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Research article

Screening of physicochemical treatment processes for reducing toxicity of hair care products wastewaters

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ABSTRACT

Toxicity reduction in wastewaters from small hair care products manufacturing companies using coagulation/flocculation/sedimentation or flotation, membrane separation and powdered activated charcoal adsorption was evaluated. Raw wastewater composition varied widely within and especially between companies, but all exhibited high acute toxicity to *Daphnia similis* (EC(I)50; 48 h < 0.02–0.33%). Coagulation with aluminum sulfate and polyaluminum chloride aided by cationic or anionic polymers, as well as filtration on ultra (UF) and nanofiltration (NF) membranes efficiently removed turbidity (>99%) and oil and grease (>99%) and all treated samples exhibited similar dissolved organic matter contents. However, elimination of acute toxicity was only achieved after UF on submerged hollow fiber membrane, while other membrane modules (tubular UF and NF) produced filtrates with residual toxicities equal to or higher than the wastewater samples treated by coagulation processes. Adsorption removed up to 90% of the soluble COD remaining after coagulation or membrane processes, but did not eliminate acute toxicity, possibly because of the presence of activated charcoal or substances leached from it in the treated samples. The results indicate the need for further studies to develop treatment strategies that can guarantee non-toxic effluents at costs compatible with those of the simple manufacturing processes used at small-scale cosmetics manufacturing plants.

1. Introduction

Brazil is the world's fourth largest hair care products consumer market, with total annual sales of over 29 billion dollars in 2016 (ABIHPEC, 2017). The relative simplicity of hair care products batch preparation processes has led to the pulverization of production among thousands of small-scale enterprises. For example, Minas Gerais State is home to over 230 cosmetic manufacturing plants (ABIHPEC, 2017), most of which operate under minimum environmental supervision, especially with regard to wastewater discharges.

Cosmetic products wastewaters composition depends on the type of product being prepared, but in general includes high COD and concentrations of organic compounds with low biodegradability, such as surfactants, oils and grease, dyes, fragrances and preservatives (Bautista et al., 2007; El-Gohary et al., 2010; de Melo et al., 2013; Perdigón-Melón et al., 2010). Several studies have been published in recent years on the potential of both conventional and advanced physicochemical processes for treatment of cosmetic industries effluents (Aloui et al., 2009; Banerjee et al., 2016; Bautista et al., 2007; Boroski et al., 2009; Bradai et al., 2012; El-Gohary et al., 2010; Monsalvo et al., 2014; Perdigón-Melón et al., 2010), however most did not specify the type of products (hair care, skin care, etc.) the wastewater originated from nor did they address wastewater toxicity before or after treatments.

Wastewater treatment at small hair care products plants in Minas Gerais, when it exists, usually involves conventional physicochemical processes (coagulation - flocculation - sedimentation - adsorption). In a previous study (de Melo et al., 2013), wastewaters from a small-scale hair care products company were shown to continue to exhibit very high acute and chronic toxicities after such treatment. Phase I of the US EPA's toxicity identification evaluation (TIE) protocols (USEPA, 1991) indicated that non to moderately polar, volatile or sublatable organic compounds caused the toxicity and toxicity levels correlated with wastewater COD, suspended solids and surfactants contents. Therefore, this study was undertaken to evaluate the potential for toxicity reduction using established physicochemical processes, including coagulation/flocculation, membrane separation and adsorption, that are already

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used or would be most readily accepted at small hair care products companies. Less consolidated processes, such as advanced oxidation, were not considered for this same reason.

2. Material and methods

2.1. Wastewaters

Raw wastewater was collected at two small-sized manufacturers located in Minas Gerais, Brazil that produce various hair care products, including shampoos, conditioners, oils, lotions, mousses and masques in batch processes. The companies were selected based on the variety of products they manufactured, their positive and proactive attitude towards improving effluent treatment, as well as the logistics involved in sample collection. Company A employs 48 workers and consumes approximately 50000 L of industrial water per month, while company B has five employees and a monthly industrial water consumption of approximately 20000 L. The types and volumes of the different products produced vary throughout the year according to market demands. Therefore, wastewater generation, treatment (present only at company A) and release to the environment is intermittent, without any routine operational control of water use or effluent flow to receiving waters.

Wastewater sources include mixing tank wash waters, packaging operation losses, laboratory wastes, disposal of raw materials and products past their expiration dates and of spent solutions from regeneration of the ion exchange columns used to prepare deionized water for product preparation. Sanitary sewage is not mixed with industrial effluents at either company.

Given the variability of wastewater characteristics because of the variable production demands, three random samples were collected at each company over the course of a year. The interval between sample collection was defined by the time required to carry out the numerous analyses in each sample. Company A samples were collected from the holding tanks used to store wastewaters between periodic batch physicochemical treatment while at plant B samples were combined wastewaters from one day's production, since this company has no wastewater treatment system in place. Upon arrival in the laboratory, samples were sieved (1 mm opening) to remove coarse solids, homogenized and stored frozen in 20-L containers until used. Fig. 1 illustrates the experimental design adopted for screening the treatability of wastewaters by physicochemical processes in bench-scale treatments. The acronyms used to identify wastewater treatments evaluated in this study are listed in Table 1.

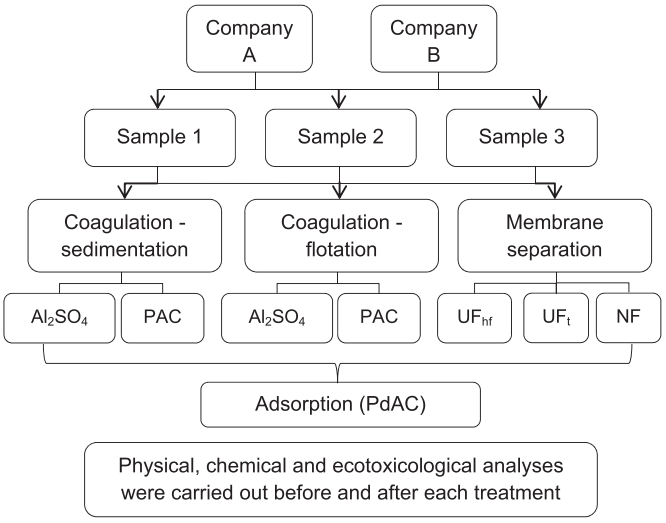

Fig. 1. Experimental design adopted for screening of physicochemical processes for toxicity reduction in hair care products manufacturing plants A and B raw wastewaters. (PAC = polyaluminum chloride; PdAC = powdered activated charcoal).

2.2. Coagulation-flocculation processes

Coagulation-flocculation-sedimentation (CFS) experiments were performed in a jar test apparatus (model JT-203, Milan-EC) using the following constant conditions: mixing for 60 s at 120 rpm ($G = 110\ s^{-1}$), followed by flocculation for 15 min at 50 rpm ($G = 30\ s^{-1}$) and sedimentation for 45 min, without mixing. When necessary, sample pH and alkalinity were adjusted immediately before mixing and coagulants were then added. When used, flocculation aids were mixed slowly to the solutions in the jars after rapid mixing.

Coagulation-flocculation-dissolved air flotation (CFF) experiments were performed using a bench-scale system (model 218/LDB, Ethik Technology) composed of a saturation chamber and a set of three acrylic jars, with bottom inlets for air-saturated liquid. The saturation chamber (model 218-3, Ethik Technology) contained water and compressed air inlets, a pressure gauge and a pressure regulator valve. Liquid saturation conditions were fixed at a pressure of 5 bar for 15 min, while mixing and flocculation conditions were the same as those used in the CFS tests. Recirculation rate for distillated water was set at 10% v/v. Both CFS and CFF were conducted at room temperature (22-28 °C).

Constant conditions were used in all tests since the purpose was not to optimize mixing, flocculation, sedimentation or flotation parameters, but rather to study potential of these treatments for reducing wastewater toxicity through removal of organic matter. The potential of the different treatments was readily compared because the operating conditions were held constant, although no overall optimization was attempted.

Aluminum sulfate (AS, iron free, 47% aluminum sulfate and approximately 7.5% Al_2O_3, Quimisa S/A) and polyaluminum chloride (PAC, 23.5% chloride and 16.95% Al_2O_3, Colina Química Nacional Ltda.) were used as coagulants, while commercial high molecular weight anionic (W 1010) and medium molecular weight cationic (M1009) polymers (powdered, 100% active ingredients, Quimisa S/A) were used as flocculation aids. Lime (CaO, 0.18 N) or hydrochloric acid (HCl, 0.1 N) solutions were used for pH adjustment when needed. AS and PAC were selected since they are the most commonly used coagulants in the Brazilian industrial sector. Stock solutions were prepared at the same Al_2O_3 concentration (15.5 g/L) to facilitate comparison of applied doses, while cationic and anionic polymer stock solutions of 1 g/L were used due to their high viscosity.

Initially, experiments were conducted with coagulants (AS or PAC) or flocculation aids (anionic or cationic polymers as primary coagulants) alone, followed by tests with coagulants (AS or PAC) and polymers (anionic or cationic) combined. The test conditions (initial pH x doses) that produced the greatest turbidity reductions and most compact sludge layers for each coagulant in CFS treatments were triplicated to produce sufficient sample volumes for complete physical, chemical and ecotoxicological analyses. These same conditions were also employed for the CFF treatments. Sludge volumes produced using the selected CFS and CFF conditions were quantified by direct measurement in the reaction jars.

2.3. Membrane separation processes

Performance of two ultrafiltration and one nanofiltration membrane (Table 2) was evaluated. The UF_{hf} module (PAM