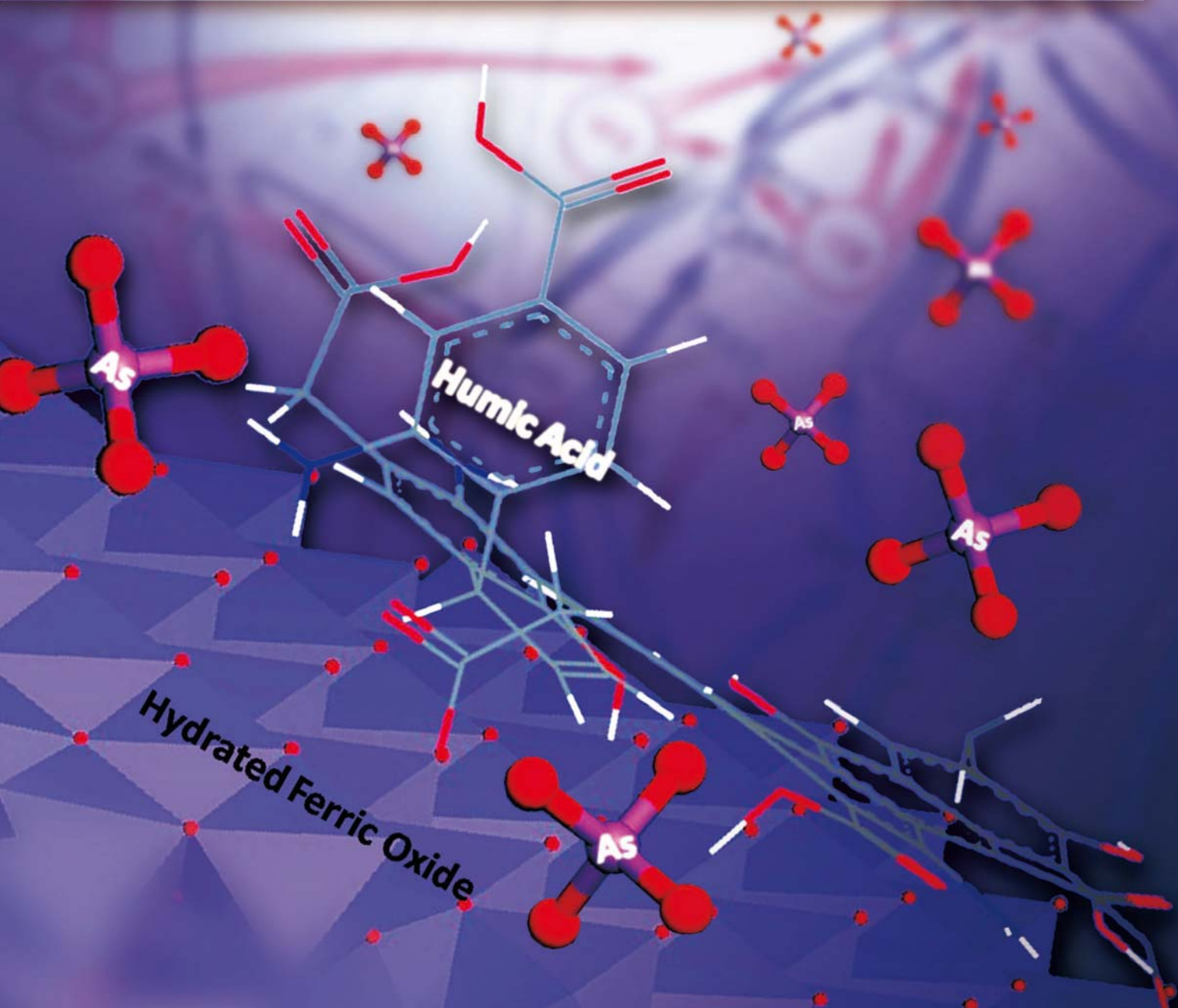


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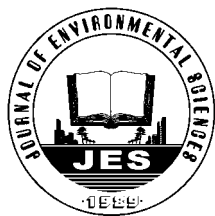
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Catalytic bubble-free hydrogenation reduction of azo dye by porous membranes loaded with palladium nanoparticles

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ABSTRACT

Catalytic bubble-free hydrogenation reduction of azo dye by porous membranes loaded with palladium (Pd) nanoparticles was studied for the first time. The effects of Pd loading, dye concentration and reuse repetitions of membranes were investigated. In reduction, the dye concentration decreased whereas the pH rose gradually. An optimal Pd loading was found. The catalytic membranes were able to be reused more than 3 times.

Introduction

Azo dyes, containing the azo chromophoric group in addition to aromatic systems and auxochromes, are the largest class of commercial dyes used in the industries of textiles, food, pharmaceuticals, cosmetics, paper, etc. (Méndez-Paz et al., 2005). Their environmental impact either as pollutants or carcinogens is of major concern (Pinheiro et al., 2004). Most azo dyes with complex aromatic structures are highly recalcitrant to conventional biological wastewater treatment processes (Greaves et al., 2001). Therefore, pre-treatment processes are required to decolorize the dyes efficiently (Hong et al., 2012). Nevertheless, traditional flocculation (Supaporn et al., 2010) and adsorption (Laszlo et al., 1997) simply transfer the pollutants from aqueous solution to another phase rather than destroying them. Oxidation by ozone, H₂O₂ or chlorine dioxide (Nadupalli

et al., 2011) is expensive. Fenton, photo-Fenton (Elmorsi et al., 2010) or electrochemical advanced Fenton oxidation processes (El-Desoky et al., 2010; Wang et al., 2012) have a harsh working pH range (pH 2–4) and may generate large volumes of iron sludge for further disposal. The quantum yields of photo-catalytic degradation using TiO₂ are low due to hole-electron recombination (Li et al., 2009). By reduction with sulphide (Pereira et al., 2010), zero-valent iron (Fan et al., 2009; Zhang et al., 2010) or Fe-Ni (Bokare et al., 2007) nanoparticles, the azo bonds can be cleaved to give aromatic amines, resulting in decolorization and enhancement of biodegradability (Chang et al., 2006). However, acidic conditions are usually needed in reduction by zero-valent iron (Lin et al., 2008).

Hydrogen is the cleanest reducing agent and has received a great deal of attention. Traditionally, catalytic reduction by hydrogen is carried out in a stirred tank reactor, venturi, fluidized-bed, loop, trickle-bed, compact multichannel or monolithic reactor, etc. (Bradley et al., 1992; Henglein, 2000). In these devices, the limited

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gas-liquid interfacial area changes with hydrodynamic conditions, resulting in difficulty in process control and scale-up. The dispersive contacting mode may also cause problems such as flooding, hazard, emulsion and foams.

In this article, the catalytic bubble-free hydrogenation reduction of azo-dye solution by porous membranes loaded with palladium (Pd) nanoparticles is reported for the first time. Congo Red (CI 28, 22120) was employed as a model dye. Pd nanoparticles were immobilized on the membranes' inner surfaces by the gas-liquid membrane contacting method (Jia et al., 2011). In the reduction, the azo dye aqueous solution and hydrogen, being on opposite sides of the hydrophobic polypropylene hollow fiber membrane, come in contact and react at the mouth of the membrane pores. The membranes provide substantially large and stable gas-liquid interfacial area, which is favorable for mass transfer, process design and scale-up. All the hydrogen can be absorbed in this non-dispersive contact-reaction process, and the problems of emulsion and foaming can be avoided. Therefore, this method provides a novel candidate for catalytic reduction of azo dyes and various other unsaturated organic compounds. The effects of Pd loading, dye concentration and number of reuses of membranes were investigated.

1 Experimental

1.1 Materials

Congo red was provided by Beijing Xudong Chem. Co. Ltd., China. The chemical reagents, including PdCl₂, hydrochloric acid and sodium chloride, were analytical grade. Ultrapure water was employed. Five hundreds mL of 2.0 × 10⁻⁴ mol/L PdCl₄²⁻ aqueous solution containing 0.0170 g of PdCl₂, 8.0 mL of 0.1% (V/V) hydrochloric acid solution and 0.2240 g of NaCl was prepared.

The membrane contactor was made up of twenty symmetric polypropylene hollow fiber membranes with average pore diameter of 0.05 μm, porosity of 60%, O. D. 0.5 mm, I. D. 0.4 mm and effective length of 45.0 mm (Tianjin Blue Cross Membrane Technology Co. Ltd., China). The fibers were well-separated by two porous stainless steel sheets mounted at the two ends of the membrane module.

1.2 Preparation of calibration curve of Congo Red solution

Congo Red aqueous solutions with concentrations of 1.25 × 10⁻⁶, 2.50 × 10⁻⁶, 5.00 × 10⁻⁶, 1.00 × 10⁻⁵ and 2.50 × 10⁻⁵ mol/L were prepared. The UV-Vis absorption of the solutions was analyzed by a UV-Vis spectrophotometer (Cintra 10e, Australia). The calibration curve was obtained by plotting the absorbance at 498 nm vs. the dye concentration.

1.3 Loading of Pd nanoparticles on membrane inner surface

The experimental set-up was schematically shown in our previous paper (Jia et al., 2011). Pure hydrogen with gauge pressure of 1 kPa entered into the shell side of the membrane reactor. The PdCl₄²⁻ solution (2.0 × 10⁻⁴ mol/L, 80.0 mL) in the stirred tank flowed through the membrane lumens at a flow rate of 8 mL/min at 20°C, contacted with the hydrogen permeating through the membrane pores, reacted and then flowed back to the stirred tank for circulation. After reaction, the membranes were rinsed with pure water 5 times, the dried in vacuum to constant weight. The Pd loading, *L* (%), on the membranes' inner surface is expressed as:

$$L = \frac{W_1 - W_0}{W_0} \times 100\% \quad (1)$$

where, *W*₀ (g) is the weight of the pristine membranes, and *W*₁ (g) is the weight of the catalytic membranes. After gold sputtering, the membrane inner surface was observed with cold-field scanning electron microscopy (SEM S-4800).

1.4 Catalytic bubble-free reduction of dye solution

The schematic diagram of the membrane contactor is shown in Fig. 1. The dye solution of 100.0 mL in the stirred tank flowed through the catalytic membranes lumens and then flowed back to the stirred tank for circulation until the absorbance of the solution at 498 nm attained a constant value, indicating that adsorption equilibrium was reached. Then, pure hydrogen (pressure gauge of 1.0 kPa) was introduced into the shell side and then the catalytic membrane pores, contacted and reacted with the dye solution. The pH of the dye solution was monitored. An aliquot (2.0 mL) sample was withdrawn at set intervals during reaction, and the absorbance at 498 nm was measured by a UV-Vis spectrophotometer to determine the dye concentration. The decolorization efficiency, *D*, is expressed as:

$$D = \frac{C_0 - C_t}{C_0} \times 100\% \quad (2)$$

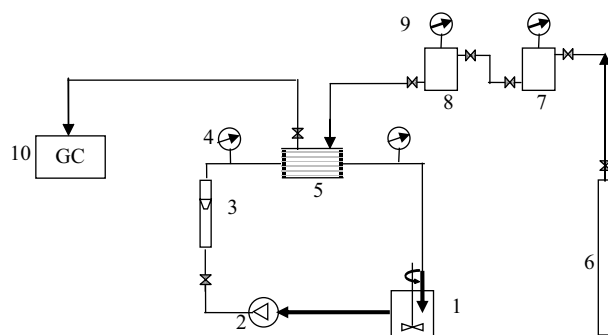


Fig. 1 Schematic diagram of bubble-free hydrogenation. (1) stirred tank; (2) peristaltic pump; (3) liquid rotameter; (4) pressure gauge; (5) membrane contactor; (6) H₂ generator; (7) gas supply tank; (8) buffer tank; (9) barometer; (10) gas chromatometry.

where, C_0 (mol/L) is the initial concentration of the dye, and C_t (mol/L) is the dye concentration at time t .

The degradation products of the dye were identified by electrospray ionization mass spectra (ESI-MS, Waters Premier XE, cone voltage of 20 V, positive ions or negative ions mode) with methanol-H₂O (80/20) as solvent (flow rate of 2.0 mL/min). The leaching of Pd in the dye solution was assessed by inductively couple plasma atomic emission spectroscopy (ICP-AES, Spectro).

2 Results and discussion

2.1 Effects of Pd loading on dye reduction

Pd was immobilized on the membrane inner surface by the membrane contact-reaction method. When the reaction time was 30, 60 and 90 min, the corresponding Pd loading was 10.2%, 21.8% and 26.3%, respectively. The SEM images show the Pd nanoparticles formed on the membrane inner surface (Fig. 2). With the increase in Pd loading, more and more membrane pores were covered with Pd nanoparticles. For Pd loading of 26.3%, almost all the membrane pores were covered.

The effects of Pd loading on reduction were investigated at Congo Red concentration of 2.50×10^{-5} mol/L. Figure 3a shows the change of the UV-Vis absorption spectra of Congo Red solution during reduction for Pd loading of 21.8%. As the reduction proceeded, the solution color became lighter, and the absorption at 498 and 340 nm

originating from the azo conjugated structure disappeared after 7.0 hr. Meanwhile, a new band at 285 nm emerged, which is ascribed to the absorption of aromatic amines (Perkampus, 1992).

Figure 3b, c shows that the dye concentrations declined while the pH rose during reduction. With increasing Pd loading (10.2%, 21.8% and 26.3%), the time needed for complete decolorization was 15, 6 and 21 hr, respectively. From the above results, it can be seen that there exists an optimal loading of Pd: when the loading is too low (e.g. 10.2%), the number and surface area of Pd nanoparticles are limited (Fig. 2a), resulting in low reduction rate; too high Pd loading (26.3%) leads to the blockage of membrane pores (Fig. 2c) and also low reduction rate.

2.2 Effects of dye concentration and reuse times

The effects of dye concentration were investigated using membranes with Pd loading of 17.9%. With rising dye concentrations (0.5×10^{-5} , 1.0×10^{-5} and 1.5×10^{-5} mol/L), the time needed for complete decolorization was 3.0, 4.5 and 6.0 hr (Fig. 4a), and the average reduction rates were found to be 1.7×10^{-6} , 2.2×10^{-6} and 2.5×10^{-6} mol/(L·hr), respectively. That means the average reduction rate increased slightly with dye concentration.

The effect of membrane reuse was studied at Congo Red concentration of 2.5×10^{-5} mol/L. After each reaction, the membranes were cleaned with pure water for 30 min and then reused in the next reaction. For the membranes having Pd loading of 10.2%, with the increase in reuse repetitions, the decolorization efficiency at reaction time of 15.0 hr

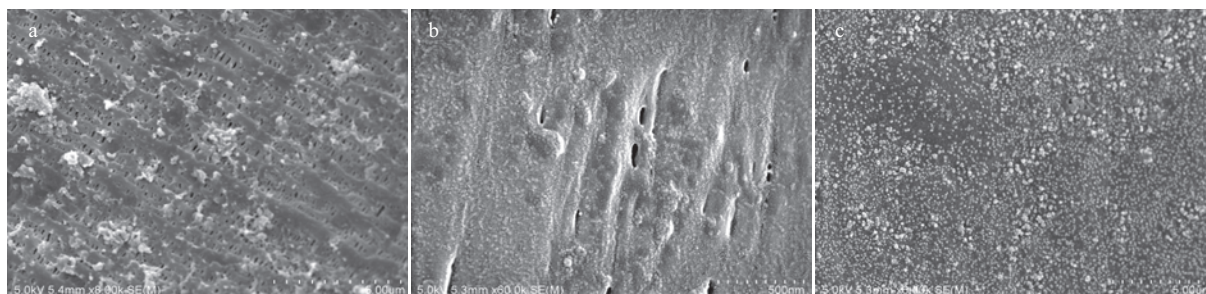


Fig. 2 SEM images of catalytic membrane inner surfaces. (a) Pd amount of 10.2%; (b) Pd amount of 21.8%; (c) Pd amount of 26.3%.

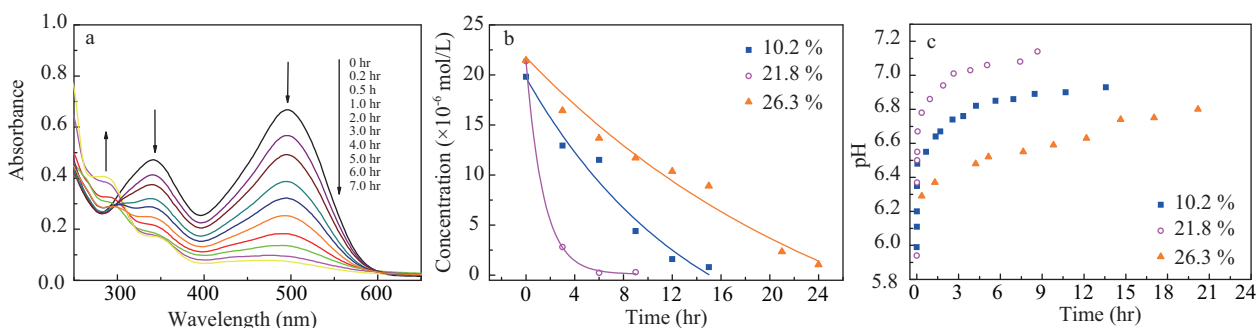


Fig. 3 Effects of Pd amount on reduction of Congo Red. (a) evolution of UV-Vis absorption spectra of Congo Red solution during reduction for membrane of Pd amount of 21.8%; (b) change of dye concentration for membranes with various Pd amount; (c) pH evolution of Congo Red solution in reduction.

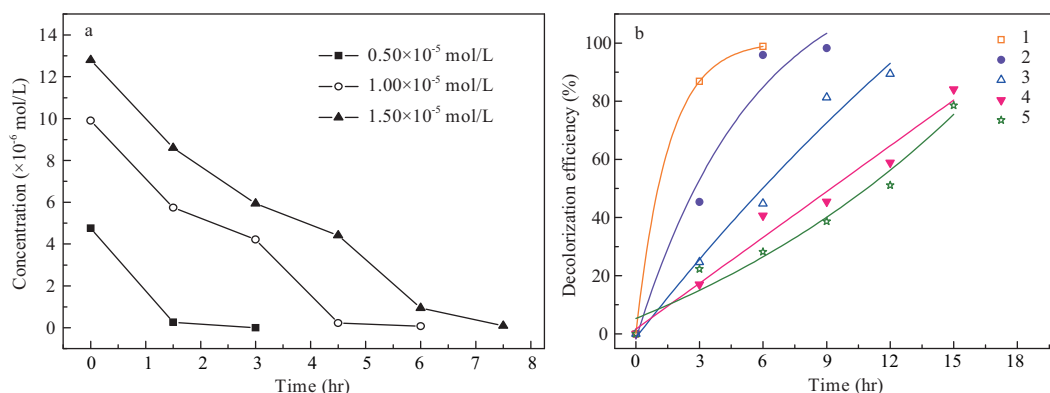
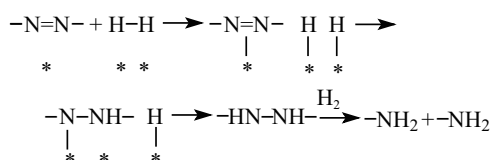


Fig. 4 (a) Effects of dye concentrations on reduction for membranes of Pd amount of 17.9%, (b) Effects of reuse times on the decolorization efficiency for membranes of Pd amount of 21.8%.

decreased from 96% to 87% and 82%. For membranes with Pd loading 21.8%, the decolorization efficiency at time of 6.0 hr decreased from 99% to 82%, 43%, 40% and 25%, respectively (**Fig. 4b**). Therefore, longer reaction time was needed for reused membranes to decolorize completely. To explore the reason for the performance deterioration of reused membranes, the Pd leached in the dyes solution was measured by ICP-AES. It was found the amount of leached Pd in the dye solution was only 0.04 $\mu\text{g}/\text{mL}$, indicating that the Pd nanoparticles were stable in the reaction. It was noticed that the reused membranes became pale red even after rinsing, meaning that the adsorbed dyes or intermediates on the membrane surface were not completely removed. Membrane wetting phenomena may also contribute to the deterioration of reused membranes. Efficient rinsing and wetting prevention can improve the performance of reused membranes.

2.3 Degradation mechanism

The reaction mechanism is described as the following:



where, -N=N- represents the dye molecule, and * represents the active site on Pd surface. The π electrons of Congo Red coordinate with the active site of Pd, and hydrogen dissociatively adsorbs on the Pd catalyst; then, a transfer-addition reaction occurs between a hydrogen atom and the dye, in which a hydrogenated azo group is generated; lastly, the intermediate undergoes further hydrogenolysis to generate 3,4-diamino-naphthalene sulfonate and benzidine. **Figure 5** gives the reduction equation for Congo Red.

Figure 6a shows the ESI-MS spectrum of Congo Red aqueous solution. The peak at m/z 325.4 is the quasi-molecular ion peak $[\text{M}-2\text{Na}]^{2-}$. **Figure 6b** illustrates the ESI-MS spectrum of the reduction product. The

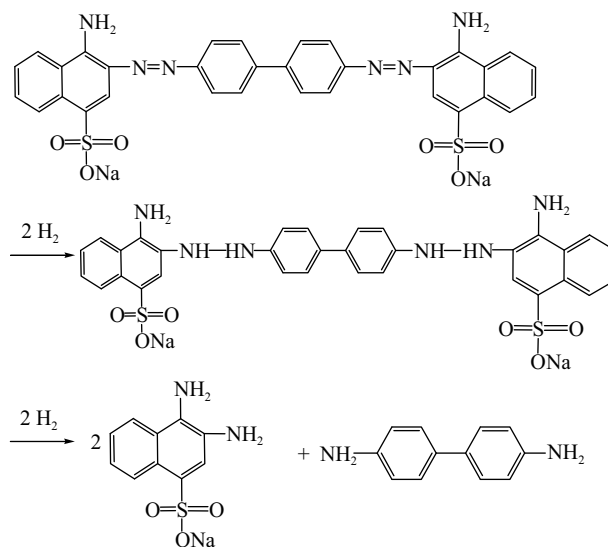


Fig. 5 Reduction equation of Congo Red.

peak at m/z 284.7 is contributed by $[\text{M}'+\text{Na}]^+$ of 3,4-diamino-naphthalene sulfonate, and the peak at m/z 185.4 corresponds to $[\text{M}''+\text{H}]^+$ of benzidine. As a result of formation of aromatic amines (dissociation constant of benzidine: $\text{p}K_{b1} = 9.30$, $\text{p}K_{b2} = 10.37$), the pH of the dye solution increases during reduction. It was well known that most aromatic amines are biodegradable (Pinheiro et al., 2004), though strongly dependent on factors such as the types of microbial population and the availability of oxygen.

3 Conclusions

Congo Red was degraded by catalytic bubble-free hydrogenation reduction with Pd-loaded porous membranes. During the reduction, the dye concentration declined, whereas the pH value rose due to the generation of aromatic amines. There exists an optimal Pd loading which provides sufficient catalytic surface as well as hydrogen

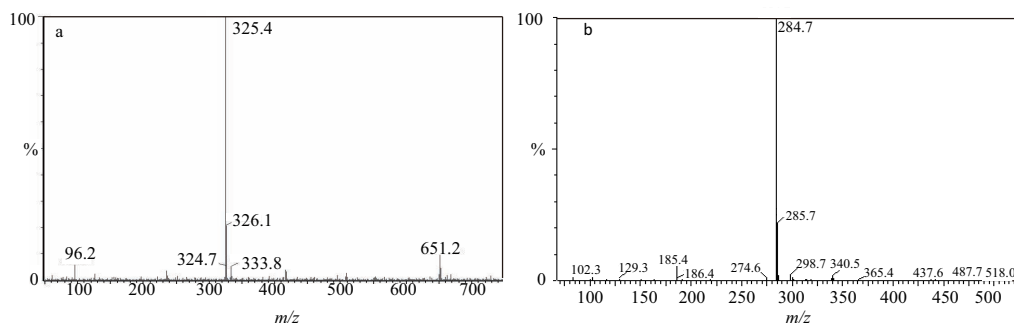


Fig. 6 Electro spray ionization mass spectra of Congo Red (a) and the degradation products (b).

transportation channels. The membrane can be reused more than 3 times. This result provides a safe, versatile and prospective candidate for hydrogenation of azo dyes and some other unsaturated compounds.

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