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Impacts of main parameters on the regeneration process efficiency of several ion exchange resins after final purification of olive mill effluent

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The key for the cost-efficient application of ion exchange (IE) processes relies on the fact that resins should be effectively regenerated to ensure their operation for the maximum possible working cycles. The main objective of this research work was to examine the effect of the main parameters affecting the regeneration process of several IE resins previously used for the purification of secondary-treated olive mill wastewater (OMW-2ST), comprising regenerant concentration and type, operating temperature and flow rate. For this purpose, the impacts on the regeneration and rinse times as well as the regeneration efficiency were addressed. Results show regeneration time decreased but rinse time increased with regenerant concentration increment for all resins. As a general rule, minor flow rate (2.5 L h^{-1}) provided the highest possible regeneration, whereas efficiencies above 96% were noted upon the lowest temperature examined (298 K), which have both important implications on the cost-efficiency of the regeneration and integral effluent treatment process. Finally, 0.6 L HCl 4% and 0.8 L H_2SO_4 2% solutions achieved complete regeneration of Dowex Marathon C and Dowex MAC 3 resins, whereas 0.7 L and 1 L NaOH 4% ensured complete regeneration of Amberlyst A26 and Amberlite IRA-67 resins, respectively.

1. Introduction

Industrial wastewater is subjected to strict environmental legislation, thus making its adequate management a key issue. In this regard, there is a plethora of technologies described in the scientific literature regarding wastewater treatments. Within this context, novel IE resins developed over the past few decades have promoted this technology as a suitable separation and purification process for wastewater treatment [1–3].

Ion exchange (IE) is an adsorption process whereby a target ion in the aqueous phase is transferred to a solid phase, mainly synthetic resin, and exchanged with a like-charged presaturant ion that diffuses into solution [4]. Because many aqueous contaminants are charged, IE is used to remove numerous contaminants from water and wastewater including several heavy metals and anionic pollutants [5].

During IE processes the effluent to be purified passes through a fixed bed filled with IE resin. The key for the cost-efficient implementation of an IE process is to ensure that the IE resins can be easily and effectively regenerated, in order to guarantee their operation for the maximum possible working cycles [6]. For that purpose, IE processes must be stopped periodically and the IE resin regenerated. In the regeneration step the resin is returned to its original state and the ions that had been substituted on the resin go back into solution. Cationic resins are usually regenerated by using a concentrated acid solutions, and anionic resins by means of a concentrated base. The net result is a solution that is much more concentrated than the feed to the system [7]. For many substances, such as heavy metals, this is usually the first step in a recovery process. For others, it is merely a mean of concentration before ultimate disposal occurs.

Hydroxide (OH^-) and chloride (Cl^-) as well as hydrogen ion (H^+) and sodium (Na^+) are the most commonly used presaturant ions in anion exchange and cation exchange processes, respectively, because they readily exchange with contaminant ions [7,8]. As a result, highly concentrated base or acid solutions in one hand or sodium chloride (NaCl) solution in the other hand are required to regenerate contaminant-saturated (i.e., exhausted) IE resin. In the combined systems comprising cationic resins and anionic resins in series, using acid such as HCl and base like NaOH as regenerants would give as a result the formation of H_2O in the regenerant outlet stream, thereby facilitating the disposal of final regenerant waste. This fact presents many benefits from an environmental point of view. In this case, both acid and base are used in excess quantities. Meanwhile, the advantages of NaCl as a regenerant

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include high aqueous solubility, low human toxicity, and low cost when compared to the alternate regeneration chemicals considered in this study. There are, however, recognized disadvantages associated with the disposal of waste streams high in NaCl. For instance, high strength NaCl solution that is sent to a wastewater treatment plant inhibit biological processes [9] and increase the NaCl content of receiving waters, which in turn have adverse impacts on aquatic organisms and ecosystems [10].

Different regenerant solutions have been used for cation and anion exchangers. For instance, Millar et al. used hydrochloric acid solutions to regenerate strong-acid cation exchange resins [11]. Also, Marañón et al. regenerated a series of cation and chelating resins using hydrochloric acid solutions [12].

On the other hand, Caetano et al. used NaOH and methanol/water solutions for MN200 anion exchange resin regeneration [13]. In addition, Sowmya and Meenakshi (2014) studied the regeneration of a novel quaternized anionic resin, by using NaCl solution [14].

In previous research works, the final purification of olive mil wastewater coming from the two-phase continuous decanting process (OMW-2) was carried out through IE system comprising several combinations of two resins in serial connection: one cationic resin and the other one anionic resin [15,16]. This effluent is a hardly treated by-product generated during olive oil production which constitutes an important environmental problem, since its disposal into watercourses leads to deterioration of natural water bodies, pollution and environmental degradation [17]. Previous to this final treatment by means of IE technology, OMW-2 was conducted to a secondary treatment based on Fenton reaction-advanced oxidation process followed by a flocculation–sedimenta tion step and filtration-in-series through different filtration medium. This pretreatment was able to remove the organic and phenolic pollutants of OMW-2. However, high levels of monovalent ions, mainly sodium and chloride, as well as iron, were found in the pretreated effluent, derived from the dosage of the catalyst and neutralizing agents during the secondary treatment. Furthermore, a significant phenols amount above maximum legislated standard level was present in this secondary-treated olive mill wastewater (OMW-2ST) [18].

The goal of this research study was to examine the effect of the main parameters affecting the regeneration process of several IE resins - Dowex Marathon C strong-acid cation exchange resin, MAC 3 weak-acid cation exchange resin, Amberlyst A26 strong-base anion exchange resin and Amberlite IRA-67 weak-base anion exchange resin - previously used for the purification of OMW-2ST. For this purpose, the impacts on the regeneration and rinse times as well as the overall regeneration efficiency of the IE resins were addressed as a function of the regeneration operation variables, comprising the regenerant concentration and type, the operating temperature and the flow rate.

Table 1
Physicochemical properties of selected IE resins (from manufacturer specifications) and experimental data [19,20].

IE resin	Dowex mac 3	Dowex Marathon C	Amberlite IRA-67	Amberlyst A26
Resin type	Weak acid-cation	Strong-acid cation	Weak-base anion	Strong-base anion
Matrix	Polyacrylic, macroporous	Styrene-DVB, gel	Crosslinked acrylic gel	Styrene-divinylbenzene
Functional group	Carboxylic acid	Sulfonic acid	Tertiary amine	Quaternary ammonium
Ionic form as shipped	H^+ form	H^+ form	OH^- form	OH^- form
Ion exchange capacity[a], eq L^{-1}	3.8	1.80	1.60	0.80
Water content, %	44–52	50–56	56–64	66–75
Shipping density, g L^{-1}	750	800	700	675
Maximum experimental adsorption capacity, mg g^{-1}	11.3	28.0	7.5	22.0

[a] Ion exchange capacity is the minimum wet capacity as specified by the manufacturer.

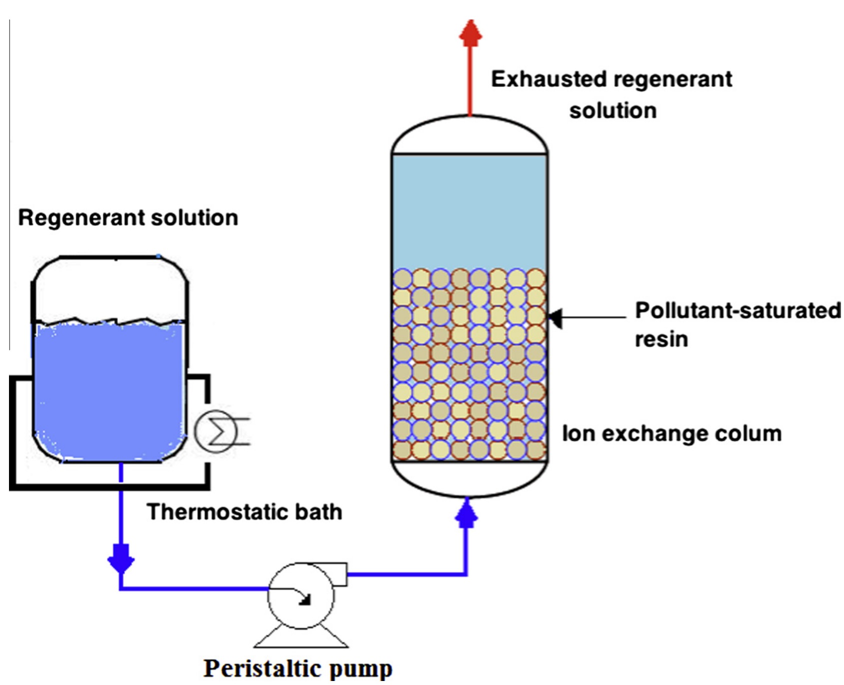

Fig. 1. Scheme of the IE equipment used for regeneration experiments.