



Curcumin based biocompatible nanofibers for lead ion detection

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ABSTRACT

In this study, we aimed to develop a simple, biocompatible and selective colorimetric sensor strip for detection of lead (Pb²⁺) using curcumin loaded cellulose acetate (CC-CA) nanofibers. The nanofibers were fabricated by electrospinning method. Curcumin functionalized nanofibers were characterized by various techniques such as polarizing optical microscope, spectrofluorometry, FTIR and HR-TEM. The average diameter of the optimized polymeric nanofibers is 104 ± 0.016 nm. The fabricated sensor strips undergo a color change from yellow to orange on interaction with Pb²⁺. The selectivity of the sensor system was studied against various heavy metal ions such as Ba²⁺, Ca²⁺, Co²⁺, Cu²⁺, Mg²⁺, Ni²⁺ and Zn²⁺ and was observed that the sensor system was selective towards Pb²⁺. This method provides the advantages of simplicity and cost efficiency for quantitative detection of Pb²⁺ with a detection limit of 20 μ M by visually and 0.12 ± 0.01 μ M from the linear graph with the co-efficient of 0.997. To the best of our knowledge, this is the first work reporting on detection of Pb²⁺ based on curcumin nanofibers and the results are promising for development of low-cost disposable sensors for rapid and real-time applications.

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1. Introduction

Quantitative detection of heavy metals is of great importance in the field of environmental, water and food monitoring. Heavy metals such as lead, mercury, arsenic and chromium (Pb²⁺, Hg²⁺, As³⁺ and Cr²⁺) are essential for human metabolism but become toxic if present in excess [1]. The side effect of heavy metal includes headaches, nausea, vomiting, abdominal pain, neurological disorders and liver damage [2–5]. Hence, research and development in the area of environment and biomedical research received good attention in the detection of heavy metals [6,7]. Lead belongs to the family of heavy metals and its pollution causes serious threat to human health and environment. According to World Health Organization (WHO), the maximum permissible level of Pb²⁺ is 0.05 mg/L and the elevated levels of Pb²⁺ can cause serious diseases including high blood pressure, cardiovascular, hypertension, neurological problems, liver and kidney functions. It is more harmful to children and affects the IQs and mental disorders [8–11].

A variety of conventional methods are being used to detect Pb²⁺, such as atomic adsorption/emission spectrometry, inductively coupled plasma mass spectrometry, anodic stripping voltammetry and

reversed-phase high-performance liquid chromatography coupled with UV–vis or fluorescence detection [12–15]. Despite their efficiency in detecting Pb²⁺, they have several limitations such as multi-step processes, trained person, laboratory based detection (off-site), time-consuming and require sophisticated equipments. Hence, it is essential to develop new and user-friendly methods for simple, inexpensive, reliable and rapid method for detection of Pb²⁺. Colorimetric sensors received great attention owing to their advantages of easy to use, low-cost and on-site applications [16]. Various reports showed the use of nanomaterials, polymers, DNAzymes, oligonucleotides and proteins for detection of Pb²⁺ [17–21]. However, the search for new materials and sensor devices is ever demanding considering the problems associated with Pb²⁺ detection.

Curcumin [1,7-bis (4-hydroxy-3-methoxyphenyl)-1, 6-heptadiene-3, 5-dione], a main constituent of rhizomes of *Curcuma longa*, is commonly used as a natural food additive [22]. Curcumin possesses excellent pharmacological activities. The 1, 3-diketone moiety of curcumin form a stable keto–enol tautomeric form and it can readily chelate with the heavy metal ions to form complexes and is also an effective scavenger molecule for free radicals [23–25]. Owing to its properties, curcumin is widely used in treatment of various diseases including cancer. However, limited work has been done on the use of curcumin for sensor application. Feng et al. [26] studied the detection of Yttrium in the presence of sodium dodecyl benzene sulfonate based on fluorescence variation with a detection limit of 0.1583 ng mL^{−1}. Wu et al. [27] demonstrated

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the use of curcumin for colorimetric and fluorescent sensing of fluoride ion. Chaicham et al. [28] synthesized the derivatives of difluoroboron curcumin and studied the photophysical properties for cyanide detection. Recently, Bettini et al. [29] reported the curcumin capped Ag nanoparticles for the removal of nickel ion. Most of these works involves the curcumin in its aqueous phase.

In this context, we aimed to establish a new and simple method for detection of Pb^{2+} using curcumin based nanofibers. Electrospun nanofibers have great potential for sensor applications because of their large specific surface area, porosity and ease of functionalization. Cellulose acetate (CA), a natural biopolymer present in the cell wall of the green plants and animals is used in this study [30,31]. The suitable solvent system for preparing CA nanofibers is acetone/N, N-dimethylacetamide (DMAc) (2:1) as a dual solvent which provides an average fiber diameter in the range of 100 nm–1 μm [32]. CA has been used as a good adsorbent for the removal of heavy metal ions. Taha et al. reported the use of nanofibrous membrane for removal of Cr^{4+} using amine-functionalized cellulose acetate/silica composite with maximum adsorption capacity of 19.46 mg/g [33]. Tian et al. demonstrated a CA nanofibrous membrane modified with poly (methacrylic acid) for the adsorption of Cu^{2+} , Hg^{2+} and Cd^{2+} ions [34]. Ting et al. used cellulose based nanofiber membrane for Cu^{2+} , Cd^{2+} and Pb^{2+} with adsorption capacities of 30.96, 19.63 and 34.70 mg/g, respectively [35]. Recently, Li et al. reported the colorimetric sensor strips for detection of Pb^{2+} (LOD of 0.2 μM) using polyamide-6/cellulose nanofibers immobilized with nanogold probe [36].

In this work, we have developed a simple and biocompatible colorimetric strip for the detection of Pb^{2+} . CC-CA nanofiber membrane was prepared using electrospinning method. This membrane was directly used for Pb^{2+} detection without any further treatment or modification. The membrane exhibits visual color change from yellow to orange on interaction with Pb^{2+} . The CC-CA nanofiber membrane exhibits specific response for the detection of Pb^{2+} . To the best of our knowledge, this is the first work reporting the detection of Pb^{2+} with the combination of cellulose acetate and biocompatible curcumin dye.

2. Experimental

2.1. Materials

Curcumin was purchased from Himedia. Cellulose acetate (CA), acetone and N, N-dimethylacetamide (DMAc) were purchased from Loba Chemie. Barium chloride, cadmium sulfate, calcium sulfate, cobalt acetate, lead acetate, copper acetate, magnesium chloride, nickel acetate and zinc acetate were purchased from Merck Chemicals. All other chemicals are analytical grade and used without further purification.

2.2. Fabrication of CA and CC-CA nanofibers

Various concentrations (6, 8 and 10 wt%) of CA solution were prepared by dissolving the calculated amount of polymer in dual solvents acetone: DMAc at room temperature. Before electrospinning, the polymer solution was stirred for 7 h until the formation of homogenous solution without any air bubbles. After the preparation of clear polymer solution, nanofibers were spun using electrospinning method. Similarly, CC-CA solutions were prepared by dissolving 1 mM of curcumin powder to the optimized concentration of CA polymer solution. The mixture was continuously stirred for 6 h at room temperature.

The prepared CA polymer solution was electrospun using a syringe with an 8 gauge stainless needle at an applied voltage of 15–25 kV, tip to collector distance of 10–25 cm and a flow rate

of 0.2–0.5 mL/h. Electrospinning of cellulose acetate (CA) polymer solution was performed in horizontal alignment having a grounded aluminum foil which serves as a collector.

2.3. Characterization methods

The preliminary optimization of nanofibers was carried out by polarizing optical microscope, Leica (DM750P-Germany). FTIR (Shimadzu–Japan) was carried out to study the functional groups present in the electrospun nanofibers. Absorption spectra were recorded using UV-T90 PG (England) spectrometer with 1.0 cm quartz cell. Fluorescence studies were carried out using spectrofluorometry, (Shimadzu–Japan). HR-TEM analysis was used to study the surface morphology of nanofibers (JEOL JEM-2100, Japan). The diameter of the nanofibers was calculated by using image J software.

3. Results and discussion

In this work, we have fabricated CA and CC-CA nanofibers using electrospinning method. The fabricated nanofibers were exposed to Pb^{2+} , which shows visual color change from yellow to orange in the concentration range from 10 nM–1 mM.

3.1. Optimization of CA and CC-CA nanofibers

CA solution were prepared in various concentrations (6, 8 and 10 wt%). At low concentration (6 wt%), only polymer drops are formed due to the dominance of surface tension than the electrostatic force. With 8 wt%, beaded nanofibers were obtained. Smooth and uniform nanofibers were formed with a polymer concentration of 10 wt% without beads. Hence, 10 wt% of polymer solution was selected as an optimized concentration for the preparation of CA nanofibers. At the flow rate of 0.2 mL/h, uniform nanofiber was observed due to the enough time for evaporation of the solvent compared to high flow rate (0.3–0.5 mL/h) conditions. The average nanofiber diameter was larger than those of the nanofibers spun at lower flow rate (0.2 mL/h). In low or weaker applied voltage (15 and 20 kV), nanofibers were not uniform and the diameter of the nanofibers were larger compared to higher voltage 25 kV. Hence, in the case of high voltage, solution stretching takes place due to the stronger columbic forces and this leads to reduction in the fiber diameter. Fig. 1(a) shows the polarizing optical microscope image of the optimized CA nanofibers spun with a 20 cm distance and Fig. 1(b) shows the prepared CA nanofiber membrane.

Similarly, CC-CA nanofibers were prepared by optimizing the parameters such as concentration of the polymer solution, flow rate, tip to collector distance and applied voltage. For the electrospinning process, a high applied voltage (25 kV) was applied. The electrospun nanofiber was collected on a collector (aluminum foil) placed at a distance of 20 cm from the syringe tip. The flow rate of the polymer solution was 0.2 mL/h. Fig. 2 shows the polarizing optical microscope image of CC-CA nanofibers and CC-CA nanofiber membrane.

3.2. FTIR studies

The FTIR spectrum of curcumin is given in Fig. 3(a). For curcumin, the bands observed at 3510 cm^{-1} , 1627 cm^{-1} , 1504 cm^{-1} , 1280 cm^{-1} and 1145 cm^{-1} are attributed to the phenolic –OH stretching, –C=O group, –C=C vibrations, aromatic –C–O stretching and C–O–C stretching modes, respectively. The FTIR spectrum of CA nanofibrous membrane was shown in Fig. 3(b). The peak absorbed at 1743 cm^{-1} was corresponding to –C=O stretching of acetyl group. Absorption band at 1426 cm^{-1} was assigned to the asymmetric vibration of –CH₃. CC-CA electrospun nanofiber was

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