International Journal of Engineering

Preparation and Characterization of Polyaniline/Sb₂O₃ Nanocomposite and its Application for Removal of Pb(II) from Aqueous Media

R. Khalili[a], H. Eisazadeh[b]*

[a] Babol University of Technology, Babol, Iran
[b] Chemical Engineering Department of Shomal University, Amol, Iran.

ABSTRACT

Nanocomposite of polyaniline (PAn) containing nanometer size Sb_2O_3 was prepared in aqueous media via in situ chemical oxidative polymerization method at room temperature in the presence of hydroxypropylcellulose (HPC) as a surfactant. The ability of the product for removal of lead ions from aqueous solution was studied. The chemical structure and morphology were studied by fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM) and X-ray diffraction (XRD). The results indicated that Sb_2O_3 and HPC influence the properties of products. Batch studies were carried out to estimate the influence of pH and contact time on Pb(II) removal percentage. The results showed that the PAn/Sb_2O_3 nanocomposite had a considerable ability for removal of Pb(II) from aqueous solution. Optimum conditions for Pb(II) removal were found to be pH 3 and equilibrium time 30 min. It was also found that the equilibrium adsorption isotherm was better described by Freundlich adsorption isotherm model.

1. INTRODUCTION

The removal of toxic metals from water is an issue of great interest in the field of water pollution control, which is an important cause of water pollution. Numerous metals such as chromium, cadmium, zinc, mercury, lead, etc., are known to be toxic. Lead is one of the most toxic contaminants released into the natural waters from various industrial activities such as metal plating, oil refining and battery manufacturing [1]. The presence of lead in drinking water even at low concentration may cause illness such as anemia, brain disease and hepatitis [2].

Methods used to remove Pb(II) from wastewater include chemical precipitation [3], chemical reduction [4], ion exchange [5], membrane separation [6] and biosorption [7]. However, most of these methods are only suitable for the removal of Pb(II) in low concentrations and often require comprehensive processing and high cost. Adsorption is a promising

method for such conditions, specifically using low-energy requirements [8, 9].

The synthesis of metal–organic polymer and polymer–inorganic nanoparticles have potential applications in industries and have attracted much attention recently due to their interesting properties [10-13]. To prepare the nanoscale materials successfully, several approaches have been employed such as physical mixing [14], the sol–gel technique [15], in-situ chemical polymerization in an aqueous solution with the presence of polymer monomer and inorganic particles [16], emulsion technology [17], sonochemical process [18], and ir-radiation technique [19].

Polyaniline has a reactive N-H group in a polymer chain flanked on either side by a phenylene ring, imparting a very high chemical flexibility. It undergoes protonation and deprotonation in addition to adsorption through nitrogen, which have a lone pair of electrons, and it is responsible for the technologically interesting chemistry and physics [20]. Conductive polymers such as polyacetylene, polyaniline, polypyrrole, and polythiophene, have attracted so much research interest in wide range applications such as rechargeable

*Corresponding Author Email: Eisazadeh@hotmail.com (H. Eisazadeh)

batteries [21], electromagnetic interference (EMI) shielding [22], antistatic coatings [23], gas sensors [24], optical devices [25] and removal of heavy metals [26–28]. Polyaniline has attracted considerable attention because of its unique electrical, optical and electrooptical properties and its numerous potential applications [29].

In this study, PAn/Sb$_2$O$_3$ nanocomposite was synthesized via in situ chemical oxidative polymerization method in aqueous media at room temperature. The product was used for removal of Pb(II) using batch sorption method.

2. EXPERIMENTAL

2. 1. Instrumentation A magnetic mixer model MK20, digital scale model FR 200, scanning electron microscope (SEM) model KYKY-EM3200, Fourier transform infrared (FTIR) spectrometer thermo nicolet model Nexuf 670, X-ray diffraction (XRD) model Equnox 3000, oven Binder model FD 23 and an atomic fluorescence spectrophotometer Perkin-Elmer Corp. model 2380 were employed.

2. 2. Reagents and Standard Solutions Materials used in this work were aniline (extra pure > 99%, d =1.02 g/cm^3), ammonium peroxydisulfate (APS), sulfuric acid, hydroxypropylcellulose (HPC) and antimon oxide (Sb$_2$O$_3$) from Merck. The stock solution of Pb(II) was prepared by dissolving Pb(NO$_3$)$_2$ in distilled water. All reagents were used without further purification and used as received. Aniline monomer was purified by simple distillation. Distilled water was employed throughout this work.

2. 3. Synthesis of Polyaniline/Sb$_2$O$_3$ Nanocomposite In this method, 1.0 g Sb$_2$O$_3$ was added to the aqueous solution of 100 mL sulfuric acid 1.0 M containing 1.0 g APS and 0.4 g HPC along with stirring. After 30 min, 1.0 mL aniline monomer was injected to the stirred solution. The reaction was carried out for 5 h at room temperature. Consequently, the product was centrifuged and dried at temperature about 50 $^\circ$C in oven for 48 h.

As a reference sample, pure PAn was prepared using the same method without Sb$_2$O$_3$ and HPC (100 mL sulfuric acid 1.0 M containing 1.0 g APS and 1.0 mL aniline monomer).

2. 4. Batch Adsorption Experiment Adsorption experiments were carried out by completely mixed batch reactor (CMBR) technique to remove lead ions from water. A 25 mL of Pb(II) solution was added to

the beaker containing the desired adsorbent. Experimental variables considered were initial concentration of Pb(II) 50 mg/L, contact time 15-45 min, pH 3, 5, 7, 10 and dosage of PAn/Sb$_2$O$_3$ 250 mg/25 mL. The amount of adsorption at equilibrium (mg/g) was computed as follows:

$$q_e = \frac{(C_0 - C_e)V}{m} \tag{1}$$

where C_0 and C_e are the initial and equilibrium solution concentrations (mg/L). V is the volume of solution (L) and m is the weight of adsorbent used (g).

3. RESULTS AND DISCUSSION

3. 1. Morphology of Nanocomposite The morphology of product was analyzed by SEM. As shown in Figures 1, 2 and 3 the size and homogeneity of particles are dependent on antimon oxide and HPC. SEM micrograph of PAn without Sb$_2$O$_3$ is shown in Figure 1. As can be seen in Figures 2 and 3, by adding Sb$_2$O$_3$, the particle size decreased and the homogeneity of particles increased. It can be seen that the Sb$_2$O$_3$ nanoparticles with average particles size of 30 nm not only dispersed on the surface of the PAn particles, but also embedded into the PAn matrix.

In addition, by comparison of Figures 2 and 3, it can be deduced that the surfactant influences the morphology of product, because, surfactant prevents gross aggregation of particles. It has been found that surfactant influences the rate of polymer formation, particle size, size distribution, morphology and homogeneity of particles [30].

3. 2. FTIR Spectroscopy The structure of obtained product was determined by FTIR analysis. Figure 4 shows the FTIR spectra of pure PAn, PAn/Sb$_2$O$_3$ and PAn-HPC/Sb$_2$O$_3$. The FTIR spectroscopy has provided valuable information regarding the formation of polyaniline composite. FTIR analysis was done to identify the characteristic peaks of product. For instance, pure PAn shows the presence of characteristic absorption bands at 1563 cm^{-1} (C=C stretching vibration of the quinoid ring), 1468 cm^{-1} (stretching vibration of C=C of the benzenoid ring), 1294 cm^{-1} (C-N stretching vibration), 1119 cm^{-1} (C-H in-plane deformation) and 782 cm^{-1} (C-H out-of-plane deformation) [31].

As can be seen in Figure 4, the bands at 1563, 1468, 1294, 1119 and 782 cm^{-1} were shifted to 1558, 1473, 1298, 1106 and 779 cm^{-1}, in PAn/Sb$_2$O$_3$ nanocomposite respectively, and it proves the interaction of antimon oxide nanoparticles with the different reaction sites of polyaniline. The possible interaction between antimon oxide nanoparticles and the nitrogen side of polyaniline in the composite may be the reason for the band shift at 1563–1558 cm^{-1}.
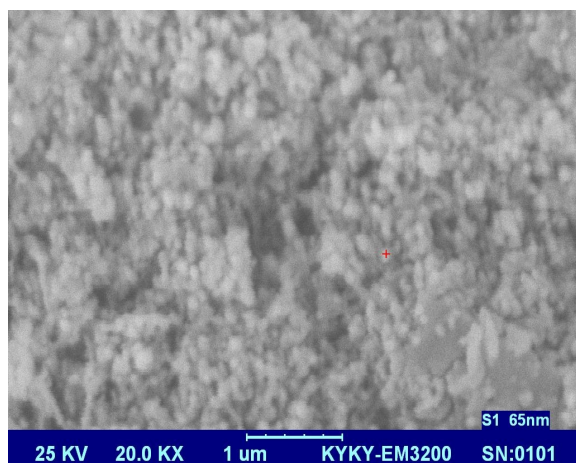
Figure 1. SEM image of pure PAn generated in aqueous media ($(NH_4)_2S_2O_8$) = 10 g/L)
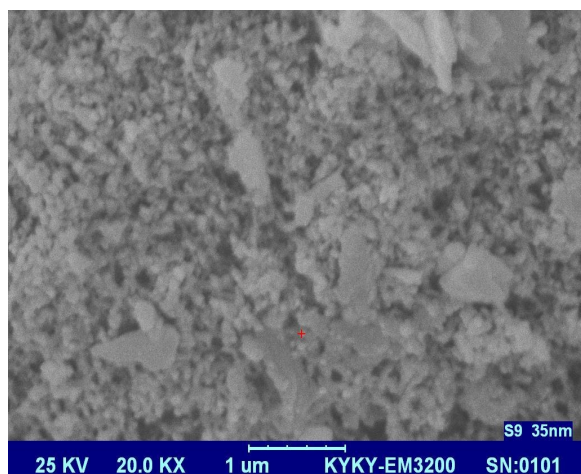
Figure 2. SEM image of PAn/Sb_2O_3 generated in aqueous media (Sb_2O_3 = 10 g/L, $(NH_4)_2S_2O_8$) = 10 g/L).
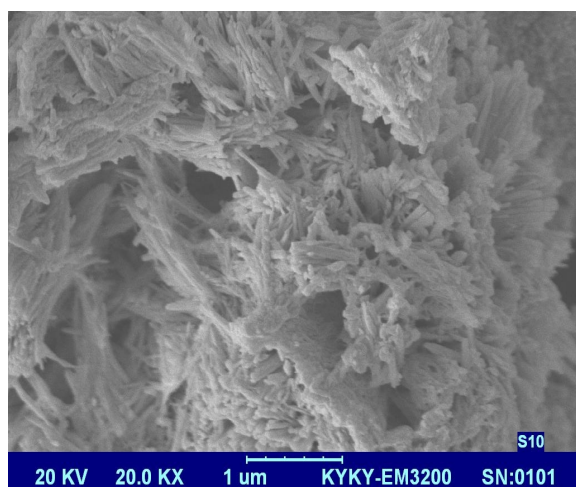
Figure 3. SEM image of PAn-HPC/Sb_2O_3 generated in aqueous media (Sb_2O_3 = 10 g/L, HPC = 4 g/L, $(NH_4)_2S_2O_8$) = 10 g/L).
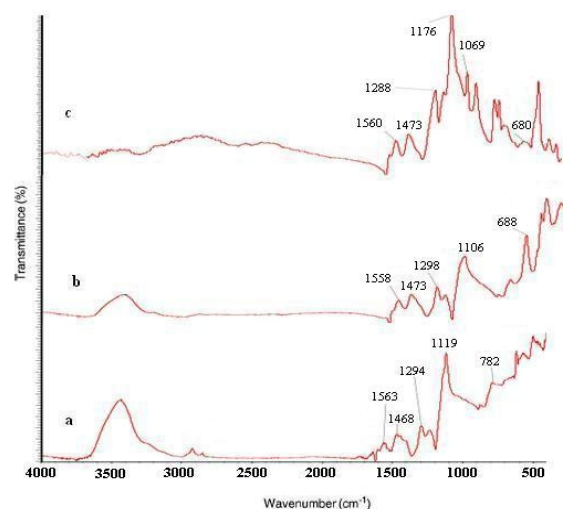

Figure 4. FTIR spectra of (a) PAn, (b) PAn/Sb_2O_3 and (c) PAn-HPC/Sb_2O_3 nanocomposite
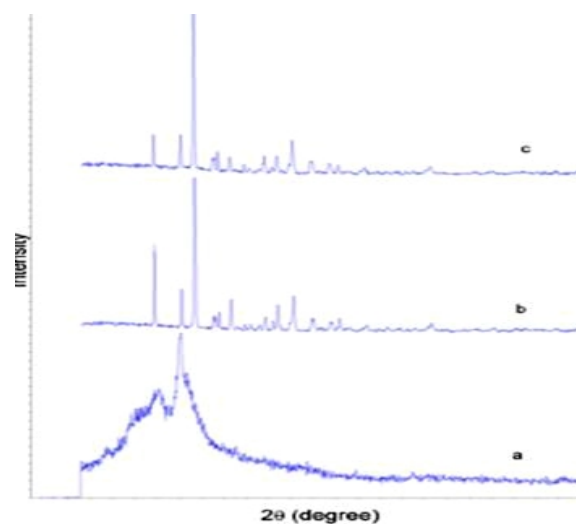

Figure 5. XRD pattern of (a) PAn, (b) Sb_2O_3 and (c) PAn/Sb_2O_3 nanocomposite

3. 3. X-ray Diffraction   The crystalline nature of nanocomposite was determined from XRD analysis. The XRD patterns of pure polyaniline, antimon oxide and PAn/Sb_2O_3 nanocomposite are shown in Figure 5. Polyaniline is innately amorphous and therefore there are no sharp peaks for polyaniline. The pattern 5(b) is XRD pattern of Sb_2O_3 nanoparticles. The diffraction peaks centered at 2θ= 19.34°, 25.42°, 28.33°and 50.53° in pattern 5(c) were same as pattern 5(b), which confirmed the existence of Sb_2O_3 nanoparticles in the polyaniline matrix.

The average crystallite size was estimated from the integral intensity of the XRD using the Scherrer's equation [32]:

$$D=0.89\lambda/\beta\cos\theta \tag{2}$$

where λ is the X-ray wavelength, D is the average diameter of the crystals in angstroms, θ is the Bragg angle in degree and β is the line broadening measured by half-height in radian. The crystallite size of antimon in the nanocomposite, calculated by Equation (2), is about 30 nm. When the peak at $2\theta = 25°$ was selected for calculating the average diameter, the average size of the PAn/Sb_2o_3 was obtained 55 nm.

3. 4. Effect of pH   The pH value of aqueous solution is a significant controlling parameter in the adsorption process. These pH values influence the surface charge of adsorbent, the degree of ionization and speciation of adsorbate during adsorption. In order to evaluate the effect of this parameter on the adsorption, the experiments were carried out at different initial pH = 3, 5, 7 and 10. The experiment was done by PAn/Sb_2O_3 nanocomposite, with an initial lead ions concentration of 50 mg/L, with contact time of 30 min. The results are shown in Figure 6. Removal of lead ions increased with decreasing solution pH and an optimum value was obtained at pH=3. At pH values higher than 7, precipitation of $Pb(OH)_2$ occurs which leads to inaccurate interpretation of adsorption. The possible reason for pH=3 is the synergistic effect of surface complexation and ion exchange. Also the more absorptive sites in PAn/Sb_2O_3 may also play an important role in the improvement of adsorption capacity.

3. 5. Influence of Contact Time   Figure 7 shows the influence of contact time on sorption of lead ions by PAn/Sb_2O_3. For these cases, initial lead concentration was 50 mg/L and pH = 3 was used for Pb(II). Also dose of 0.25 g in 25 mL PAn/Sb_2O_3 was used. When contact time was 30 min then little change of sorption rate was observed. This result demonstrates that adsorption of lead ions was fast and the equilibrium obtained after 30 min of contact time. As a result, a contact time of 30 min was selected for further experiments.

3.6. Adsorption Isotherms   The adsorption isotherm for the removal of lead ions was studied using concentration level of 50-200 mg/L at an adsorbent dosage of 250 mg/25 mL. The adsorption equilibrium data are comfortably represented by adsorption isotherms, which correspond to the relationship between the mass of the solute adsorbed per unit mass of adsorbent (q_e) and the solute concentration for the solution at equilibrium (C_e).

3. 6. 1. Langmuir Adsorption Isotherm   The obtained data were fitted to the Langmuir adsorption isotherm [33] applied to equilibrium adsorption assuming monolayer adsorption onto a surface with a

finite number of same sites and was represented as follows:

$$\frac{C_e}{q_e} = \frac{1}{q_m K_1} + \frac{C_e}{q_m} \tag{3}$$

A linear plot of C_e/q_e versus C_e was employed in Figure 8 to determine the value of q_m (mg/g) and K_l (L/mg). The obtained data and correlation coefficients (R^2) was listed in Table 1.
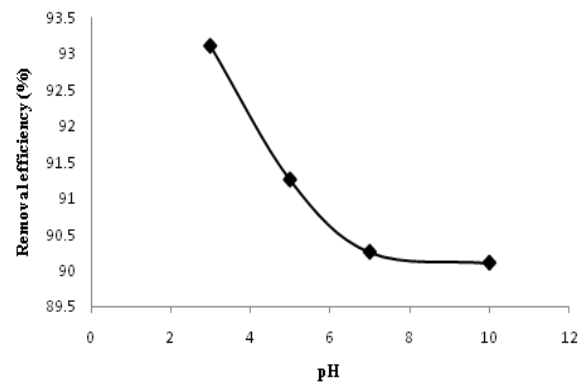

Figure 6. Effect of pH on the removal efficiency using PAn/Sb_2O_3: (the initial concentration, contact time and amount of adsorbent were 50 mg/L, 30 min and 0.25 g, respectively)
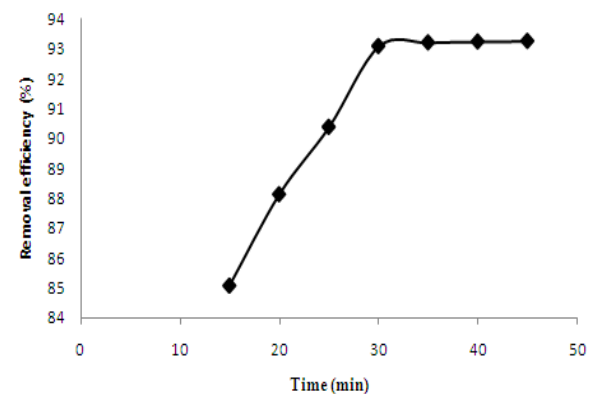

Figure 7. Effect of contact time on the removal efficiency using PAn/Sb_2O_3: (the initial concentration, pH and amount of adsorbent were 50 mg/L, 3 and 0.25 g, respectively)

TABLE 1. Langmuir and Freundlich adsorption isotherm constants for lead ions on PAn/Sb_2O_3

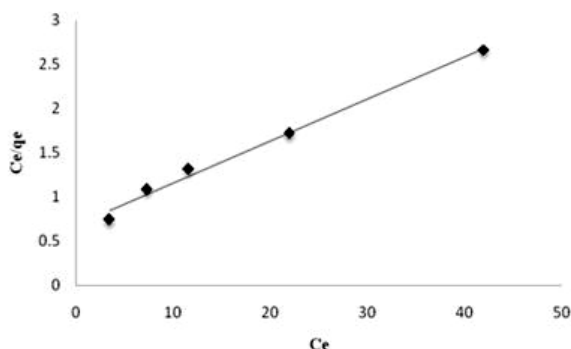| Langmuir constants	q_m (mg/g)	K_l (L/mg)	R^2
	21.05	00.0697	0.9905
Freundlich constants	K_F $((mg/g)/(mg/L)^{1/n})$	n	R^2
	2.527	1.9820	0.9926

Figure 8. Langmuir plot for the adsorption of lead by PAn/Sb$_2$O$_3$: pH, 3; contact time, 30 min; adsorbent dosage, 250 mg/25 Ml
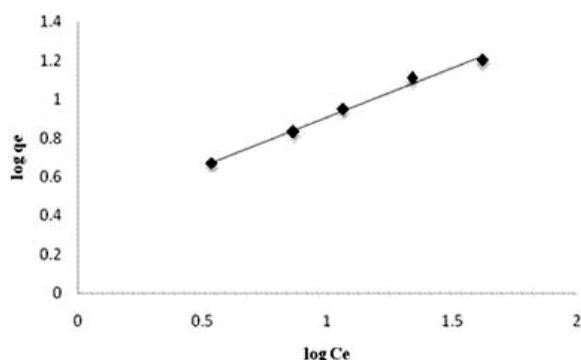

Figure 9. Freundlich plot for the adsorption of lead by PAn/Sb$_2$O$_3$: pH, 3; contact time, 30 min; adsorbent dosage, 250 mg/25 mL.

TABLE 2. The effect of initial concentration of Pb(II) on the removal of lead ions (initial concentration :50 (mg/L))

Type of adsorbent	Final concentration (mg/L)	Removal percentage (%)
PAn	5.79	88.42
PAn-HPC	4.3	91.4
PAn/Sb$_2$O$_3$	3.44	93.12
PAn-HPC/Sb$_2$O$_3$	3.98	92.04

TABLE 3. The effect of initial concentration of Pb(II) on the removal of lead ions (initial concentration :75 (mg/L))

Type of adsorbent	Final concentration (mg/L)	Removal percentage (%)
PAn	8.95	88.06
PAn-HPC	8.52	88.64
PAn/Sb$_2$O$_3$	7.3	90.26
PAn-HPC/Sb$_2$O$_3$	7.46	90.05

3. 6. 2. Freundlich Adsorption Isotherm   The obtained adsorption data were fitted to the Freundlich adsorption isotherm [34] which is expressed by the following equation:

$$q_e = K_F C_e^{\frac{1}{n}} \qquad (4)$$

A linear form of this expression is:

$$log\ q_e = log\ K_F + \frac{1}{n}\ log\ C_e \qquad (5)$$

The Freundlich isotherm constants K_F and n are constants incorporating all factors affecting the adsorption process such as adsorption capacity and intensity of adsorption. The constants K_F and n were computed from Equation (5) using Freundlich plots as shown in Figure 9. The values of Freundlich constants and correlation coefficients (R^2) for the adsorption process are also exhibited in Table 1. The values of n between 1 and 10 (1/n less than 1) represent a positive adsorption. The n values obtained for the adsorption process showed an advantageous adsorption. As can be seen in Table 1, experimental data are better fitted to the Freundlich (R^2=0.9926) than the Langmuir (R^2=0.9905) adsorption isotherm.

3. 7. Effect of Initial Concentration of Pb(II) on the Adsorption   The Pb(II) sorption percentage for 50 and 75 mg/L aqueous solution at pH = 3 and contact time of 30 min was investigated. The amount of adsorbent was adjusted to 250 mg/25 mL.

As can be seen in Tables 2 and 3, by increasing the initial concentration of Pb(II), the removal percentage of lead ions reduced. At high initial concentration, the absorbent surfaces became saturated with the lead ions and the residual metal ions concentration in the solution increased. By comparison of Tables 2 and 3 it can be concluded that antimon oxide effectively influenced the removal percentage. The particle size of composite was decreased by adding Sb$_2$O$_3$. As a result, the total surface of adsorbent increased and also the removal percentage increased. It was also found that the PAn/Sb$_2$O$_3$ nanocomposite has a considerable ability for the removal of Pb(II) from aqueous solution.

4. CONCLUSIONS

In this paper, polyaniline/Sb$_2$O$_3$ nanocomposite was synthesized via in situ chemical oxidative polymerization method at room temperature in aqueous media by using HPC as a surfactant. Its capability for the removal of lead ions from aqueous solution was also studied. The characteristics of PAn/Sb$_2$O$_3$, such as morphology and molecular structure, were investigated. It was found that by adding Sb$_2$O$_3$ nanoparticles an important effect on the particle size and morphology of

the resulting product was obtained. The molecular structures of the products were determined by FTIR spectroscopy. The results indicated that the intensities of the peaks were dependent on the antimon oxide and HPC. FTIR spectra showed that Sb_2O_3 particles were available in the nanocomposite. The results of XRD demonstrated the crystalline structure of Sb_2O_3 and partly the crystalline structure of PAn in the PAn/Sb_2O_3 nanocomposite. Batch adsorption experiment was carried out for the removal of lead ions from aqueous solution. The adsorption characteristics were tested at different pH values and contact time. The results indicated that the PAn/Sb_2O_3 nanocomposite had a considerable ability for the removal of Pb(II) from aqueous solution. Optimum conditions for lead removal were found to be pH 3 and equilibrium time of 30 min. It was also found that the equilibrium adsorption isotherm was better described by Freundlich adsorption isotherm model.

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R. Khalili[a], H. Eisazadeh[b]

a Babol University of Technology, Babol, Iran
b Chemical Engineering Department of Shomal University, Amol, Iran.

چکیده

نانوکامپوزیت پلی آنیلین شامل ذرات اکسیدآنتیموان (Sb₂o₃) در محیط آبی به روش پلیمریزاسیون شیمیایی در دمای اتاق و در حضور هیدروکسی پروپیل سلولز (HPC) به عنوان پایدارکننده تهیه شده است. توانایی محصول تولید شده در حذف یون سرب در محلول آبی مورد بررسی قرار گرفت. خواص محصولات از قبیل ساختار شیمیایی و شکل شناختی با استفاده از طیف سنجی مادون قرمز فوریر(FTIR)، میکروسکوپ الکترونی روبشی(SEM)و پراش اشعه ایکس(XRD) بررسی شده است. نتایج حاکی از آن است که اکسیدآنتیموان و هیدروکسی پروپیل سلولز تاثیر بسزایی برروی خواص محصول تولید شده دارد. برای تخمین اثرات pH و زمان تماس بروی درصد حذف از روش Batch studied و یک راکتور اختلاط کامل ناپیوسته استفاده شده است. نتایج نشان می دهد که نانوکامپوزیت PAn/ Sb₂o₃ توانایی قابل توجه ای برای حذف یون سرب در محلول آبی دارد. شرایط بهینه برای حذف در۳=pH و زمان تماس تعادلی ۳۰ دقیقه بدست آمد. همچنین نتیجه می شود داده های جذب با مدل جذب فرندلیچ بهتر توصیف می شود..