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Effects of ultrasonic and hydrodynamic cavitation on the treatment of cork wastewater by flocculation and Fenton processes

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Dedicated to Professor Bernd Ondruschka on the occasion of his 70th birthday

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ABSTRACT

This paper reports that ultrasonic (US) and hydrodynamic cavitation (HC) are efficient strategies for the environmental remediation of cork wastewater (CW). It is necessary to remove toxic, inhibitory or refractory organic matter from CW using physical and chemical techniques (pre-treatment) prior to performing conventional biological treatment. After this biological treatment, it is also critical to further decontaminate (post-treatment) in order to meet the discharge limitation. The pre-treatment of diluted CW using Fenton oxidation (FE) alone led to COD and polyphenol (PP) removal values of 30% and 61%, respectively, while HC and US resulted in 83–90% increases in COD reduction and 26–33% increases in PP reduction. Whereas 55% and 91% COD and PP removal were achieved using flocculation (Floc) alone, COD elimination was increased by a further 7–18% under HC and US. No noticeable improvement in PP elimination was observed. US did not enhance the Floc decontamination of the original concentrated CW, however, considerable quantities of white biofilm were surprisingly generated on the CW surface after the pre-treatment, thus indicating the improvement of biodegradability of the resulting liquid. In fact, the post-treatment stage, using FE alone after having filtered the biofilms, led to reductions of 53% in COD and 90% in PP. The HC and US protocols resulted in 26–34% increases in COD elimination. HC further enhanced PP elimination caused by FE, while US resulted in lower levels of PP elimination.

1. Introduction

Cork is essentially composed of suberin, which is a hydrophobic, waxy substance present in the walls of the cells, making them resistant and impermeable to water. Cork oak (*Quercus suber*) is native to the Mediterranean area, yet is also cultivated in the United States and India with an annual production of around 200,000 tons worldwide with Portugal (ca. 50%) and Spain (ca. 30%) being the major producers of the actual cork stopper [1]. Although cork is indeed a versatile material that has found widespread use from cloth to furniture, the market is dominated by the production of wine stoppers. Cork wastewater (CW) comes from the cooking of large, curved planks which are immersed in boiling water for one hour in order to improve their physico-chemical characteristics. The immersion water can be generally used for repeated boiling cycles (10–30 times). CW is a black liquor containing a complex mixture of organic substances, including both small molecules and

macromolecules, which often show colloidal behavior, making the degradation and separation processes quite difficult. The polluting nature of CW is readily noticeable in view of its high chemical oxygen demand (COD: ~6500 mg L⁻¹) and polyphenol content (PP: ~1000 mg L⁻¹), which indicate the level at which CW inhibits the activity of microorganisms [1–3].

The toxicological properties of these effluents and the high volumes produced (~1200 L ton⁻¹ cork) [1], mean that they must be treated by sophisticated and tedious processes prior to their delivery for human consumption [2,4]. While there is no doubt that the high content of organic matter in cork wastewater and in the effluents of numerous other industries, such as food and paper, is a serious environmental issue, there is, however, a clear increase in interest in reusing water pollutants for nutrient recovery and energy savings [5].

The process of flocculation (Floc), or coagulation, using common inorganic salts, such as aluminum and ferric chloride, has been known

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as a means for removing total solids (TS) and colloidal matter from CW for some time [6,7]. In this process, COD reduction (30–65%) and PP removal (70–90%) can be achieved using Floc [8], however, the remaining contaminants inhibit biological treatment [2]. Ultrasonic cavitation (US) technology has proven itself to be a suitable tool for combination with traditional suspension separation methods [9]. Likewise, a US-chitosan enhanced Floc process for treating algal-bloom-related turbidity in water has also been developed. Turbidity removal (~99%) for the US/Floc of algae was comparable to those of the conventional rapid mix coagulation process, proving that ultrasonic mixing can be used to improve turbidity removal [10–12]. Power densities in the range of 100–250 W L⁻¹ are sufficient to provide adequate mixing for chemical distribution and flocs formation.

The US driven particle Floc process is a well-known and useful technique for solid-liquid separation and usually works at MHz frequencies. This separation, however, is generally only applicable to particles with diameters similar to, or smaller than, the ultrasound wavelength, as particles are trapped at the pressure nodes or anti-nodes of the standing wave. The flocculation of larger particles (mm-sized) is therefore difficult to achieve using MHz-range ultrasound and would require lower frequencies, although studies into this are scarce and often lead to contradictory interpretation. A recent study, however, showed that the successful Floc of large particles in water at 20.3 kHz most likely arises from a different mechanism involving the adhesion of oriented bubbles to particles, in the acoustic field, which then move to the anti-nodes of the standing waves [11,13,14].

The enhanced, and sometimes synergistic, effects of sonochemical and sonophotocatalytic methods are much more pronounced when used in tandem with advanced oxidation processes (AOPs), such as Fenton oxidation (FE), O₃/UV and O₃/H₂O₂ [15–18]. Moreover, combined AOPs, such as hybrid FE/Floc and Photo-FE processes, have been successfully tested for the COD reduction of CW and have achieved up to 90% COD reduction [19,20]. The maximum degradation (66.4%) of 0.5% (v/v) *p*-nitrophenol was observed using a combined US/FE process [21].

In principle, AOPs whose effectiveness is based on the transient formation of reactive oxygen species can be boosted under cavitation collapse [18,21]. The extreme, localized conditions that occur under bubble collapse – temperatures as high as 4500–5000 K and pressures higher than 1000 atm – cause mechanical and chemical effects that can induce numerous physico-chemical changes. Mechanical effects arise from the turbulent flow, microjets and shear forces which occur at solid-liquid interfaces. The chemical effects are related to the direct pyrolysis of volatile substrates in the collapse cavities [22] and the highly reactive species [23] such as hydroxy (OH) and hydroperoxy (HO₂) radicals and hydrogen peroxide (H₂O₂), which are formed by sonolytic degradation in liquids containing O₂, O₃, and H₂O₂.

Besides ultrasonic cavitation, hydrodynamic cavitation (HC) has also been widely investigated, since 2000, as a means to treat organic wastewater [24]. HC bubbles are generated at large pressure differentials that are induced by an orifice plate, venturi, impeller or rotor within a moving liquid. The collapse of bubbles generates localized hot spots and is accompanied by a number of physical effects, including emulsion and erosion. HC can also bring about a number of sonochemical effects, such as the formation of active oxidants and the oxidative degradation of organic pollutants [25]. The cavitation intensities generated in HC reactors are lower than in their acoustic counterparts, but cavitation yields are higher than in all sonochemical equipment (ultrasonic horn, ultrasonic bath, dual frequency flow cell) [24,26]. Moreover, the energy efficiency (ratio of the energy transferred into the system in relation to supplied electrical energy) is higher in HC reactors [27]. Several reports have documented the efficiency of HC in the degradation of phenolic effluents in the oxidation, ozonation [23,28,29], and FE processes [30–33].

Scaling effects have also been reported; whereas a pilot-scale operation using HC/FE and an initial *p*-nitrophenol concentration of

5 g L⁻¹ gave a maximum removal value of 63.2%, the same method only led to 56.2% degradation in solutions containing 10 g L⁻¹ of *p*-nitrophenol [34]. The effect of HC combined with chemical oxidation processes, such as hydrogen peroxide (HC/H₂O₂), ferrous activated persulfate (HC/Na₂S₂O₈/FeSO₄), HC/FE (HC/FeSO₄/H₂O₂), advanced Fenton (HC/Fe/H₂O₂), and Fenton-like process (HC/CuO/H₂O₂), have all been investigated in the degradation of 2,4-dinitrophenol (DNP) at optimized conditions of pH 4, 35 °C and an inlet pressure of 4 bar. The experimental results showed that the HC/FE process is more feasible and successful than HC combined with other Fenton-like processes and that it furnishes complete degradation [35–37]. Accordingly, it is fair to say that the US/FE and HC/FE processes are efficient and widely-applicable technologies for removing phenol and phenolic compounds from aquatic effluents.

Cork wastewater generally contains high concentrations of organic matter and impurities which are toxic, inhibitory or refractory for subsequent biological systems [3]. Pre-treatment is therefore necessary to remove the majority of harmful components prior to performing biological treatment. The high initial concentration of organic matter means that it is difficult to meet the discharge standard using pre-treatment and biological treatment alone. It is thus critical that post-treatment is carried out after biological treatment. We report herein the preliminary results of the pre- and post-treatment of several CW batches from a large cork industry with Floc, US/Floc, HC/Floc, FE, US/FE and HC/FE. The extent of COD and PP removal has been tested in order to evaluate the effects of US and HC on the Floc and FE processes.

2. Materials and methods

2.1. Chemicals and materials

Reagent grade FeSO₄·7H₂O (Riedel-de-Haën), 35% H₂O₂ (Sigma-Aldrich), 96% H₂SO₄ (Carlo Erba) and NaOH (98% purity) pellets (Carlo Erba) were obtained from commercial suppliers and used as received.

The original CW, which displayed a high concentration of organic pollutants, was provided by CICYTEX in Mérida (Spain). Samples used throughout this study were diluted in deionized water. The COD, PP, TS and total suspended solids (TSS) content ranges in the original CW were found to be 5.0–6.5 g L⁻¹, 943–1088 mg L⁻¹, 2.0–2.8 g L⁻¹ and 1.0–1.4 g L⁻¹, respectively, at pH values of 4–6.

2.2. Experimental setup

Stirring: Stirring was performed at 500–800 rpm, either at room temperature or 30 °C, using an AREX heating magnetic stirrer (VELP Scientifica, Italy).

US devices: 400 and 800 mL CW samples were sonicated at 30 °C in a REUS-US (France) cup horn at 25 kHz and total input power of 150 W. A cooling jacket with circulating water allowed for good temperature control, as described elsewhere [38]. An Elma US bath (Transsonic 460, 35 kHz, 85 W, 2 L) was used to prepare the CW samples from the original CW using US/Floc for post-treatment.

Seeing as the HC reactor has a fixed volume of 800 mL, this volume was selected for US treatment. While a 400 mL solution volume (higher power density) was used to optimize the operative parameters for Floc and US/Floc treatments.

HC device: The homemade lab-scale loop-system, which consists of a centrifugal pump, manometers, a flow-meter, a water-reservoir and a heat exchanger as well as being equipped with online instrumentation for measuring conductivity and temperature has been reported previously [32,39]. The core component; the centrifugal pump (GY-028-2, 2750 rpm, ~0.7 MPa, ~1700 L h⁻¹), was provided by Speck Pumpen, Walter Speck GmbH & Co. KG (Roth, Germany). HC studies were conducted on 800-mL samples in suction mode with 6 mm throat Venturi at 500 W and 30 °C. A schematic of the 6 mm throat Venturi

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