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Partial purification of iron solutions from ripe table olive processing using ozone and electro-coagulation

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ABSTRACT

This study investigates the application of electro-coagulation and ozonation technologies for the partial depuration of ferrous solutions deriving from the color fixation stage of ripe olive processing. Different operational conditions were investigated along with the possibilities of combining the two techniques sequentially. In electro-coagulation the best depuration conditions were obtained using a current density of 25 mA cm^{-2} for 40 min; COD elimination reached 30%, the phenols and color of the solutions were also considerably reduced and the dissolved Fe was completely removed. On the contrary, the microbial population mainly composed of lactic acid bacteria and yeasts, hardly decreased. The application of ozonation also reduced the total phenols, color and COD of solutions, but did not decrease the iron concentration. However, it considerably affected the microbial population in a higher proportion than electro-coagulation. In light of the results obtained, the best working conditions consist of applying 40 min electro-coagulation with aluminum in the anode and iron in the cathode at 25 mA cm^{-2} current density, followed by the storage of a partially purified solution bubbling ozone to reduce the microorganism population and even eliminate it completely when 6 g O_3 L^{-1} were added. This would allow for a possible reuse of these solutions for preparing new fixing color solutions or as cover brine in packaging.

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1. Introduction

Olives darkened by oxidation [1] are commonly known by their original American name: ripe olives. The production of this preparation was reached around 630,000 tons in the 2011/2012 season, 30% of the world's table olive production [2].

The darkening process consists of successive treatments of the fruits with a dilute solution of NaOH (lye); during the intervals between lye treatments the fruits are suspended in water through which air is bubbled [3]. Nowadays, a preservation solution is added to the tap water in this phase of oxidation [4]. Throughout this operation the fruits darken progressively, but the color formed is not stable permanent and fades progressively after oxidation; to prevent this deterioration, the fruits are immersed in ferrous lactate or gluconate for several hours [5]. The product has a final pH above 4.6 and its preservation is only achieved by sterilization [3].

Electro-coagulation (EC) is an electrochemical method consisting in treating polluted water whereby sacrificial anodes dissolve to produce active coagulant precursors (usually aluminum or iron cations) in the solution. The generation of coagulants in situ means that EC process does not require the addition of any chemicals. The gases produced at the cathode during the electrolysis of water and metal dissolution allow the resulting flocks to float [6].

EC has been successfully used in the treatment of wastewaters from food industries such as alcohol distillery [7], dairy [8], olive oil mill [9] and oil refinery [10]. In green table olive processing, the combined biological and EC treatments of the debittering solution (lye) and washing water reduce the chemical oxygen demand (COD) up to 97% [11]. Also, EC application to wastewaters from the packaging industry results in a practically colorless solution of lactic and acetic salts which could be reused in packing [12]. In natural black table olives, the EC of wastewaters by boron-doped diamond electrodes led to 73% COD removal [13]. However, this technology has not yet been studied for the treatment of effluents from the darkening process of ripe olive industries.

Ozone has been declared Generally Recognized as Safe (GRAS) and it was admitted as a direct food additive [14,15]. The treatment of wastewaters with ozone from the table olive industry [16] and olive oil extraction process [17] lightened the final color of the solutions due to destruction of the brownish polyphenol polymers.

The green olive alkaline solutions (lye and washing waters) treated with ozone may be reused as fermentation brines after

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adding the appropriate NaCl quantity [18,19]. Also, Spanish green olives may be packed with diluted (1:1) ozonated fermentation brines with similar organoleptic characteristics to those using fresh brine [20]. In ripe olives, preservation solutions can be reused in a new preservation cycle after ozone treatment, the resulting quality of the final product being similar to the ones stored in a fresh acid solution [4].

Color fixation solutions in the darkening process could be reused after the addition of the required quantity of ferrous gluconate if the organic matter, mainly phenols, is removed. These compounds can be chemically bonded to the ferrous ions when adding more ferrous gluconate and preventing the cations penetrating into the olives, forming the Fe–phenol complexes in the liquid and the color of olive not being fixed [5].

In the present work, the use of EC and ozonation to treat ferrous gluconate solutions from ripe olive processing was studied. Particularly, the effect of various operating conditions (current intensity and contact time of solutions in EC or amount of O_3 bubbled) on the elimination of COD, iron, phenols, color and microorganism survival was investigated. This information is essential both to assess the possibility of reusing these solutions by the industry and also for scaling up the treatment at the industrial level.

2. Experimental

2.1. Ferrous solutions

Ferrous gluconate solution samples were collected for the darkening processes of ripe olives carried out at the Instituto de la Grasa pilot plant (CSIC, Spain) similarly to what industrial processes are [3]: relationship olives/liquid, 1/1; lye treatment (2.5% NaOH, 5 h to reach stone); first washing only tap water (20 h); second washing (tap water/preservation solution, 1/1, 24 h), ferrous gluconate solution (0.1%, 20 h).

The initial physico-chemical and microbiological characteristics of the solutions are shown in Table 1. The high population of microorganisms, especially lactic acid bacteria (LAB) and yeasts were found in these solutions due to the preservative liquid reuse in washing during oxidation [4]. This is the first time that this fact is mentioned in a publication.

2.2. Electro-coagulation procedure

The EC was carried out in a plexiglas parallelepiped reactor (2.0 L). Two pairs of Al/Fe (anode/cathode) electrodes connected in a bipolar mode were placed in the electrochemical reactor, each one with dimensions of 13 cm × 6.5 cm × 0.2 cm; the distance between the lowest part of the electrode and the cell was 0.8 cm and between electrodes was 1 cm; the total effective surface area of the electrodes was 91 cm^2. Prior to each experiment; the electrodes were immersed in 1% HCl for 12 h and rinsed with distilled water.

The volume of liquid treated each time was 1.6 L. The liquid was magnetically stirred by two bars, allowing a correct homogenization of the wastewater. A direct current was imposed by a stabilized power supply (Quasar 500, CRS Industrial Power Equipment, Calco, Lecco, Italy) for 1 h and the range of current density variations was 10–75 mA cm^{-2}. The temperature was monitored with a thermometer and maintained stable (20–25 °C) during the experiments by cooling the solution with a cooling jacket. All experiments were performed in duplicate and samples (10 mL) were withdrawn at 5 min intervals. Before their analysis, the samples were centrifuged to remove any suspended solid.

The electrical energy consumption per unit mass of organic load removed (*SEEC*) was calculated using the following equation:

$$SEEC = VIt/(C_0 - C)Q \tag{1}$$

where V, voltage in volts; I, current in amperes; t, time in hours; Q, volume of wastewater in liters; and $(C_0 - C)$, pollutant load removed in g O_2/l.

2.3. Treatment with ozone

The ozone was produced by TODOZONO1, Mod TD ZN equipment (Colmenar Viejo, Madrid, Spain). Aliquots of 2.0 L of solution were put in a cylindrical test tube (52 cm × 8.5 cm diam.). The solutions were then treated by bubbling ozonated air with an ozone concentration of 3.63 mg L^{-1} at a flow rate of 200 L/h (3.63 g O_3 h^{-1} L^{-1} of solution) through a synthetic glass diffuser introduced at the bottom of the tube. The pH of the solution was allowed to evolve freely. All experiments were performed in duplicate and samples (10 mL) were withdrawn at 1 h intervals.

The electrical energy consumption was estimated from the power consumption indicated by the manufacturer of the ozone-producing equipment (50 ***w***) and the time (*h*) to produce a given amount of ozone in g (*Q*) per unit volume of solution (L).

$$E(\mathrm{kW\ h/L}) = 50 * 10^{-3} * Q * (1/3.63) = 0.0137 * Q \tag{2}$$

2.4. Experimental design

The effect of various EC operating conditions (current intensity and contact time) and bubbled O_3 quantity during ozonation treatment on the elimination of COD, iron, phenols and color, was

Table 1
Initial characteristics of ferrous gluconate solutions and after tested treatments to remove pollution. Electric power and aluminum consumptions.

Treatments	COD (mg O_2 L^{-1})	Total phenols (mg L^{-1})	Color (A_{440}–A_{700})	Iron (mg L^{-1})	Microorganism population ($\log_{10}$ CFU mL^{-1})		Electric consumption (kW h m^{-3})	Consumed aluminum (g m^{-3})
					LAB	Yeast		
Initial[a]	4700(88)[e] a[f]	171 (9) a	1.05 (0.08) a	51 (5)a	6.5 (0.3) a	5.5 (0.3) a	–	–
EC[b,c] (40 min)	3300 (94) c	80 (7) b	0.27 (0.05)c	0 b	6.3 (0.2) a	5.3 (0.1) ab	6.5	0.89
Ozone[b] (5 g O_3 L^{-1})	4200 (97) b	55 (8) c	0.46 (0.04) b	51 (4)a	5.2 (0.3) b	4.9 (0.1) b	68.5	–
Ozone + EC[b,c] (5 g O_3 L^{-1}) + (30 min)[d]	3190 (99) c	30 (12) d	≈0 d	0 b	4.5 (0.2) c	4.4 (0.2) c	68.5 + 7	2.5
EC[c] + Ozone[b] (40 min) + (6 g O_3 L^{-1})[d]	3200 (95) c	15 (5) d	≈0 d	0 b	0 d	0 d	6.5 + 82.2	0.89

[a] Averages of samples collected from 6 different darkening process.
[b] Averages of two experiments.
[c] EC at 25 mA cm^{-2} current density.
[d] Time (min) by EC and bubbled O_3 (g L^{-1}) by ozonation.
[e] Standard deviation in parenthesis.
[f] Column values followed by the same letter do not differ at the 5% level of significance according to Duncan's multiple-range test.

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