



Research article

Removal of textile dyes and metallic ions using polyelectrolytes and macroelectrolytes containing sulfonic acid groups

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ABSTRACT

This work reports the removal of textile dyes and metallic ions by means of adsorption and coagulation-flocculation using two polyelectrolytes and two macroelectrolytes containing sulfonic acid groups. The adsorption of textile dyes was studied in aqueous solutions containing cationic dyes and in wastewater containing a vat dye. Also, removal of vat and naphthol dyes was studied using the process of coagulation-flocculation. The results show these materials possess elevated adsorption capacity, and they accomplished removal rates above 97% in aqueous solutions. The removal of the vat dye improved the quality of the wastewater notably, and an uncolored effluent was obtained at the end of the treatment. The treatment using adsorption decreased the values for coloration, conductivity, suspended solids, and pH. The removal of vat and naphthol dyes by means of coagulation-flocculation was studied as well, and removal rates of 90% were obtained. The polyelectrolytes and macroelectrolytes also proved effective in the adsorption of metallic ions in wastewater. The treatment using adsorption accomplished high removal rates of metallic ions, and it showed greater selectivity towards Cu^{2+} , Fe^{3+} and Pb^{2+} . A decrease in the content of solids as well as the values for COD and conductivity was observed in the wastewater as well. The analyses of FT-IR indicated that cationic dyes and metallic ions were chemisorbed by means of ionic exchange.

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1. Introduction

Nowadays, a growing interest in the development of technologies that allow the purification of the effluents from different industries before they are discarded exists, and even more so, in accomplishing an optimal quality of the treated water to be reused in the productive process. Particularly, the textile, mining and metallurgical industries consume enormous volumes of water, and their effluents severely impact the environment because of the toxic nature of their residues (Pang et al., 2011). Metallic ions contained within these effluents are non-degradable pollutants (Fu and Wang, 2011), and elemental ions such as mercury, cadmium, and lead are considered persistent pollutants (Arceivala and Asolekar, 2007). Dyes are organic molecules that can be

degraded; they could generate byproducts which are even more toxic than the dyes themselves (Peláez-Cid et al., 2008). Activated carbon is the most commonly used adsorbent material in the treatment of textile wastewater because of its high adsorption capacity (Peyratout et al., 2001). However, polymeric materials are starting to be used in place of activated carbon to remove specific dyes because of the wide array of chemical functionalities they possess as well as their ease of renewability (Moradi et al., 2012; Verma et al., 2012).

The use of polymeric materials such as polyelectrolytes constitutes a viable alternative to remove textile dyes and metallic ions from polluted waters (Joo et al., 2009). The ionic groups present in the polyelectrolytes produce an electrostatic field that attracts diverse organic and inorganic substances, and this constitutes the basis for the development of technologies such as ionic exchange, coagulation-flocculation, membrane filtration, and adsorption (Chowdhury et al., 2004; Taleb et al., 2009). Diverse materials have been studied as alternatives to substitute activated carbons in the

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adsorption of textile dyes. Different unconventional adsorbents have been prepared from natural materials, and they have been studied as adsorbents for textile dyes. However, the low adsorption capacities in materials such as rice husk (40.6 mg g^{-1}), oat husk (38.2 mg g^{-1}), banana peel (20.8 mg g^{-1}), kaolin (20.5 mg g^{-1}), orange peel (18.6 mg g^{-1}) and vegetable ash (12.7 mg g^{-1}) (Batmaz et al., 2014) makes their use in wastewater unattractive, even if their cost is extremely low (Blackburn, 2004).

Diverse studies have shown that different polyelectrolytes exhibit superior adsorption capacities compared to the aforementioned unconventional adsorbents. Studies made on polymethacrylic acid show that it possesses an adsorption capacity of 169.64 mg g^{-1} for methylene blue (Bajpai et al., 2012) and 102 mg g^{-1} for Basic Yellow 28 (Panic et al., 2013). Literature also reports the use of poly(epichlorohydrin-dimethylamine) as adsorbent, with a maximum adsorption capacity of 68.69 mg g^{-1} for Reactive Blue K-GL (Li et al., 2007). Kaner reported the synthesis of polyelectrolytes containing sulfonic acid groups with an adsorption capacity ranging between 100 and 160 mg g^{-1} for different dyes (Kaner et al., 2010). It can be observed that polyelectrolytes constitute a good alternative as adsorbents useful in the removal of textile dyes. Nonetheless, it is important to mention that the studies made on adsorption of dyes on commercial ionic exchange resins such as Pall-SB6407 and Whatman-DE81 showed that these possess adsorption capacities lower than 32 mg g^{-1} for Cibacron Blue 3GA and Cibacron Red 3BA (Liu et al., 2007). Besides, other polyelectrolytes based on commercial monomers have shown limited adsorption capacities. On the other hand, the adsorption capacity for crystal violet for a network of poly(acrylic acid-co-acrylamide-co-methacrylic acid) and amylase was 28.60 mg g^{-1} (Li, 2010). Hence, the design and synthesis of new polymers to be used as adsorbent materials constitutes an important line of research in the development of materials useful in the removal of pollutants from water.

Asides from having good adsorption capacities, polyelectrolytes can be easily regenerated without losing their adsorptive properties. For example, Coskun reported the synthesis of hydrogels from monomers containing sulfonic and carboxylic acid groups, and these did not exhibit any loss in their adsorption capacity for methylene blue even after ten cycles of adsorption and desorption (Coşkun and Delibaş, 2012). This way, polyelectrolytes constitute a viable alternative to replace activated carbon as adsorbent materials with the possibility of being regenerated. As well as adsorption, coagulation-flocculation is another technique which is widely used to treat wastewater. Diverse inorganic salts such as $\text{Al}_2(\text{SO}_4)_3$, AlCl_3 , FeCl_3 , $\text{Fe}_2(\text{SO}_4)_3$, and FeSO_4 have been used as coagulant-flocculant agents, but they are being replaced by polyelectrolytes because these improve the results of the treatment by forming massive floccules which allow the removal rates to be increased and the volume of sludge to be decreased (Taleb et al., 2009). Literature reports the use of different polyelectrolytes in the processes of coagulation-flocculation. Some of these are, chitosan (Peyratout et al., 2001; Moradi et al., 2012), poly(sodium styrenesulfate) (Joo et al., 2009), and carboxymethylcellulose (Verma et al., 2012). Finally, polyelectrolytes have also shown potential as adsorbents to remove metallic ions (Feng et al., 2015; Ghimici et al., 2009; Al Hamouz and Ali, 2012). Such is the case for chitosan (Rhazi et al., 2002) and its derivatives (Varma et al., 2004; Wan Ngah and Hanafiah, 2008), cross-linked sulfonated polymers (Huck and Bonn, 2000), and poly(1-vinylimidazole) (Takafuji et al., 2004).

This work reports the use of polyelectrolytes and macroelectrolytes containing sulfonic acid groups as adsorbents to remove textile dyes and metallic ions. The use of the polyelectrolytes as flocculant agents used to treat real effluents from the textile industry was also studied.

2. Materials and methods

2.1. Polyelectrolytes as adsorbents

Polyelectrolytes poly(*p*-acryloyloxybenzaldehyde) modified with *o*-aminophenylsulfonic acid (poly(*p*-ABz) mod *o*-AAFS) and poly(*p*-acryloyloxybenzaldehyde) modified with *p*-aminophenylsulfonic acid (poly(*p*-ABz) mod *p*-AAFS), as well as macroelectrolytes hexakis[4-((2-sulfophenyl)imino)methyl]phenoxy] cyclotriphosphazene (HoASIM) and 2,2,4,6-tetrakis-[4-((2-sulfophenyl)imino)methyl]phenoxy]-4,6-bis-[4-formylphenoxy] cyclotriphosphazene (TpASIM), which were used in this study, were synthesized in accordance to the procedure previously reported by Herrera González et al. (2016). The structures of these materials are shown in Fig. 1.

Polyelectrolytes poly(*p*-ABz) mod *o*-AAFS and poly(*p*-ABz) mod *p*-AAFS contain sulfonic acid groups in the *orto*- and *para*-positions, respectively. Besides, both contain carboxylic acid groups in their structure. Macroelectrolytes HoASIM and TpASIM also contain sulfonic acid groups in their structures in the *orto*- and *para*-positions, respectively. Characterization via FT-IR confirmed the presence of sulfonic acid groups both in the polyelectrolytes and macroelectrolytes. Both polyelectrolytes were synthesized by chemical modification in accordance to the study made by ICP, and the percentage of modification was 24.38% for poly(*p*-ABz) mod *o*-AAFS and 63.35% for poly(*p*-ABz) mod *p*-AAFS. Phenylsulfonic acid groups were introduced during chemical modification and they bonded to the pendant groups on the polymeric chain by means of an imine group (Herrera González et al., 2016). The polyelectrolytes and macroelectrolytes were crushed and sieved to a particle size ranging between 74 and $250 \mu\text{m}$ (meshes 200 and 60). The surface of the polyelectrolytes and macroelectrolytes was analyzed by means of scanning electron microscopy (SEM). The SEM images were obtained using a JEOL microscope model JSM-6300 using an acceleration voltage of 30 kV. The samples were mounted on a graphite tape, and they were then covered in gold.

2.2. Calibration curves for cationic dyes

For this study, the basic dyes designated in accordance to Color Index were: Basic Blue 9 (BB9), Basic Violet 3 (BV3), Basic Red 18 (BR18), Basic Blue 3 (BB3), Basic Yellow 21 (BY21), Basic Yellow 28 (BY28), and a mixture of Basic Blue 159 and Basic Blue 3 (BB159-3), with a ratio of approximately 5:1. Basic dyes are soluble in water, and dissociate forming cations which contain the chromophore groups, and hence, they are known as cationic dyes. Cationic dyes are capable of establishing strong electrostatic bonds with acid polyelectrolytes, such as the ones being studied in this work, because they possess anionic nature. The most important characteristics of the dyes that were studied are shown in Table 1. The values for maximum absorbance wavelength (λ_{max}) were obtained using ultraviolet visible (UV-Vis) absorption spectrophotometry using a HACH spectrophotometer model DR 5000.

To determine the concentration of the dye in the aqueous solutions at equilibrium, calibration curves for the cationic dyes were constructed. Aqueous solutions of cationic dyes with concentrations ranging between 200 and 2000 mg dm^{-3} were prepared. The λ_{max} for the UV-Vis spectrum for each dye was used as the detection wavelength. The calibration curves obtained were adjusted to the experimental data using the method of least squares. The equations corresponding to the calibration curves are shown in Table 1, where C represents the concentration and A corresponds to the absorbance at λ_{max} . A value for the correlation coefficient (R^2) close to one indicates an appropriate adjustment to linearity in the equations obtained.

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