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Fabrication of polyaniline nanofibers by chronopotentiometery

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ABSTRACT

We have electrochemically synthesized polyaniline nano fibers with optimized process parameters (viz. concentration of monomer and dopant, applied current density, deposition time, etc.) on ITO coated glass substrate. The nano fibers of polyaniline were subjected to UV Visible, SEM and Raman spectroscopy. UV Vis spectra show two prominent peaks at 317 and 418 nm, which confirm the presence of different forms of polyaniline. Raman spectra confirm the formation of polyaniline. SEM image of synthesized nano fibers showed a flower like structure with an isotropic growth rate. Copyright © 2012 VBRI press.

Keywords: Polyaniline; chronopotentiometery; nanofibers; UV-visible; SEM; Raman spectroscopy.



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Introduction

Conducting polymers, which are treated as synthetic metals in modern vocabulary have opened new horizons in the industrial as well as in the scientific world [1]. Conducting polymers show outstanding properties viz. modulated conductivities [2-4], chemical specificities, easy processing and environmental stability. These fascinated chemists and physicists to explore them in various scientific and technological applications. Conducting polymers have emerged as a great tool in various fields such as sensors [5-8], smart materials [9], electronics [10] and drug release systems, etc. [11-13].

Among the conducting polymers, polyaniline has attracted much interest due to its interesting properties such as electrical conductivity and environmental stability. It differs from other conducting polymers such as polyacetylene, polythiophene and polypyrrol in terms of electronic states that can be controlled by variation in

number of electrons and protons per repeated unit. Moreover, the ease in synthesis and processibility, polyaniline has applications in the area of sensors, light emitting diode, electrochemical capacitor, rechargeable batteries, etc.

Conducting polymers synthesized in the form of nano materials extensively of particular concern since their properties differ from the properties of corresponding macroscopic materials. Recently, different forms of polyaniline including nano wires, nano tubes, nano fiber, etc. have been investigated due to their innovating applications [14-18]. Various methods have been adopted for the synthesis of nano structure such as micro emulsion [19-20], soft and hard template methods [21], interfacial polymerization [22] etc. However, these methods require relatively large amounts of surfactants and it is very difficult to use the surfactants after polymerization. This shortcoming can be reduced by directly deposited polyaniline nano fibers onto the substrate. In addition, it had been found that electrochemically synthesized polyaniline films show different transitions, suggesting that the film have good porosity and conductivity can be controlled to some extent. Electrochemical synthesis has been widely used for the preparation of polyaniline nano structure films. The nano structure and morphology of conducting polymers play important roles in determining material properties and the high surface to-volume ratio of the nanostructures make them potential candidates in a variety of applications [23-24]. Recently, various authors fabricated polyaniline nano fibers for gas sensing applications [25-26]. Zhang et al. [27] synthesized aqueous dispersed conducting polyaniline nanofiber, new electrode material for super capacitor through pseudo-high dilution technique. Recent reviews on polyaniline nano fibers cover synthesis methods of nano fibers, properties and applications of nano fibers as well as modifications of polyaniline nano fibers for novel applications [28].

Herein, we report a versatile electrochemical approach for the synthesis of polyaniline nano fiber under ambient condition. We have optimized various process parameters on ITO electrode using chronopotentiometery/galvanostatic technique where a fixed oxidation current is applied with no control over the resulting potential of the system. In order to obtain the advantageous surface morphology, adhesiveness and homogenous polymer matrix of conducting polymer film, it is of paramount importance to optimize process parameters. To establish predicted results at low current density i.e. splitting of supporting electrolytes in cations and anions and controlled polymerization potential is discussed.

Experimental

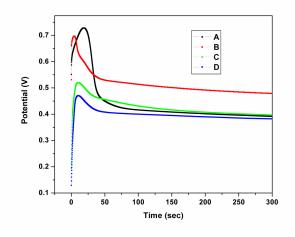
All chemicals used in the experiment were GR grade. Aniline monomer (MERCK, 99.5% purity) was double distilled prior to synthesis. Sulphuric acid (H_2SO_4) (MERCK, >99% purity) was used as a supporting electrolyte. An aqueous solution (20 ml) of aniline and dopant H_2SO_4 were prepared in 0.1:0.5 molar concentration ratio in double deionized (DID) water for

the synthesis process. The electropolymerization was carried out by the chronopotentiometery technique with CHI 660C electrochemical workstation. The standard three electrode setup was employed in one compartment electrochemical cell. A rectangular ITO sheet of size $20 \times 10 \times 0.25$ mm was used as working electrode whereas a platinum sheet of size $20 \times 40 \times 0.25$ mm was used as a counter electrode. The reference electrode was an Ag/AgCl electrode. The current density and deposition time were kept constant at 0.7 mA/cm^2 and 300 sec respectively (optimized value).

The scanning electron microscopy images were obtained using Bruker AXS, ZEISS EVO 40 EP scanning electron. The micro RAMAN investigation has been carried out using Renishaw InVia Raman microscope. The UV Visible absorption of polyaniline nano fibers deposited on ITO coated glass substrate were recorded using a U-3300 Spectrophotometer in the wavelength range 300-800 nm

Table 1. Optimization of various process parameters.

S. No.	Monomer conc. (M)	Supporting electrolyte conc. (M)	Depositio n time (sec)	Current density (mA/cm ²)
A	0.05	0.25	300	1.00
В	0.025	0.15	300	0.80
C	0.75	0.01	300	0.70
D	0.1	0.5	300	0.70
E	0.1	0.6	300	0.70
F	0.15	0.7	300	0.90
G	0.1	0.6	300	0.55



 $\textbf{Fig. 1.} \ \, \textbf{Chronopotentiograms} \ \, \textbf{recorded} \ \, \textbf{during} \ \, \textbf{synthesis} \ \, \textbf{of polyaniline} \ \, \textbf{nano} \ \, \textbf{fibers}.$

Results and discussion

Optimization of process parameters for the deposition of polyaniline thin films

Electrochemical synthesis of aniline on ITO substrate has been investigated as a function of various reaction parameters that seems to affect the formation polyaniline. Various attempts have been made for appropriate polymerization by varying the process parameters and few of them are depicted in **Table 1**. It is clear from the table

that the deposition time was constant, i.e., 300 sec. Chronopotentiograms obtained during electrochemical polymerization of polyaniline with different concentration of monomer and supporting electrolytes were shown in **Fig. 1**.

It is clear from figure that all the curves show favorable polymerization potential, while in some of the cases morphology and adhesivity was not liable for further study. However, in case of curve D we observed low polymerization potential, smooth surface, morphology and adhesivity as well as the occurrence of nano fiber. Gade et al. recorded the lowest polymerization potential for the electrochemical synthesis of polypyrrol with good surface morphology and conductivity [29] at low polymerization potential. Albeit, at low current density below 0.7 mA/cm² few curves are in correlation with the ideal curve but the deposition was not good enough, which is in good agreement with the earlier reported data [30]. Since, the nature of the curves is almost identical but the obtained polyaniline nano fibers with optimized process parameters are shown in the curve D. In fact, few curves have not been shown in figure, which overshoot during the synthesis process. This reflects the inadequacy in optimizing proper process parameters, formation of dimmers and oligomers. Since the anion transformation plays a major role during the redox process with supporting electrolyte, which reasonably ensures the favorable growth rate, adhesivity and uniformity of nano fibers.

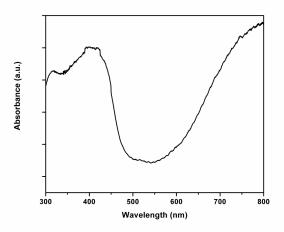


Fig. 2. UV-visible spectra of polyaniline nano fiber prepared by electrochemical polymerization.

In a few cases the behavior of the synthesis process exceeds during the first few seconds possibly reflects complicated formation of dimmers and oligomers. After proper process optimizations with low polymerization potential as obtained for curve D suggesting that building up on the plateau which results in the smoother polymerization of the polymer. Concentration gradient occurs between bulk and substrate which results in the diffusion of electrolytes and subsequent deposition on the electrode. Hence above discussed curve D ensures the proper oxidation and transformation of anions which results in good polymer film deposition. The curve smoothness of the desired curve ascribes the predominance

of polymerization. Both the mechanism and kinetics of the electrochemical polymerization of aniline were extensively investigated [31-33].

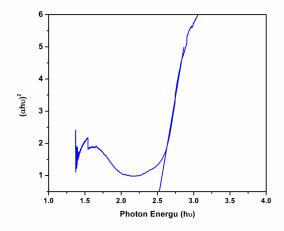


Fig. 3. The dependence of $(\alpha hv)^2$ on photon energy (hv) for as synthesized polyaniline nano fibers.

UV-Visible spectroscopy

UV-Visible spectra of polyaniline nano fiber deposited on ITO recorded in the wavelength range of 300-800 nm is shown in **Fig. 2**. In the UV visible spectrum (**Fig. 2**), two prominent absorption peaks were observed at 317 and 418 nm. This point out the presence of a polaron and/or bipolaron levels involving the valence band and conduction band. The 320 nm band is attributed to the π - π * transition in the benzoid ring [**34-35**]. The peak at 418 nm is attributed to the protonation of polyaniline (polaron and bipolarons), indicative of the conducting state. The peak position was in close agreement with literature results in conventionally prepared polyaniline nano fibers [**36-37**]. Normally a conducting polyaniline salt shows a band at 420 nm [**38**].

The optical absorption spectra can be used for the calculation of the value of an energy band gap (E_g) for polymer sample. The absorption coefficient (α) near the band edge varies with the photon energy (hv) as:

$$\alpha(hv) = B(hv - E)^n / hv, \qquad (1)$$

where n has discrete values i.e. 1/2, 3/2, 2 and 3 for direct allowed, direct forbidden, indirect allowed and indirect forbidden electronic transitions, respectively. In the studied range of wavelengths the factor B can be assumed to be constant. The value E_g for the direct transition was obtained from extrapolation of the straight line portion of $(\alpha hv)^2$ versus (hv) on the hv axes as shown in **Fig. 3**. The calculated value of band gap is 2.52 eV which is in agreement with reported earlier [39-40].

Raman spectroscopy

Raman spectroscopy was performed to characterize the structure of the synthesized polyaniline nano fibers. **Fig. 4** shows the Raman spectrum of the polyaniline nano fibers at room temperature. The characteristics Raman bands of

the polyaniline nano fiber observe at 1174, 1333, 1420, 1487 and 1595 cm⁻¹. A small band at 1174 cm⁻¹ has been assigned to an in plane deformation of the C-C bond of the quinoid ring of the polyaniline nano fibers [41]. The bands at 1382 cm⁻¹ and 1595 cm⁻¹ corresponds to C-N⁺ stretching and C-C stretching of the quinoid ring respectively [42]. A band 1487 cm⁻¹ assigned to the formation of bipolarons [43]. The band at 1247 cm⁻¹ corresponds to the C-N stretching in polaronic units [35]. However, the peak at 1407 cm⁻¹ credit to have contributions from both C-C stretching of quinoid units and ring stretching vibrations of phenazine like structures [44]. The band observed in the 1100-1140 cm⁻¹ region is the characteristics of conductive polyaniline and is due to the charge delocalization on the polymer backbone [45].

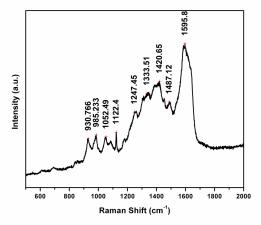


Fig. 4. Raman spectrum of polyaniline nano fiber with optimized process

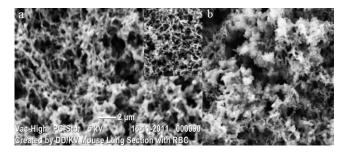


Fig. 5. SEM of polyaniline nanofiber on ITO coated glass substrate with optimized process parameters (a) showing nanofibers with fibrillar morphology and fibrillar with inset giving magnified view of nano fibers and (b) flower-like structure.

Scanning electron microscopy

Fig. 5 (a) and (b) shows the SEM images of synthesized nano fibers. It's clear from the images that there is identical growth rate of polyaniline nano fibers i.e. isotropic growth of nano fibers. Moreover, the image shows homogeneous nucleation and high porosity which indicates its reliability using as sensing material. The substrate is covered by a dense network of nanofibers with fibrillar morphology [46]. Fig. 1(b) gives magnified image of polyaniline nano fibers which appears to be a flower like structure [46-48]. Nanofiber seems to be quite flexible in nature as they are curved (Fig. 1(a) inset). Various researchers [18, 49] also found flower, lotus leaf like, rose like, cauliflower like, etc. morphology in polymer nano fibers synthesized by different methods. The diameters are in the range of 80-130 nm.

It is well reported in literature that the diameter of polyaniline nanofibers was strongly affected by the oxidization ability of oxidant, in which higher oxidation potential generated larger diameter. Although the present investigation reveals that polyaniline nano fibers have excellent porosity, which explore the possibility to use these nano fibers in gas sensing applications.

Conclusion

We have fabricated polyaniline nano fibers on ITO coated glass substrate by electrochemical polymerization using chronopotentiometery technique. A dense fibrillar structure of polyaniline nano fibers is clearly evident in the SEM images. SEM images indicate usability of nano fibers as gas sensing applications. Raman spectra indicates that the presence of a quinoid ring in polyaniline structure. UV visible spectra showed the presence of different rings, which is consistent with the earlier reported data. This paper only presents optimization of process parameters and more efforts in this regard still going on.

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