% Total Phenol removal and a significant amount of oil and grease removal. These results are in agreement with Kestioglu [21].

However, when the acid cracking was used as a pretreatment to alum and iron chloride salts precipitation, improvement of the COD removal efficiencies were not observed. These results do not correspond to the data found by Kestioglu [21], probably due to the different characteristics and source of the OMW used in the study.

Comparison of the COD removal efficiency after different chemical treatments and at varying dosages, is represented in Fig. 3.

Based on the results obtained in all the coagulation experiments, lime with a dosage of 20 g/l at pH 12, was chosen as the optimal chemical treatment for the OMWs used in the present study.

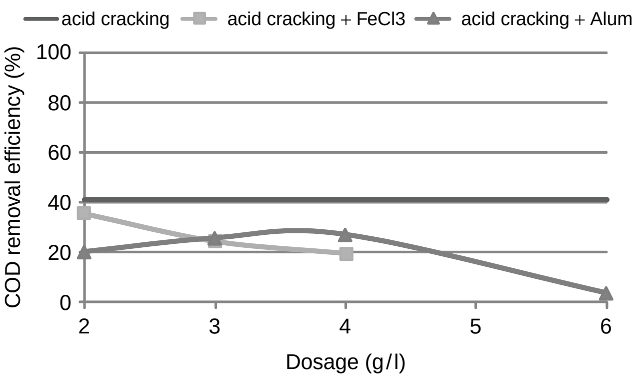

Fig. 3. COD removal efficiency for different chemical treatments at varying dosages.

3.2. Biological process

3.2.1. SBR

During period I, the effluent COD concentration decreased continuously with time; consequently, removal efficiency improved, finally reaching an average value of 70%. However, when the length of the cycle was reduced to 3 d (period II) and later on to 1 d (period III), the biomass responded negatively and the removal efficiency drastically diminished. The progressive accumulation of the pollutants within the reactor, due to the reduced performances, did not allow to evaluate average efficiency. These results indicated that, though the high removal observed in period I, the biomass was not completely acclimatized to the OMW and, particularly, to the potentially toxic phenols contained in the influent. Therefore, in the following periods (IV and V) it was needed to modify the operative conditions so as to favor biomass-degrading capability recovering. The new operation strategy was able to improve the removal efficiency which was about 40% during period IV and finally reached an average value of 56 % in period V. Kinetic studies were also periodically carried out within typical operative cycles of the SBR at regime conditions in period V. The results obtained are reported in Fig. 4.

3.2.2. Batch test

The supernatant after lime precipitation was used as feed for the batch test. Prior to be fed to the batch, pH value of the supernatant was adjusted to eight through sulfuric acid addition; besides, the initial COD concentration was diluted so as to be comparable with the value reached in the SBR at the end of the fill phase. This allowed to investigate on the response of the SBR biomass to the supernatant from the chemical test.

The average results of the batch test are reported in Fig. 4 along with those from a typical kinetic test performed in the SBR. It can be noted that the filtered COD (COD_F) concentration was reduced in both batch

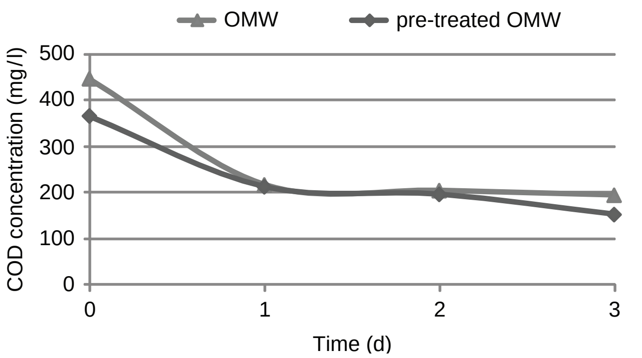

Fig. 4. COD concentration with time in the batch test and in a typical SBR cycle.

and SBR to about 200 mg/l; then, only in the batch, it decreased further to 150 mg/l, for a final COD removal efficiency of about 60%.

4. Conclusions

A combined biological and chemical-physical process for the OMW treatment was investigated. The results from the chemical precipitation experiments highlighted that lime, with a dosage of 20 g/l and pH 12, was the optimal reagent. The supernatant from the lime precipitation showed also to have high biodegradability and therefore to be suitable to be used as influent for a subsequent biological process. A lab-scale SBR plant was operated by feeding it with diluted OMWs as used in the previous chemical tests.

The operative conditions of the SBR were modified with time to allow biomass to acclimate to the OMWs and, particularly, to their toxic compounds. In the final stage of the study, the average COD removal efficiency reached about 60%.

The results obtained from the study, though only preliminary, demonstrated suitability of the proposed treatment scheme for the OMWs.

The study will continue by optimizing the biological treatment process with the final aim to produce an effluent suitable for reuse in the OMWs factory. Besides, it would be also interesting to investigate the opportunity to recover valorisable compounds from the chemically precipitated sludge.

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