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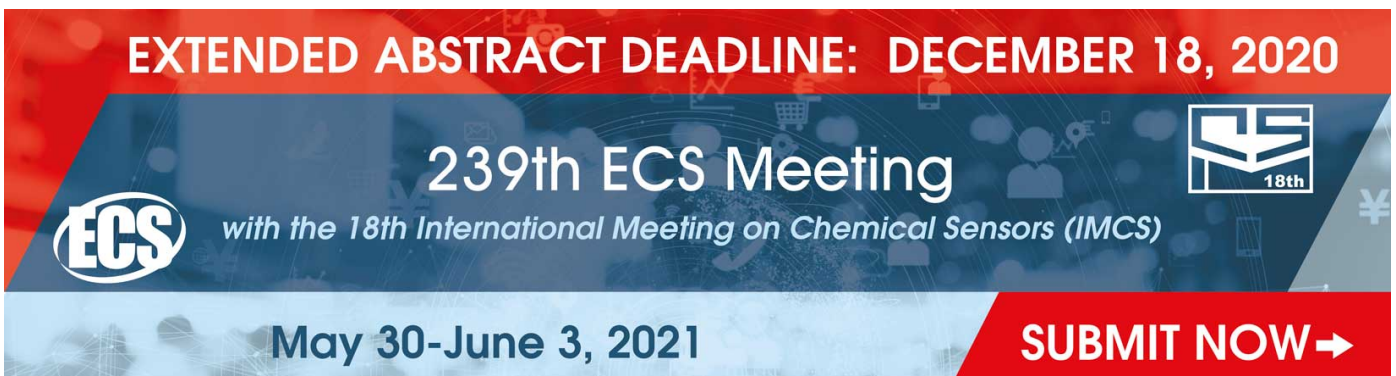
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Core-shell particles based on porous carbon@Fe₃O₄ for efficient removal of dyes from textile effluents

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Abstract. The removal of textile toxic contaminants is an essential process to provide clean water to the increasing population rate. It requires a design of a selective material to adsorb dyes pollutants with stability, effectiveness and reusability. Regeneration efficiency and recovery extent (RCE) are one of the critical factors to measure the performance of the adsorption process. In this study, magnetic Core-shell beads based on porous carbon@Fe₃O₄ NPs with high specific surface area were successfully synthesized by simple Co-precipitation method. The structural, textural, chemical and morphological proprieties of the prepared materials were studied by XRD, FTIR, Raman spectroscopy, nitrogen adsorption-desorption, XPS, SEM and TEM. The adsorption kinetic and isotherm data were well fitted to the pseudo-second-order and Langmuir models. Magnetic particles exhibited an excellent ability to adsorb methylene blue (MB) from aqueous solutions with maximum MB adsorption capacity of 180.42 mg/g (porous carbon@Fe₃O₄-NPs powder) and 49.66 mg/g (magnetic Core-shell beads based on porous carbon@Fe₃O₄-NPs).

The synthesized core shell adsorbent showed good magnetic response, high regeneration efficiency with excellent recovery extent. We believe that in the near future these new magnetic Core-shell beads based on porous carbon@Fe₃O₄-NPs can be used as an alternative to solve the separation problem involving the material pollutant interfaces and can be commercialized as a benchmark adsorbent for the removal of toxic dyes.

1. Introduction

In recent years, nano-Fe₃O₄ has received great attention as a new kind of functional materials for potential applications especially in the field of wastewater treatment. Porous carbon have also been considered excellent candidates for wastewater treatment [1]. However, numerous studies have been conducted to develop porous carbon with large specific surface area and interesting physicochemical characteristics [2]. In the present work, magnetic Core-shell beads based on porous carbon (PC)@Fe₃O₄-NPs with high specific surface area were successfully synthesized by simple Co-precipitation method.

2. Material and methods

The core-shell particles used in this work were prepared in three stages. The first step is to prepare the



porous carbon then the iron nanoparticles (Fe_3O_4) have been impregnated by co-precipitation method and finally core-shell magnetic particles were formed by using Ca^{2+} ions with alginate polymer. X-ray photoelectrons spectroscopy (XPS) was carried out using Thermo Fisher Scientific ESCALAB 250Xi X-ray photoelectron spectroscopy system equipped with Al $K\alpha$ X-ray source = 1486.7 eV for chemical states of synthesized $\text{PC}@Fe_3O_4$. Thereafter, kinetics (adsorbate-adsorbent contact time ranging from 0 to 120 min) and equilibrium isotherms (dye amount ranging from 5 to 90 mg/L of MB) adsorption experiments were carried out using batch method. The remaining MB amount after adsorption was determined by using UV-spectrophotometer JENWAY 6705 at maximum wavelength, $\lambda_{\text{max}} = 664$ nm. $\text{PC}@Fe_3O_4$ and Core-shell beads based on $\text{PC}@Fe_3O_4$ -NPs reusability were performed under optimum conditions using 20 mL of HCl (0.1 M).

3. Results and discussions

Chemical states of the elements in the surface of the $\text{PC}@Fe_3O_4$ were observed using X-ray photoelectron spectroscopy (XPS) (Figure 1). The XPS spectrum of C 1s of PC is shown in Figure. 1b. The peaks at 283.0, 284, 286.2, 287.8, and 288.6 eV were attributed, respectively, to the bonds C-Fe, C-C/C=C, C-O, C=O, and O-C=O³. The deconvolution of the O1s illustrated in Figure. 1c indicates three crests at, respectively, 530 eV (C-OH), 532 eV (C=O), and 533 eV attributed to the sp^2 -bonded carbon atoms⁴. Similar C 1s and O 1s peaks were also observed after the functionalization of PC_350 by Fe_3O_4 -NPs, as can be seen, respectively, in Figures 1d and 1e. From Figures 1a and 1f the binary peaks shown in the range around 700 and 724 eV are typical of, respectively, Fe 2 $p_{3/2}$ and Fe 2 $p_{1/2}$ [5–7]. The high determination XPS spectra revealing the existence of both Fe^{2+} and Fe^{3+} species in the Fe_3O_4 NPs. The Fe 2p (Figure 1) peaks of $\text{PC}@Fe_3O_4$ observed at 710, 712 and 717 eV could be assigned, respectively, to Fe^{2+} , Fe^{3+} in octahedral coordination ($\text{Fe}^{3+}(\text{o})$), and Fe^{3+} in tetrahedral coordination ($\text{Fe}^{3+}(\text{t})$) of Fe_3O_4 [3,8,9]. Thus, the XPS data confirm the formation of Fe_3O_4 -NPs via the co-precipitation synthesis route and the development of Fe_3O_4 nanoparticles at the $\text{PC}@Fe_3O_4$ surface. The prepared magnetic particles were found to exhibit excellent ability to adsorb methylene blue (MB) from aqueous solutions with maximum MB adsorption capacity of 180.42 mg/g (porous carbon@ Fe_3O_4 -NPs powder) and 49.66 mg/g (Core-shell beads based on porous carbon@ Fe_3O_4 -NPs). The adsorption removal of MB decreases as the number of regeneration cycles increases. After four cycles, 93.02% and 80.19% of regeneration efficiency of the regenerated porous carbon@ Fe_3O_4 -NPs and Core-shell beads based on porous carbon@ Fe_3O_4 -NPs were still achieved, respectively. In addition, the recovery extents of PC, $\text{PC}@Fe_3O_4$ and Core-shell beads based on porous carbon@ Fe_3O_4 -NPs were examined and presented in Figure 2b. After the five filtration cycles, RCE were found to be 96%, 84%, and 40% mg for Core-shell beads based on porous carbon@ Fe_3O_4 -NPs, $\text{PC}@Fe_3O_4$ and PC, respectively, indicating that Fe_3O_4 and alginate maintain the relatively of extents recovery of PC. Practically in the case of Core-shell beads based on porous carbon@ Fe_3O_4 -NPs adsorbents, almost Core-shell particles were recovered.

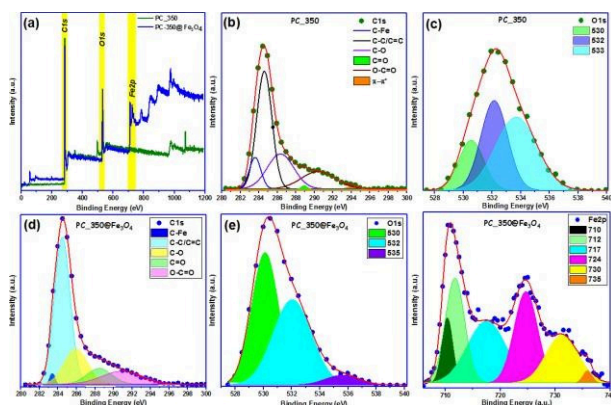


Figure 1. a) XPS patterns of PC and PC@Fe₃O₄ and Normalized XPS spectra b) C 1s of PC c) O 1s of PC d) C 1s of PC@Fe₃O₄ e) O 1s of PC@Fe₃O₄ and f) Fe 2p of PC@Fe₃O₄.

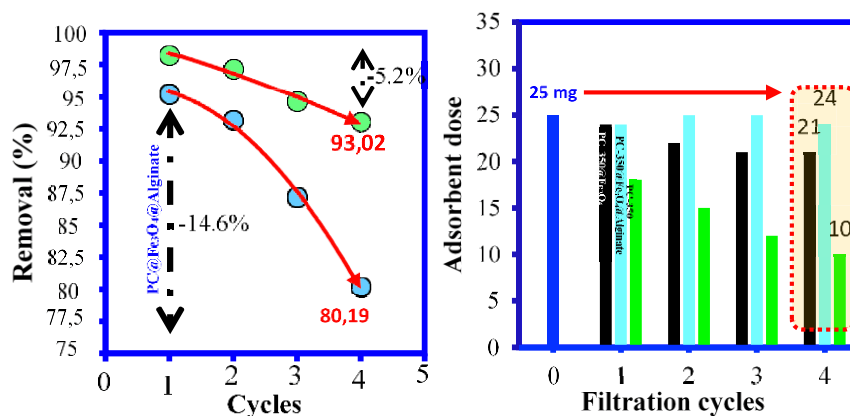


Figure 2. a) Regeneration efficiency and b) Recovery extents (RCE) of the adsorbents.

4. Conclusion

The synthesized core shell adsorbent showed good magnetic response, high regeneration efficiency with excellent recovery extent. It is likely believable that these new magnetic Core-shell beads based on porous carbon@Fe₃O₄-NPs can be used, in the near future, as alternatives to solve the separation problem involving the material pollutant interfaces and can be marketed as a benchmark adsorbent for the toxic pollutants removal.

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