Fabrication of Cellulose Acetate/Iron Oxide Electrospun Nanocomposite as a New Sorbent for Removal of Arsenic from Aqueous Solution

Maryam Akhondi¹, Daryoush Afzali^{1*}, Ali Mostafavi²

 1- Department of Environment, Institute of Science and High Technology and Environmental Sciences, Graduate University of advanced Technology, Kerman, Iran
 2- Chemistry Department, Shahid Bahonar University of Kerman, Kerman, Iran

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Abstract

In this study, iron oxide/cellulose acetate nanocomposite was prepared by electrospinning method and its performance was evaluated for removal of arsenic. Here, oleic acid coated magnetite nanoparticle was synthesized by co-precipitation method and was impregnated in cellulose acetate solution. This magnetite impregnated polymer solution, was electrospun to form nanocomposite polymer fiber. The cellulose acetate/iron oxide nanocomposite membrane was characterized by SEM and FTIR. The magnetic properties of composite nanofibers were characterized by using vibrational sample magnetometry analysis. The nanocomposite was used to remove arsenic ions from aqueous solution. Batch adsorption experiments were carried out to study the sorption behavior of arsenic ions as a function of pH, contact time and initial concentration. Experimental results showed that the maximum capacity of the cellulose acetate-iron oxide nanocomposite membrane for removal of arsenic from low concentration is 0.36 mg/g at pH 9. For better investigation of the adsorption mechanism, two isotherm models, Langmuir and Freundlich were tested. Based on the isothermal results, adsorption data were fitted well to Langmuir isotherm. The reusability of the nanocomposite membrane was confirmed for several adsorption and desorption processes by acid-alkali treatment.

Keywords

Nanocomposite; Sorbent; Nanofibers; Magnetite nanoparticle; Arsenic removal.

1.INTRODUCTION

Electrospinning is currently one of the most promising techniques to produce nanofibers membrane and it is a relatively simple and inexpensive method due to their large surface areas and high porosity with very small pore size in comparison to conventional textiles [1, 2]. In addition to electrospinning method provides flexibility for operational nanocomposite formation using different species into fibers. A number of metal and inorganic nanoparticles have been incorporated into electrospun nanofibers, which help the nanocomposite fibers to obtain, desired functional [3, 4]. Iron oxides are the most widely used classes of adsorbents for arsenic removal due to their high removing capacity, strong binding of adsorbed arsenic, their low cost [5] and the properties of being easily separated, collected and reused by an external magnetic field [6]. Recently, it has been reported that iron oxide (Fe₃O₄) based materials are very effective in the removal of arsenic (arsenate and arsenite) due to their strong adsorption activities. Therefore a lot of

D. Afzali; E-mail: daryoush_afzali@yahoo.com

research works has focused on polymer based composites filled magnetic material in nanometer size [7].

In the recent years, arsenic contamination in water has become a serious threat to environmental and human health. Arsenic is known to easily deposit in certain organs by drinking arsenic laden water and long term exposition to arsenic may cause a wide range of health effects including skin lesions such as hyperkeratosis and pigmentation changes, circulatory disorders, diabetes and cancers of bladder, lung, kidney and skin [8, 9]. Arsenic in natural water primarily occurs in the forms of As (V) and As (III), known as arsenate and arsenite respectively [10, 11]. While arsenite is 25-60 times more toxic than arsenate and more mobile in the environment [12]. Removal efficiencies for As (III) are usually less than those of As (V) furthermore in many cases, pretreatment of As (III) to oxidize it to As (V) may be necessary to efficiently remove arsenic from drinking water where conventional oxidants are used, e.g., ozone, chlorine, and permanganate that are capable of

^{*} Corresponding author:

rapidly oxidizing arsenite to arsenate [13]. Because of arsenic threat to humans, the World Health Organization and the US Environmental Protection Agency implemented a reduction of the arsenic maximum contaminant in drinking water from 50 to 10 µg L⁻¹ in 2006 [14]. Therefore, it is really necessary to remove arsenic from water to make sure that our environment is safe. A number of techniques, namely coagulation, precipitation, membrane filtration, ion exchange, and adsorption have been used to remove toxic elements including arsenic from the aquatic environment [15, 16]. Among the available technologies applicable for water treatment, membrane nanofiltration (NF) and adsorption have been identified as promising technology to remove arsenic from water [17]. In the adsorption technique many of the adsorbent can be considered as cheap or low cost and require little processing [18] but most of them show disadvantages such as low adsorption capacity, a low efficiency/cost ratio, and ineffectiveness at high metal concentration and recycling of powdered nanoparticle materials has been difficult. The NF process can overcome the problems of adsorption technique; however, it has the disadvantages of cost and membrane fouling. Some researchers combine different separation processes for heavy metal removal in order to minimize the limitations of using them alone [19-211.

Biopolymers have attracted much interest in the application of metal removal as they are safe because they do not generate solid waste, and they are biodegradable. Cellulose is an abundant and renewable resource found in most parts of the world, which makes it a cheap raw material for various applications. But, it is not effective in its natural form due to limited adsorption sites and low stability [22]. Among the cellulose derivatives, cellulose acetate polymer is an important cellulose ester polymer in industry with favorable physical properties such as high modulus, adequate flexural and tensile strength. So it is usually used as a component in adhesives. Furthermore, it can also be used as reverse osmosis [23] and nanofiltration membranes [24].

This paper describes the fabrication of cellulose acetate polymer (CAP) membrane with homogeneous dispersion of Fe₃O₄ nanoparticles by electrospinning method. The obtained electrospun iron oxide nanocomposite fibers were further explored as adsorbents for removal of arsenic ions from aqueous solution. In this work, the surface of Fe₃O₄ nanoparticles modified with an oleic acid ligand exhibit an improved uptake for capturing arsenic ions. The mechanism of arsenic ion elimination by the membranes was investigated by application of adsorption isotherms. The reusability of the membranes was examined

adequately by simple acid-alkali treatment. SEM, FTIR, and VSM were applied for characterization of the prepared membranes and nanoparticles.

2.EXPERIMENTAL

2.1.Materials

Cellulose acetate polymer ($M_W = 30,000, 39.8 W_t$ % acetyl content), iron chloride (II) hepta hydrate (97%), iron chloride (III) tetra hydrate (99%), arsenic standard solution (1000.0 mg L⁻¹), oleic acid (99%) and N, N dimethyl formamide (DMF), (purity > 98%) were purchased from Sigma-Aldrich. All other chemicals were purchased from Merck.

2.2.Instrumentation

The nanocomposite membrane was synthesized by electrospinning system (Model Fnm-ES1000, Fanavaran Nano-Meghyas, Iran). The concentration of arsenic was determined by atomic absorption spectrophotometer (equipped with a graphite furnace atomic GTA-110 model SpectrAA220, Varian, Australia). The pH was measured using a pH meter (Model pH691, Metrohm, Switzerland). The magnetic properties of composite were characterized vibrational sample magnetometry analysis (Model 7400 Lake Shore, Lakeshare, USA)

2.3. Preparation of Fe3O4 nanoparticles

Fe₃O₄ magnetite nanoparticles were synthesized co-precipitation method [25]. Briefly, bv FeCl₃.6H₂O and FeCl₂.4H₂O were dissolved in deionized water with 2:1 molar ratio and stirred. The solution was deoxygenated by N₂ gas and the ammonium hydroxide solution was slowly added to the solution to adjust the pH value at 10 under vigorous stirring. The solution immediately became black due to formation of Fe₃O₄ precipitates. Then an amount of oleic acid was added to the solution as a surfactant to prevent from agglomeration. Then precipitate was washed twice with deionized water, as a solvent, in order to prevent the presence of impurities in the final product and, later on, with ethanol, to remove the excess surfactant from the solution. Finally, the particles were separated by a permanent magnet and dried in a vacuum desiccator. Below is the reaction for the formation of Fe₃O₄ nanoparticles: $Fe^{2+} + 2Fe^{3+} + 8NH_3.H_2O \longrightarrow Fe_3O_4 + 8NH_4 +$ $4H_2O$

2.4. Preparation of polymer solution and fabrication of mixed nanocomposite fibers

The solvent was a mixture of acetone and DMF in 2:1 (acetone to DMF) volume proportion. The weighted cellulose acetate and Fe₃O₄ nanoparticles were placed in a glass and acetone was added in order to form a dilute dispersion. Dissolution of the

polymer and dispersion of the Fe_3O_4 particles were rapidly achieved by application of ultrasound. During this procedure evaporation of acetone occurred. After complete dissolution and dispersion, the solutions were cast in Petri dishes at room temperature in a fume hood. Operation of the fume hood ensured an unsaturated atmosphere, which results in a more intense solvent evaporation and this, in turn, results in a faster increase in the viscosity of the solution. As the viscosity of the solution increases sedimentation of the dispersed particles is prevented. After evaporation of the solvent at room temperature for at least 12 h, the films were further dried for another 12 h under vacuum at 333 K. Then they were dissolved in the mixture of acetone and DMF in the desired concentrations. To avoid sedimentation of the particles, the dissolution of the films was carried out in a shaking bath at room temperature. These solutions were used for the electrospinning experiments.

2.5. Electrospinning experiments

The electrospinning of the solutions was carried out by keeping constant flow solution rate (3.0 mL h^{-1}), constant needle to target distance (15 cm) and by applying 25 kV voltage between the target and the needle. The target was a rotating drum covered with cotton cloth.

2.6. Adsorption experiments of iron oxide nanocomposite fiber

This part of experiments was implemented to investigate the electrospun nanocomposite fibers potential for arsenic ions removal. We have studied the effects of adsorption experimental parameters such as initial arsenic concentration (0.02-0.15 mg L⁻¹), time (0-180 min) and pH (5.5-11). Batch adsorption experiments were carried out by soaking 0.02 g of CAP-Fe₃O₄ nanocomposite in 100 mL of arsenic solutions. The concentration of arsenic, was monitored by atomic absorption spectrometry and the rejection (R) percent of arsenic and the amount of adsorbed arsenic (q, mg g⁻¹) were calculated from following equations, respectively:

$$R = \frac{c_{f_-}c_p}{c_f} \times 100 \tag{1}$$
$$q_e = \frac{c_{f_-}c_p}{m} \times v \tag{2}$$

Where V (L) is the volume of aqueous phase and m (g) is the mass of nanocomposite fibers, C_f and C_p are arsenic ion concentration (mg/L) in feed and permeate, respectively.

2.7. Desorption of arsenic from cellulose acetateiron oxide nanocomposite

The best performed membrane in arsenic removal was selected for reusability test. The nanocomposite fibers, were kept for shaking with HNO_3 1 mol L⁻¹ at room temperature. The concentration of arsenic ions was determined by graphite furnace atomic absorption spectrometry. This procedure was sequentially performed for five times with duration of 60 min for each cycle of test. The desorption capacity and desorption efficiency were calculated according to the following equations:

Desorption capacity $= C_a V_a/m$ (3)

Desorption efficiency = $C_a V_a/q_e m$ (4) Where $C_a (mol /L)$ is arsenic concentration in acid solution at desorption equilibrium. V_a (L) is the volume of acid solution used for desorption, m is the mass of the sorbent before adsorption of arsenic ions $q_e (mg /g)$ is the amount of arsenic adsorbed at equilibrium.

2.8. Nanocomposite characterization

Surface morphology of the electrospun polymer cellulose acetate iron oxide nanocomposite membrane fibers was observed using a scanning electron microscopy (SEM). The FTIR analysis was used to analysis the interaction between the Fe₃O₄ and polymer. Magnetic properties of the Fe₃O₄ nanoparticles were measured on vibrating sample magnetometer (VSM).

3.RESULTS and DISCUSSIONS

3.1.SEM analysis

Morphology of the nanoparticles immobilized on the electrospun fibers and average diameter of electrospun nanofibers are observed by SEM. Fig 1 shows SEM micrographs of the electrospun CAP (8 wt%, Fig. 1 a) and magnetite impregnated CAP fibers (1.4 $^{W}/_{V}$, Fig 1 b). It may be observed from the micrographs the resulting CAP and CAPmagnetite nanocomposite fiber membranes, with an addition of magnetite to the polymer solution, the average diameter of the electrospun fibers decreased considerably, and the uniform fibers with the smallest diameters and with fewer drops and beads were obtained. Since morphology of the electrospun fibers depend on the solution's properties (eg. viscosity, surface tension, conductivity, and concentration [26]). As well as the SEM micrographs show morphology of the nanoparticles are spherical with an average diameter of about 50 nm.

3.2.FTIR analysis

The results of FTIR analysis was used to analysis the interaction between Fe_3O_4 and cellulose acetate polymer. Fig 2 a, and b shows the FTIR spectra of pure CAP and CAP/Fe₃O₄ nanocomposite fibrous membrane, respectively. The main characteristics bands of CAP were assigned as follows: The characteristic absorption peak at 3500 cm⁻¹ can be attributed the presence of hydroxyl group and the peaks at 1025 cm⁻¹, 1750 cm⁻¹ and 3000 cm⁻¹ corresponding to the stretching of C–O bond and C=O group and C–H bond, respectively [27]. In Fig 2b, in addition to the characteristics peak of CAP, a new peak around 477 cm⁻¹ is assigned to the Fe₃O₄ vibration which further confirms the formation of Fe₃O₄. By comparing the IR spectra of Fe₃O₄ /CAP composite fibrous membrane with that of pure CAP fibrous membrane, the characteristics peak of –OH group was shifted to 3471 cm⁻¹ from 3450 cm⁻¹ as shown in figure. Similar shift are observed for the other peaks. The observed shifts in the peaks may be described due to the formation of hydrogen bonding between Fe₃O₄ and CAP.



Fig. 1. SEM images of the (a) CAP (b) CAP/Fe $_3O_4$ nanocomposite electrospun membrane



Fig. 2. FTIR spectra of (a) CAP (b) CAP/Fe $_3O_4$ nanocomposite electrospun membrane

3.3Magnetic properties

Investigation of the magnetic properties of the CAP-Fe₃O₄ nanocomposite membranes was carried out by VSM at 300K. Fig 3 shows the magnetic properties of Fe₃O₄ nanoparticles incorporated in nanofibers. VSM examination in Fig 3 showed that Fe₃O₄/CAP nanocomposite exhibited superaparamagnetic behaviors at magnetic field by the 65 (emu /g) saturation magnetization (Ms).

3.4.Adsorption studies

3.4.1.The effect of sample solution pH on arsenic removal

The effect of pH on the adsorption of arsenic ions from solution was investigated. The pH of the solution was adjusted by adding NaOH or HNO₃. The arsenate ions exist as different ionic species (i.e., $H_2AsO_4^-$, $HAsO_4^{2-}$, and AsO_4^{3-} increase at high pH. At pH < 9 surface charge of the membrane nanocomposite was positive and accordingly more attractive to As (V), which appears to be in forms of $H_2AsO_4^-$ and $HAsO_4^{2-}$. But when pH value exceeded pH of 9 the surface sites became negatively charged causing repulsive electrostatic effect towards arsenic species as verified by lower adsorption efficiency [28]. Thus, the removal efficiency percent of arsenic was evaluated within pH range of 5.5-11. As shown in the Fig. 4 with increase in the pH solution from 5.5- 9.5, the percent removal of arsenic increased. The maximum arsenic uptake was observed in the pH range of 9 with 98%. Therefore, the pH was adjusted to 9 in future experiments.



Fig. 3. Magnetization curves of the nanocomposite membrane containing 1.4 wt\% Fe_3O_4 measured at 300K



Fig. 4. Effect of pH on the removal efficiency of membrane nanocomposite

3.4.2. Effect of contact time on arsenic removal Contact time of arsenic on membrane nanocomposite was also examined [29]. It is clearly evident from Fig 5 that, adsorption was rapid for the first times and attained a maximum at the 1 hour, at which an equilibrium was reached. At this point, the percentage removal of arsenic almost was 98 % for all concentrations used and there after remains almost constant. As expected, the adsorption rate was initially rapid because of the vacant number of adsorption sites available on nanocomposite membrane surface.



Fig. 5. Effect of time on the removal efficiency of membrane nanocomposite

3.4.3.Effect of initial concentration on arsenic adsorption

The initial arsenic ion concentration is another important factor to influence the adsorption behaviors of the membrane nanocomposite. The experiments were carried out using various concentrations of arsenic solution under the determined optimum pH values and contact time. The effect of initial arsenic ion concentration was investigated in the range of 0.02-0.15 mg L⁻¹. The results are presented in Fig. 6. It can be seen that uptake percentages of arsenic from solution is decreased and the amount of adsorbed ions is increased with increasing of initial concentration of arsenic because the amount of loaded Fe₃O₄ nanoparticles is low.



Fig. 6. Effect of initial arsenic concentration on the removal efficiency of membrane nanocomposite 3.5. Desorption and reusability of nanocomposite on arsenic removal

The best performed nanocomposite in arsenic removal was selected for reusability test. Changing of pH is a very efficient parameter for regeneration of adsorptive nanocomposite membranes. Therefore, this chemical method was applied for reusing CAP-Fe₃O₄ nanocomposite membrane. Membrane nanofibers were kept for shaking with HNO_3 1 M for 6 h at room temperature. The nanofibers were removed and then washed using distilled water to reach neutral pH for another use. The recovered sorbent (after drying) was reused in the next cycles. The results in Table 1 confirms that membrane nanocomposite can be reused even after 5 cycles of filtration process with only 5% decrease in the removal of arsenic. It should be mentioned that the nanocomposite membrane performance had no noticeable change during five cycles of test and was approximately equal to what presented in Table 1. This confirms significant reusability and durability of prepared nanocomposite membrane for arsenic removal from water.

 Table 1. Recycling efficiency of electrospun CAP-Fe₃O₄ nanocomposite

Cycle	Removal efficiency
1	97.1±1.7
2	95.5±2.4
3	95.4±3.8
4	95.2±4.0
5	95.1±3.9

3.6. Adsorption isotherm study

The adsorption data were analyzed with the help of linear form of Langmuir isotherm and Freundlich isotherm models, expressed by the Eqs. (5) and (7), respectively. Langmuir isotherm [30] applies to adsorption on completely homogeneous surface with negligible interaction between adsorbed molecules. It is represented as:

$$q_e = \frac{\kappa_a q_m c_e}{1 + q_e c_e}$$

The linear form of the above equation is given by: $\frac{C_e}{C_e} = \frac{1}{c_e} C_e + \frac{1}{\kappa_{e-a_m}}$ (6)

(5)

Where q_e is the equilibrium adsorption capacity of ions on the adsorbent (mg g⁻¹), C_e is the equilibrium ions concentration in solution (mg L⁻¹), q_m is the maximum capacity of the adsorbent (mg g⁻¹) and K_a is the Langmuir adsorption constant (L mg⁻¹).

The equilibrium data were analyzed using the linearized form the Langmuir adsorption isotherm Eq. (4). The Langmuir constants, K_a and monolayer sorption capacity, q_m were calculated from the slope and intercept of the plot between C_e/q_e and C_e (Fig7). The results and equations are indicated in Fig 7 and Table 2.



Fig. 7. Langmuir Plot for the adsorption of arsenic on membrane nanocomposite

 Table 2.Parameters for Langmuir and Freundlich models of arsenic adsorption on cellulose acetate-Fe₃O₄ nanocomposite

Langmuir parameter		Freundlinch parameter			
\mathbb{R}^2	q_m	Ka	\mathbb{R}^2	$\mathbf{K}_{\mathbf{f}}$	1/n
0.998	0.36	0.0234	0.938	0.67	0.33

Freundlich isotherm can be applied for heterogeneous surfaces and multilayer sorption. It is expressed as:

 $q_e = K_F C_e^{\frac{1}{n}}$ (7) Linear form of the equation is: $\log (q_e) = \log(K_F) + \frac{1}{n} \log(C_e)$ (8)

Where equilibrium capacity q_e and C_e are defined as above, K_F the Freundlich constant (L mg⁻¹) and n is heterogeneity factor. The Freundlich constant, K_F and adsorption intensity, $\frac{1}{n}$ were calculated from the slope and intercept of the plot between log (q_e) and log (C_e). The results and equations are indicated in Fig. 8 and Table 2.



Fig. 8. Freundlich plot for the adsorption of arsenic on nanocomposite membrane

As seen in Fig 7, the Langmuir isotherm fits quite well with the experimental data (correlation coefficient $R^2 > 0.99$), whereas, the low correlation coefficients ($R^2 < 0.94$) show poor agreement of Freundlich isotherms with the experimental data. The monolayer adsorption capacity according to this model was 0.36 mg g⁻¹. The Langmuir isotherm fits the experimental data very well may be due to homogeneous distribution of active sites (Fe₃O₄ nanoparticles) onto nanocomposite membrane surface, since in Langmuir equation assumes that the surface is homogeneous.

CONCLUSION

In this work a new iron oxide nanocomposite fibers was successfully prepared by electrospinning of magnetite impregnated iron oxide and CA polymer solution. The nanoparticles were uniformly dispersed in the polymer matrix as shown in the SEM microstructure analysis. VSM examination demonstrated Fe_3O_4/CAP nanocomposite membranes showing superparamagnetic behaviors. The results of this work suggest that the magnetic Fe₃O₄/CAP nanocomposite can be one of

promising materials for removal of arsenic from water and wastewater. From this study, it is apparent that the removal of arsenic by Fe₃O₄/CAP nanocomposite depends on pH, contact time and initial concentration of arsenic. The results show that Fe₃O₄/CAP membrane nanocomposite can adsorbed arsenic ions better in an alkali pH range. The regeneration results confirmed that the prepared nanocomposite membrane can offer excellent reusability and the best isotherm fit for arsenic removal was obtained with Langmuir adsorption isotherm.

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ساخت نانوکامپوزیت استات سلولز /اکسید آهن و استفاده از آن به عنوان جاذبی جدید برای حذف یون آرسنیک در محلول های آبی مریم آخوندی ٬ داریوش افضلی ٬ علی مصطفوی ۲

۱– گروه محیط زیست، پژوهشکده علوم و فناوری عالی و علوم محیطی، دانشگاه تحصیلات تکمیلی فناوری پیشرفته، کرمان، ایران ۲– گروه شیمی، دانشگاه شهید باهنر کرمان، کرمان، ایران * E-mail: daryoush_afzali@yahoo.com

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چکیدہ

در این مطالعه نانوکامپوزیت اکسید آهن/ استات سلولز به روش الکتروریسی تهیه و عملکرد آن در حذف آرسنیک مورد ارزیابی قرار گرفت. در اینجا نانوذره اکسید آهن معناطیسی پوشش داده شده با اسید اولئیک به روش هم رسوبی سنتز و در محلول استات سلولز قرار داده شد. این محلول پلیمری برای تشکیل الیاف نانوکامپوزیت الکتروریسی شد. غشای نانوکامپوزیت استات سلولز/اکسید آهن توسط SEM و FTIR مورد بررسی قرار گرفت. خواص مغناطیسی نانوالیاف کامپوزیت با استفاده از آنالیز مغناطیس سنجی نمونه ارتعاشی مشخص شد. این نانوکامپوزیت برای حذف یون های آرسنیک از محلول آبی مورد استفاده قرار گرفت. خواص مغناطیسی نانوالیاف کامپوزیت با استفاده از آنالیز مغناطیس سنجی نمونه ارتعاشی مشخص شد. این نانوکامپوزیت برای حذف یون های آرسنیک از محلول آبی مورد استفاده قرار گرفت. قرامی های جذب برای مطالعه رفتار جذب یونهای آرسنیک به عنوان تابعی از HH، زمان تماس و غلظت اولیه انجام شد. نتایج تجربی نشان داد که حداکثر ظرفیت غشای نانوکامپوزیت استات سلولز–اکسید آهن برای حذف آرسنیک از ۲۰۶۰ میلی گرم بر گرم در 9 و فروندلیچ مورد بررسی قرار گرفتند. بر اساس نتایچ، داده های جذب به خوبی با ایزوترم لانگمویر مطابقت دارد. قابلیت استفاده مجدد از غشای نانوکامپوزیت برای چندین فرآیند جذب و دفع در محیط های اسید و باز مورد بررسی قرار گرفت .

کليد واژه ها

نانوكامپوزيت؛ جاذب؛ نانوالياف؛ نانوذره مغناطيسي؛ حذف أرسنيك.