



Chemical precipitation of aerobically treated olive mill wastewater

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ABSTRACT

The major water pollution problem facing the Mediterranean region is because of the olive mill wastewater (OMW), produced after the extraction of olive oil. Laboratory scale experiments were performed to determine the effectiveness of chemical treatment by the use of cationic polyacrylamide flocculant on raw olive mill effluent, coagulant aluminium sulphate (alum), precipitant calcium hydroxide (lime) blended with cationic polyacrylamide on aerobically treated whey or pig slurry coalesced with OMW. The results show that the chemical treatment is useful for colour reduction by 31.6–74.1%, COD reduction by 15.5–45%, total solids by 6–42%, phenolic compounds by 52.3% and lipids by 69.9%.

Keywords: Olive mill waste water; Cationic polyacrylamide; Coagulants; Precipitants

1. Introduction

Olive oil extraction process produces large quantities of olive mill wastewater (OMW). OMW as such is highly polluting because of high organic carbon content, lipids and aromatic compounds (mono-cyclic phenols and polyphenols). These compounds are hardly degraded by microflora as most microorganisms are sensitive to its inhibitory effects.

The wastewater treatment processes are broadly classified as physical, physico-chemical and biological. The physical and the physico-chemical processes are considered together and they include thermal processes (evaporation and incineration); precipitation/coagulation/flocculation and clarification; ultrafiltration; oxidation [1]; adsorption; electrolysis and reverse osmosis. The biological treatments are further subdivided into anaerobic and aerobic processes. The former exploits microorganisms that do not require oxygen to degrade pollutants in the effluent, while

the latter requires an external source of oxygen. All of these methods have been used with limited success, with biological treatment processes showing promising results. Major draw back of the physico-chemical processes is the final disposal of the sludge, which is reduced in volume in the biological process [2].

Even though physico-chemical process is not very effective in reducing the concentration of major pollutants (phenolic compounds and COD) in the OMW, it has a high potential for the removal of solids and colour. The ineffectiveness is because most of the organic substances present in the olive mill wastewater are difficult to precipitate (e.g. sugars and volatile acids) [3]. However, this process has been utilised to reduce the toxic effect of OMW on bacterial growth [4,5]. Solids present in the OMW can be, (i) dissolved solids (10^{-5} – 10^{-4} μm), (ii) colloidal solids (10^{-2} – 10^{-1} μm) and (iii) suspended solids (10–100 μm). Therefore, colour can be expected to be closely linked with solids. According to Perez et al. [6] the characteristic dark brown colour of the effluent is attributed to polymerisation of low molecular weight

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phenolics and is chemically related to lignin derivatives. In water and wastewater treatment plants precipitant is added to precipitate dissolved minerals. Coagulants on the other hand destabilize the particles and initiate the flocculation process. Subsequently, polymer flocculants are added to aggregate the destabilized particles into larger stable flocs for settling.

Early studies by Tsonis et al. [7] using calcium hydroxide and aluminium sulphate showed a COD reduction between 20 and 30%. Use of aluminium sulphate on centrifuged OMW was found to be effective at concentrations of 4 g/L resulting in 25% colour removal, whereas 35 g/L of lime was required for 15% decolourization [8]. Combination of iron sulphate and calcium hydroxide (10 g/L) precipitant on raw OMW was effective in removing more than 60% COD and 50% colouring compounds, chromophores [9]. Decrease in the COD of OMW resulted in the decrease of colour, which suggests a strong link between the COD and colour reduction. Study by Sarika et al. [10] has shown that use of cationic and anionic poly-electrolytes resulted in >20% removal of COD and >90% removal of TSS on raw OMW. Another study by Azbar et al. [5] with iron chloride, aluminium sulphate and iron sulphate has shown that with an optimum amount of 2.5 g/L following reductions were obtained, colour (15, 65 and 30%), COD (24, 32 and 39.7%), TOC (35, 31 and 34%) and total phenols (19, 25 and 42%).

The main objective of this paper is to study the feasibility of using cationic polyacrylamide flocculant independently and in combination with coagulant aluminum sulphate and precipitant calcium hydroxide on OMW in its original state and aerobically treated with whey or pig slurry. Removal of colour, COD, phenol, lipids and total solids are investigated.

2. Materials and methods

The raw olive mill wastewater used in this study was produced by three phase extraction process whose composition is summarized in Table 1. It was stored in airtight plastic cans and maintained at -20°C until use. Aerobically treated OMW with whey or pig slurry mixture was taken from an 18 L aerobic L.H. Fermenter 2000 Series (L.H. Fermentation, UK) which was filled with OMW (75%) and whey or pig slurry (25%) v/v. This mixture was inoculated with yeast *Candida tropicalis* ATCC 32546 obtained from the American Type Culture Collection (Virginia, USA). The flocculant cationic polyacrylamide was obtained from HISPAQUIMICA (Seville, Spain). Coagulant, aluminium sulphate was purchased from Fisher Scientific UK Ltd. and precipitant calcium hydroxide from BDH Laboratory Supplies, Poole.

Table 1

The raw composition of the olive mill wastewater, cheese whey and pig slurry.

Analysis	OMW	Cheese whey	Pig slurry
pH	5.48	4.74	9.25
COD (g/L)	90	58.30	19.20
Lipids (g/g)	0.02	0.05	0.01
Phenols (mg/L)	700	16.0	90.0
Total Solids (g/L)	66.02	34 [†]	50.97 [†]
Suspended Solids (g/L)	9.65	–	–
Volatile Suspended Solids (g/L)	7.80	–	–

[†] Cheese whey or Pig slurry with crude OMW.

2.1. Sample analyses

The pH of samples were measured using a HANNA pH meter, model HI931300 (Hanna Instruments, Singapore). Sample discoloration was observed with UV–visible spectrophotometer, Cam Spec M330, with the wavelength 395 nm. The COD and mono-cyclic phenol content were determined using Dr. Lange analyzer, model LASA 20 (Düsseldorf, Germany). Lipid content was determined by the Bligh and Dyer method (1959) [11]. Total solids were assayed by following the BS 1016 standard [12]. All analyses were carried out in triplicate with a relative standard deviation not exceeding 5%.

2.2. Experimental procedure

Appropriate amount of flocculant/coagulant/precipitant were mixed with deionised water to dissociate the crystals/powder. In the initial experiment, polymer flocculant solution containing 1 to 10 g/L of cationic polyacrylamide was added to 100 mL of raw OMW and blended using a magnetic stirrer (HI 200 M, Hanna Instruments) for 10 min. Similarly, coagulant/precipitant solutions were prepared by dissolving alum/lime in deionised water. The coagulant solution with concentrations of 2–8 g/L was initially mixed with effluent followed by the polymer flocculant. Each mixing lasted for 10 min. Table 2 shows the various proportions of flocculant/coagulant/precipitant combinations used in the study. Abbreviations used are, ECP – Effluent and Cationic Polyacrylamide, EWASCP – Effluent and Whey mixture with Aluminium Sulphate and Cationic Polyacrylamide, EWCHCP – Effluent and Whey mixture with Calcium Hydroxide and Cationic Polyacrylamide, EPASCP – Effluent and Pig slurry mixture with Aluminium Sulphate and Cationic Polyacrylamide respectively.

Table 2

Proportions of flocculant/coagulant/precipitant combinations used in the experiments.

ECP	EWASCP		EWCHCP		EPASCP	
	AS (g/L)	CP (g/L)	CH (g/L)	CP (g/L)	AS (g/L)	CP (g/L)
0	0	0	0	0	0	0
1	2	0.2	2	0.4	2	0.2
2	4	0.4	4	0.4	4	0.4
3	6	0.6	6	0.4	6	0.6
4	8	0.8	8	0.4	8	0.8
5	–	–	–	–	–	–
10	–	–	–	–	–	–

CP – Cationic Polyacrylamide, AS – Aluminium Sulphate, CH – Calcium Hydroxide, ECP – Effluent and Cationic Polyacrylamide, EWASCP – Effluent and Whey mixture with Aluminium Sulphate and Cationic Polyacrylamide, EWCHCP – Effluent and Whey mixture with Calcium Hydroxide and Cationic Polyacrylamide, EPASCP – Effluent and Pig slurry mixture with Aluminium Sulphate and Cationic Polyacrylamide.

3. Results and discussion

3.1. Coagulation and flocculation process

Of the various components present in olive mill wastewater, the dissolved constituents (molecules or ions) are held by the molecular structure of water, whereas colloids are suspended but to some extent exhibit many of the characteristics of dissolved substances. Suspended materials on the other hand consist of particles larger than molecular size and are supported by buoyant and viscous forces within the water.

Experiments were conducted to investigate the effectiveness of lime, alum and polymer flocculant on pre-treated wastewater (whey or pig slurry mixed with OMW). Lime or alum was initially added to the OMW with rapid mixing. Addition of lime increased the pH of wastewater which is favorable for the precipitation of metals ions [13]. Alum on the other hand destabilized the solution by adsorbing onto the surface of the negatively charged particles (charge neutralization) [14], forming minute aggregates distributed throughout the solution. Growth of these small particles in the solution is predominantly due to double layer compression mechanism [15]. Molecules and small particles formed become entrapped in the colloids as it is formed, or they may become enmeshed by its sticky surface [16]. The above process starts from the molecular level and grow to suspended solids that are large enough to settle. Some of the factors that influence the aggregation process are, metal ion concentration, pH, turbidity, mixing rate and temperature of the solution [17]. Certain elements in OMW remain stable and unaffected by addition of coagulant/precipitant.

When the polymer flocculant is added to the raw OMW, cationic polymer threads adsorb onto the negative particles and colloid surfaces. The areas of polymer adsorption on the particle will have a net positive charge due to the high charge density of the polymer. The positive

regions formed also get attracted to the negative regions of the other particles or colloids (heterocoagulation) [18]. In addition, the charged groups close to the surface of the particles promote flocculation by charge neutralisation. This is followed by bridging of unaffected particles and flocs formed because of the addition of coagulant/precipitant. Since bridging is affected by molecular weight and the ionic content of the solution. Only large molecules can bridge between particles [19] forming large suspended mass. During this bridging mechanism the extending threads tend to adsorb some of the stable particles, chemicals having high COD and colour which are not much affected by the addition of coagulant/precipitant.

3.2. Effect of flocculant/coagulant/precipitant on solution pH

An understanding of the variation of pH with addition of flocculants/coagulants/precipitants is essential because of its effects in the water treatment process [20]. From Fig. 1 it can be seen that no significant change in the pH was observed for raw OMW effluent (5.48) treated with cationic polyacrylamide (ECP). On the other hand, solutions treated with alum showed a small increase in the pH, which decreased subsequently with further addition. However, with the addition of lime the pH increased sharply.

In a study using ferric chloride as coagulant optimum pH range for pollutant removal was between 7 and 9 [21]. In the current study the pH range for maximum colour, COD and total solids removal with flocculant/coagulant/precipitant was between 5.06–6.07, 5.06–6.42, 5.22–6.07. From Fig. 1 it is evident that use of cationic polyacrylamide did not have a marked influence on the pH of the raw olive mill effluent. With EWASCP and EPASCP solutions the optimum amount of alum was determined to be 2 g/L. The small increase in pH is due to the hydroxide ions formed by the hydrolysis of alum. Further increase

in the concentration of the coagulant/flocculant mixture made the solution more acidic. This is because of the acidic products formed by hydrolysis of the polyacrylamide polymer by the hydroxide ions in the solution [22]. For EWCHCP the dramatic pH increase is caused by the hydroxide ions from lime. Optimum flocculant/coagulant/precipitant dosage is essential as excess chemical additives will be expensive, pose health hazard and may also interfere with biological system in the water bodies.

3.3. Effect of flocculant/coagulant/precipitant on solution colour

The variation in colour reduction percentage with increase in the flocculant/coagulant/precipitant dosage is given in Fig. 2. Except for EPASCP the other three systems studied achieved maximum colour removal of about 72.3% with 8–10 g/L flocculant or coagulant/precipitant/flocculant combination.

The colour of OMW is predominantly because of the presence of tannins and phenolic compounds [23]. In the case of ECP with the initial addition of polymer flocculant, colour attributed to large suspended solids was removed. It was found to be more effective in colour removal (64.7%) with the addition of 4 g/L than with only alum on raw OMW (25%) [8]. Further addition increased the removal of organic compounds and dissolved solids in the effluent by reacting with the cationic group in the polymer. Increased nucleation rate and floc growth rate also contribute to the colour removal. For EWCHCP and EWASCP initial addition of precipitant lime and coagulant alum resulted in substantial reduction in the addition of the cationic

polyacrylamide flocculant without compensating colour reduction. This is because the coagulant converts the soluble substances in the OMW into insoluble particles which are then agglomerated using the polymer flocculant. In the case of EPASCP the total colour reduction achieved a maximum of about 50% considerably lower compared to the other cases. This is because of the higher initial concentration of colour content of the raw pig slurry due to high concentration of phenolic compounds (Table 1).

3.4. Effect of flocculant/coagulant/precipitant on solution COD

From Fig. 3 it can be seen that both flocculant and precipitant/coagulant/flocculant combination had significant effect in COD removal. The significance of the flocculants and coagulants diminishes with increase in concentration.

COD reduction is achieved by the removal of oxygen scavenging organic compounds in the wastewater. With the initial addition of 1 g/L of flocculant to raw OMW 40.6% decrease in the chemical oxygen demand was achieved. This decrease is attributed to the presence of strong reactive groups in the flocculant, which are able to stabilize and remove the oxygen scavenging organic compounds. Suspended flocs also enhance the removal of larger organic matter due to the availability of larger surface area on which adsorption of organic matter take place. Flocs encapsulation of organic matter and unreacted chemical species further enhance the removal process. It was observed that on addition of coagulants/precipitant with the flocculant decreased the rate of COD removal. For EWASCP, EWCHCP and EPASCP COD

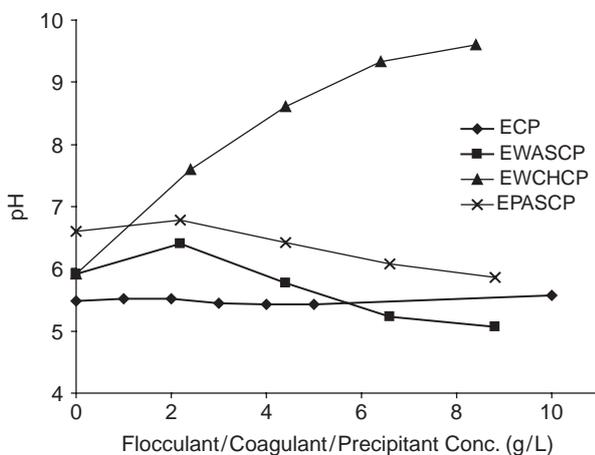


Fig. 1. Variation of pH with flocculant/coagulant/precipitant concentration for different effluent mixture (100 mL working volume, 20°C, 10 min mixing). ECP – Effluent and Cationic Polyacrylamide, EWASCP – Effluent and Whey mixture with Aluminium Sulphate and Cationic Polyacrylamide, EWCHCP – Effluent and Whey mixture with Calcium Hydroxide and Cationic Polyacrylamide, EPASCP – Effluent and Pig slurry mixture with Aluminium Sulphate and Cationic Polyacrylamide.

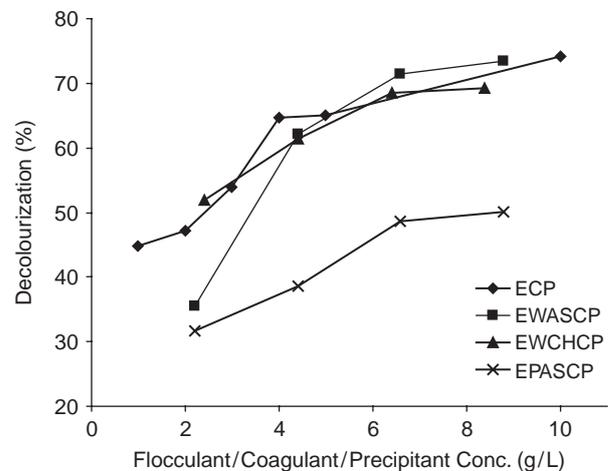


Fig. 2. Changes in percentage colour removal with flocculant/coagulant/precipitant concentration for different effluent mixture (100 mL working volume, 20°C, 10 min mixing). ECP – Effluent and Cationic Polyacrylamide, EWASCP – Effluent and Whey mixture with Aluminium Sulphate and Cationic Polyacrylamide, EWCHCP – Effluent and Whey mixture with Calcium Hydroxide and Cationic Polyacrylamide, EPASCP – Effluent and Pig slurry mixture with Aluminium Sulphate and Cationic Polyacrylamide.

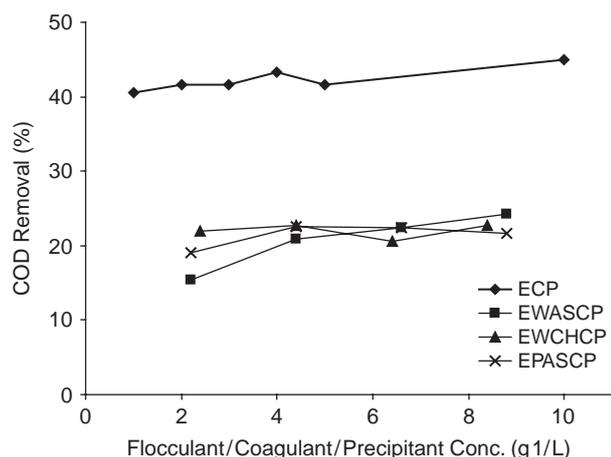


Fig. 3. Variation in COD removal with flocculant/coagulant/precipitant concentration for different effluent mixture (100 mL working volume, 20°C, 10 min mixing). ECP – Effluent and Cationic Polyacrylamide, EWASCP – Effluent and Whey mixture with Aluminium Sulphate and Cationic Polyacrylamide, EWCHCP – Effluent and Whey mixture with Calcium Hydroxide and Cationic Polyacrylamide, EPASCP – Effluent and Pig slurry mixture with Aluminium Sulphate and Cationic Polyacrylamide.

reduction was in the range of 15.5–21.9% with initial precipitant/coagulant/flocculant dosage. This lower removal is attributed to the lower initial COD due to aerobic pre-treatment of OMW with whey or pig slurry (62% COD reduction) than the raw OMW effluent.

3.5. Effect of flocculant/coagulant/precipitant on phenol and lipids

Reduction of phenolic compounds is a priority as phytotoxic effect of the raw OMW has been partially linked to the phenolic content [24]. It has been reported that these compounds inhibit the growth of bacteria [25]. Therefore reduction in these compounds is essential before anaerobic treatment or before discharge of effluent to water bodies. The phenol reduction curve follows similar pattern as that for COD with a 40.1% reduction with 1 g/L of polymer flocculant to raw OMW as shown in Fig. 4. With further increase to 10 g/L of flocculant there was a further 12.2% reduction in the concentration of phenolic compounds. This could be due to effective interaction of the cations of the polymer flocculant with the hydroxyl group of the phenolic compounds. The optimum concentration of flocculant was about 1 g/L.

Lipids are oily organic compounds which have been found to be toxic to methanogenic bacteria [26] and they are decomposed at a much slower rate than sugars or short chain volatile acids [3]. Reduction of these water insoluble compounds is essential before any biological treatment. Inorganic compounds such as alum and lime are ineffective in the removal of low lipid concentration [27]. However, with 1 g/L cationic polyacrylamide the

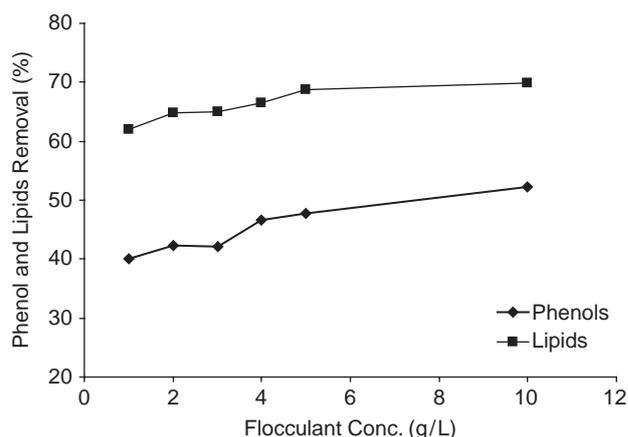


Fig. 4. Phenols and lipids removal percentage for OMW effluent treated with cationic polyacrylamide.

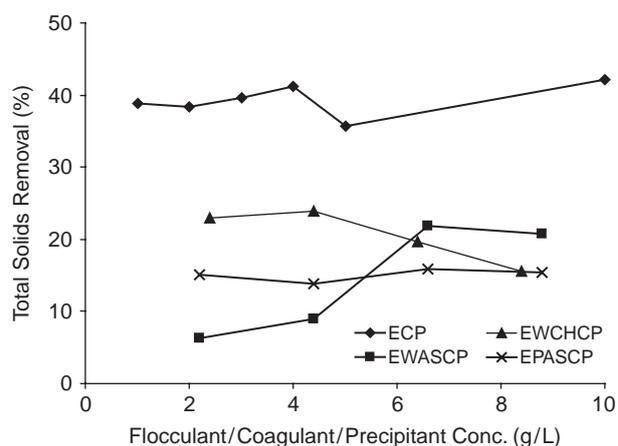


Fig. 5. Total solids removal with flocculant/coagulant/precipitant concentration for different effluent mixture (100 mL working volume, 20°C, 10 min mixing). ECP – Effluent and Cationic Polyacrylamide, EWASCP – Effluent and Whey mixture with Aluminium Sulphate and Cationic Polyacrylamide, EWCHCP – Effluent and Whey mixture with Calcium Hydroxide and Cationic Polyacrylamide, EPASCP – Effluent and Pig slurry mixture with Aluminium Sulphate and Cationic Polyacrylamide.

lipid content was dramatically reduced by 62%. Further increases in the flocculant concentration resulted in small reduction by 7.8%. The reductions are mainly due to the stable bond formation with the functional group of the lipids and by surface adhesion on the flocs. The optimum amount of the flocculant was about 1 g/L.

3.6. Effect of flocculant/coagulant on total solids

There was a marked reduction (38.8%) in the total solids content with 1 g/L flocculant. With further addition there was no significant reduction. The major reduction is caused by the presence of the negatively charged solids, which are adsorbed on to the flocculant by the process of polymer bridging. Here the polymer forms bridges across

the negatively charged solids thereby forming flocs. The lack in further reduction of the solids content may also be due to the presence of positively charged solids or the limitation of the flocculant. For EWASCP, EWCHCP and EPASCP the solids reduction was in the range of 6.4–23.9% (Fig. 5). Solids are normally destabilized by the coagulant (alum), which is then bonded together by the polymer flocculant cationic polyacrylamide. For EWCHCP, with the addition of lime notable reduction in solid content was observed. However, further addition resulted in a negative influence. The decrease in the solids removal with increase in the precipitant/coagulant/flocculant mixture could also have been caused by experimental anomaly.

4. Conclusion

Results of the present work indicated that addition of coagulant/precipitant and polymer flocculant mixture to aerobically treated wastewater and polymer flocculant alone to raw OMW was able to reduce the pollutant load significantly. Maximum colour, COD, total solids, phenol & lipids removals achieved were 74.1, 45, 42, 52.3 and 69.9%. Chemical treatment cannot replace biological processes, where a significant amount of COD reduction can be obtained. This is because firstly, the COD reduction required is greater than 80% and secondly excessive sludge is produced containing complex and toxic chemical compounds. However, optimised and right combinations of chemicals could provide an effective preliminary treatment in wastewater treatment processes.

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