



Modification and characterization of cellulose cotton fibers for fast extraction of some precious metal ions



M. Monier*, M.A. Akl, Wael M. Ali

Chemistry Department, Faculty of Science, Mansoura University, Mansoura, Egypt

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ABSTRACT

In this work, native cellulose cotton fibers were first modified through graft copolymerization of polyacrylonitrile (PAN) and then by insertion of phenyl thiosemicarbazide moieties to finally produce C-PTS chelating fibers, which were fully characterized using various instrumental techniques such as SEM, FTIR, EDX and XRD spectra. The obtained C-PTS were employed in removal and extraction of Au^{3+} , Pd^{2+} and Ag^+ precious metal ions from their aqueous solutions using batch experiments. The kinetic studies showed that the pseudo-second-order model exhibited the best fit for the experimental data. In addition, the adsorption isotherm studies indicated that the adsorption follows the Langmuir model and the maximum adsorption capacities for Au^{3+} , Pd^{2+} and Ag^+ were 198.31, 87.43 and 71.14 mg/g respectively.

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1. Introduction

As a result of their unique chemical and physical properties, precious metals like gold, platinum, silver and palladium are extensively utilized in many applications such as catalysis, electronics, anti-corrosion materials and jewelry. In addition, with in the next few years, precious metals are expected to be employed in some new environmental, biotechnological and therapeutic uses [1–3]. For these reasons, many eco-friendly methods were developed for efficient extraction and recovery of these valuable metals from their limited sources and from industrial effluents. Among the efficient process for the separation and isolation of these precious metal ions from industrial wastewater, sorption onto active adsorbents such as activated carbon [4,5], modified silicates [6,7], chelating polymeric resin [8,9], biopolymers and biomass [10–12] considered as a common and successful method.

Between all of the previously mentioned sorbents, chelating materials derived from inexpensive natural or synthetic sources considered of a great importance particularly from the economic point of view [13–18]. Usually, the chelating materials are utilized in form of beads, resin or membranes [19,23]. However, recently many studies had been focused on preparation and

application of fibrous chelating materials which derived from either natural fibers such as wool fibers [24] or synthetic fibers such as PET and polypropylene fibers [21,22,25]. Actually the chelating fibers provides many advantages when compared to the conventional chelating materials in form of beads or membranes, first of all, the ease of preparation, extraction and modification, also, the ability to be applied as felts or fabrics which provides a high efficiency and high surface area during the contact with the media and subsequently enhance the rate of both reaction and regeneration process.

In the past few years various studies focused on evaluation of modified cellulosic materials as an efficient and inexpensive biosorbents. In addition to the great advantages of cellulose as a highly abundant, cheap and biodegradable material, cellulose can also be easily modified due to the high availability of the active hydroxyl groups which play an important role in various types of reactions like oxidation, ether formation, esterification and free radical graft co-polymerization by which many cellulose derivatives are prepared [26–28].

In this article, brand new chelating fibers based on cellulosic cotton fabrics modified by insertion of phenyl thiosemicarbazide moieties (C-PTS) had been prepared for quick elimination of Au(III) , Pd(II) and Ag(I) precious metal ions from aqueous solution. The prepared C-PTS chelating fibers were fully characterized using various instrumental techniques such as elemental analysis, scanning electron microscope (SEM), FTIR and wide angle X-ray spectra. Also, various parameters such as pH, temperature, kinetics and

* Corresponding author at: Chemistry Department, Faculty of Science, Mansoura University, Mansoura, 35516, Egypt. Tel.: +20 1003975988.
E-mail address: monierchem@yahoo.com (M. Monier).

adsorption isotherms were investigated in order to evaluate the optimum adsorption conditions and maximum adsorption capacity.

2. Materials and methods

2.1. Materials

Cotton fibers were collected from the high agriculture school farm in Mansoura, Egypt and treated by desizing in 1% (v/v) H₂SO₄ and scouring in 1% (w/v) NaOH. The fibers were then washed with distilled water and absolute ethanol and finally the cleaned fibers were dried in oven at 50 °C till constant weight. Acrylonitrile (AN) (Sigma–Aldrich) was purified by treatment with 3% (w/w) NaOH solution and then washed with distilled water until free from alkali. Potassium persulphate (KPS) (Sigma–Aldrich), Thiourea (TU) (BDH–England); hydrazine hydrate (Adwic); phenylisothiocyanate (Sigma–Aldrich), AuCl₃, PdCl₂ and AgNO₃ were purchased from Sigma Aldrich. All chemicals were used as received.

2.2. Synthesis of C-PTS chelating fibers

The actual synthetic reaction had been accomplished with in the next few steps. Initially the native cotton fibers were modified by graft copolymerization with polyacrylonitrile (PAN) as in the following. 0.1 g of the cotton fibers was immersed into 100 mL conical flask containing the combined redox initiator system composed of 25 mL 2 mmol KPS and 25 mL 2 mmol TU and to which, 0.5 mL 1% H₂SO₄ was added. After which, the mixture was vigorously shaken for 5 min then, the AN monomer (2 mL) was added and the shaking was continued up to 3 h at 80 °C. In order to terminate the graft copolymerization reaction, 5 mL of 3% hydroquinone solution was added. The PAN grafted cotton (C-g-PAN) fibers were then removed from the reaction medium, washed with DMF to extract the PAN homopolymer and finally dried at 50 °C till constant weight.

Grafting percentage (GP) was evaluated according to the following mathematical expression:

$$\text{Grafting percentage (GP)} = \left(\frac{A - B}{B} \right) \times 100 \quad (1)$$

where *A* and *B* are the weight of grafted product and native cotton fibers, respectively.

The resulted C-g-PAN from the previous step, were then treated with 100 mL 10% (v/v) alcoholic hydrazine hydrate solution and the mixture was refluxed for 4 h at 80 °C. The obtained modified C-g-PAH fibers were then eliminated from the mixture, washed with ethanol and dried at 50 °C.

The modified C-PTS chelating fibers were finally manufactured by refluxing the previously prepared C-g-PAN with 100 mL 10% (v/v) alcoholic phenylisothiocyanate solution at 80 °C for 4 h. The fibers were then removed, washed with ethanol and dried at 50 °C.

Schematic presentation for the synthetic steps of the C-PTS is shown in [Scheme 1](#).

2.3. Characterization of the polymer samples

The elemental analysis (E.A.) of the native cotton fibers, C-g-PAN, C-g-PAH and C-PTS was obtained from a Perkin-Elmer 240 C Elemental Analytical Instrument (USA).

FTIR spectra were performed using a Perkin-Elmer spectrum. The fiber samples were dried overnight at 60 °C under reduced pressure and pressurized with a glass slide on top of the quartz window of the ATR instrument.

The surface morphologies of the native and modified fibers were observed using a FEI Quanta-200 scanning electron microscope (FEI

Company, The Netherlands) equipped with Oxford energy dispersive X-ray system (EDX) operating at 20 kV.

ASAP 2010 Micrometrics instrument was utilized to anticipate the specific surface area of the studied samples by N₂ adsorption isotherm and by Brunauer–Emmett–Teller (BET) method.

Crystallinity of the polymeric samples was determined using X-ray powder diffractometer (Japanese Dmax-rA, wavelength = 1.54 Å, CuKα radiation). Generator intensity was 40 kV, generator current was 50 mA. The sample was then scanned from 2θ = 5–70°, in step of 0.02°. The resultant graphs were printed out on the Origin graph plotting package.

2.4. Metal ion uptake experiments using batch method

2.4.1. Instrumentation

A Perkin-Elmer Model 5000 atomic absorption spectrometer (Perkin-Elmer, Shelton, CT-USA) was utilized for detection of the precious metal ion concentrations. The instrument was set at Ag 328.068 nm, Au 242.795 nm and Pd 324.270 nm.

2.4.2. Adsorption and desorption experiments

In all adsorption studies, the experiments were performed using batch method. 0.03 g of the studied fiber samples were placed in a small glass-stoppered bottles containing 30 mL metal ion solution with main concentration 100 mg/L (except for adsorption isotherm studies in which the concentration ranged from 10 to 400 mg/L), at 30 °C (except for thermodynamic studies in which the temperature ranged between 20 and 40 °C), pH 5 (except in pH studies in which the pH ranged between 1 and 5 using KCl/HCl for pH 1, 2, and 3; CH₃COOH/CH₃COONa for pH 4 and 5) and contact time 180 min (except for the kinetic studies in which the reaction time ranged between 10 and 120 min). The bottles were equilibrated on a thermostated shaker at 150 rpm. The percent removal and the amount adsorbed can be estimated according to the following mathematical expressions:

$$\text{Percent removal (\%)} = \frac{(C_i - C_e) \times 100}{C_i} \quad (2)$$

$$q_e = \frac{(C_i - C_e)V}{W} \quad (3)$$

where *C_i* (mg/L) and *C_e* (mg/L) initial and equilibrated metal ion concentrations, respectively; *q_e* (mg/g) adsorption capacity; *V* (L) volume of added solution and *W* (g) the mass of the adsorbent (dry).

The desorption experiments were performed as in the following: initially the precious metal ions loaded C-PTS chelating fibers were prepared by soaking 0.1 g of the fibers into 100 mL (100 mg/L) metal ion solution at pH 5.0 for 3 h and at 30 °C. The batch was equilibrated on a thermostated shaker adjusted at 150 rpm. Then, the metal ion loaded fibers were removed, washed with distilled water to get rid of the unadsorbed ions and then agitated with 100 mL 0.1 N HNO₃ solution for 60 min. The concentrations of the desorped metal ions were estimated utilizing atomic absorption techniques. The reusability of the chelating fibers were examined by repeating the above adsorption–desorption cycles for five times and the desorption percentage (*D*%) was calculated as Eq. (4).

$$D\% = \frac{C_{\text{HNO}_3}}{C_{\text{ad}}} \times 100 \quad (4)$$

where *C_{HNO₃}* is the metal ion desorbed to the HNO₃ solutions (mg/L) and *C_{ad}* is the metal ion adsorbed onto the resin (mg/L).

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