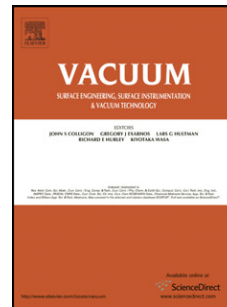


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**Two step electrochemical synthesis of Au nano particles decorated polyaniline nano  
fiber**

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**Abstract**

The present work reports the electrochemical synthesis of H<sub>2</sub>SO<sub>4</sub> doped polyaniline nano fibers (PANINFs) on conducting ITO substrate. The subsequent dissociation of HAuCl<sub>4</sub> in an acidic solution of HNO<sub>3</sub> and deposition of Au particles was carried out by using cyclic voltammetry (CV) to form Au particles decorated PANINFs (Au-PANINFs) composite film. Electrical conductivity of the Au particles decorated PANINFs has been measured by two probe method. Scanning electron microscopy (SEM) investigations of PANINFs and Au-PANINFs samples revealed good porous and fibrous structure with identical distribution of gold nano-particles coupled with the surface of PANINFs. The average diameter of the PANINFs ranges from 184nm to 210nm. X-ray diffraction (XRD) and EDAX spectra also supported the formation of Au particles on the surface of PANINFs. The structural analysis was carried out by Raman spectroscopic technique. A possible mechanism for the formation of Au-PANINFs composite has been proposed.

**Key words:** Electrochemical synthesis, polyaniline nano fiber, composite, I-V, SEM, XRD, Raman

## 1. Introduction

Composites of conducting polymers and metal particles have dormant competence to combine two leading edge technologies to yield materials with immense application in the domain of material world. Composites significantly covers a large variety of system such as one dimensional, two dimensional and three dimensional, made of distinctly dissimilar components mixed at micro and nano level. A metal containing conducting polymers amalgamates the redox properties of conducting polymer and that of metal ions [1], hence emerges with unique properties that are significant in various applications especially in gas sensing and catalytic application [2-3].

Amongst metal particles, Au has been widely used because of its unique optical and catalytic properties [3-4]. Au particles offer novel opportunities for fabricating electrochemical devices as they can facilitate electron transfer between electro-active species and electrodes [5]. On the other hand PANI is well designed and the most gifted material because of its high capacitive characteristic, low cost and ease of synthesis [6-7]. Electrochromic properties of PANI have made potential in electrochromic devices and is observed in a large number of conjugated polymers [8-9]. Au-PANI composite reveals a combination of the enviable qualities of both components like stability, high conductivity, mechanical strength and the exclusive optical properties of the metal components and easy processing favourable redox properties from conducting polymer part [10]. Liu et al. [2] studied Au nano particles decorated polyaniline nano fibers for the detection of volatile sulphur compounds in expired breath. They reported a template-free electrochemical polymerization for the fabrication of PANINFs. The formation of PANI/Au nano particles were realized by the redox reaction between the  $\text{HAuCl}_4$  and the PANI Emeraldine salt.

Metal nanocomposites have been fabricated by such as layer by layer, chemical polymerisation using the metal ions as the oxidant and reduction of metal ions and electrochemical deposition of the metal ions over the conducting polymer [11-13]. Various polyaniline-based core shell nano-composites such as Ag/polyaniline [14-15], Ni/polyaniline [16], Ag-PET [17-18], Au-silica [19] and silica-polyaniline [20-21] have been reported. The preparation of Au-PANI material is reported using preformed polyaniline by exploiting the multi-oxidative states of the polymer [22-23]. Chemical, electrochemical and radiolysis method have been adopted for synthesis of Au-PANI composites but few functional properties like easy synthesis process, adhesivity, stability and homogeneous distribution of metal particles on the surface of polymer film are yet to be achieved to surmount the teething troubles in application domain.

In this work, we report a two step electrochemical synthesis method to prepare well controlled Au-PANINFs composite. This approach has some advantages that it is simple, cost effective and ensures controlled fabrication of desired structure with optimized process parameters. Such controlled fabrication of Au-PANINFs could modify significantly the sensing or catalytic properties. Interestingly, the polymer component is used for conjugating analytes, while the gold part serves to increase the catalytic affinity/reactivity. The chronopotentiometric technique is used for fabrication of PANINFs on ITO substrate and later the synthesized surface of fibrous film is modified by the Au-particles using cyclic voltammetry which shows strong redox reactivity occurred during synthesis process. These prepared Au-PANINFs are characterized by electrical conductivity, SEM, Raman, XRD and EDAX.

## 2. Experimental details

Aniline monomer (MERCK, 99.5% purity),  $\text{H}_2\text{SO}_4$  (MERCK, >99% purity),  $\text{HAuCl}_4$  (Sigma Aldrich) and  $\text{HNO}_3$  (RANKEM) were used in the present investigation. All these reagents

were analytical grade reagents (A.R.) and used without further purifications. All the experiments were carried out with double-distilled (DD) water.

Electrochemical synthesis was carried out using a CHI 660D electrochemical workstation under computer control. The standard three electrode setup was employed in one compartment electrochemical cell. A rectangular conducting ITO sheet of size  $20 \times 10 \times 0.7 \text{ mm}^3$  was used as a working electrode whereas a platinum sheet of size  $20 \times 40 \times 0.25 \text{ mm}^3$  was used as a counter electrode. The reference electrode was an Ag/AgCl electrode. Electrochemical synthesis of PANINFs was carried out on ITO substrate in an aqueous solution of 0.1 M aniline monomer containing 0.5 M  $\text{H}_2\text{SO}_4$  using a chronopotentiometry technique. The surface modification of synthesized PANINFs was conducted in an aqueous solution of 0.0007 M  $\text{HAuCl}_4$  containing 0.005 M  $\text{HNO}_3$  solution using cyclic voltammetry (CV) between a potential window of 0.4 to 0.7 V at a scan rate of  $30 \text{ mVs}^{-1}$ .

Scanning electron microscopic images and EDAX spectra were obtained using a JEOL JSM-6490LV microscope at 25 kV after covered with a thin layer ( $\sim 10 \text{ nm}$ ) of sputtered gold. The micro-RAMAN investigation was carried out using Renishaw inVia Raman microscope. Argon laser of excitation wave length 514.4 nm was used as an excitation source. XRD pattern of the Au-PANINFs was taken using SHIMADZU XRD-7000 X-ray diffractometer with  $\text{Cu-K}_\alpha$  radiation ( $1.54 \text{ \AA}$ ) for a range of Bragg's angle  $2\theta$  ( $15 < \theta < 55$ ) at the scan rate of  $0.5^\circ/\text{min}$ . I–V characteristics measurements were made using Keithley 2400-C source meter.

### **3. Results and discussion**

#### **3.1 Electrochemical synthesis and characterization**

Electrochemical synthesis of aniline on ITO substrate has investigated as a function of various parameters such as aniline concentration, dopant, applied current density and deposition time by using chronopotentiometry technique (Table 1). The optimized values were 0.1 M, 0.5 M,  $0.65 \text{ mA/cm}^2$  and 800 sec, respectively. In this study, PANINFs film

(optimized values) was placed into 80 ml aqueous solution of  $\text{HAuCl}_4$  and  $\text{HNO}_3$  for 4-sweep segments of CV to modify the surface of polymer film at 30 mV/s scan rate in a potential range of - 0.4 to 0.7 V (Fig. 1). The scan rate was kept rather high for the four sweep segments for the uniform decoration of Au particles onto the surface of PANINFs. The scan direction of the curve is also included in Fig. 1. The formation of ion exhibits the first order dissociation of  $\text{HAuCl}_4$  in an acidic solution of  $\text{HNO}_3$ , the  $\text{AuCl}_4^-$  complex ion formed are attached to the imine sites of PANI film.

The obtained composite was green with very faint yellow shining. This change in colour can be attributed to the electrochromic process after attachment of Au ion complex or Au nano particles on the polymer surface. It is also reported that chloroaurate anions form a strong complex with the protonated imine nitrogen in polyaniline and further the chloroaurate is converted to metallic gold upon reduction at working electrode forming coordination bonding with the polymer film [24]. The changes in redox process occurring during chemical reaction in three cell compartment are clearly observed in Raman spectrum as the region 1556–1610  $\text{cm}^{-1}$  indicates that the PANINFs is in partially oxidised state which is more susceptible for attaching Au nano-particles. In case of Au-PANINFs composite strong interaction occurs between the polymer and Au-metal particles, which modify the electron transfer rate across the composite [16]. To modify the polymer surface with uniform and isotropic ionic reduction of Au-particles the scan rate was kept rather high for less sweep segments. SEM micrographs show efficient embroidery of PANINFs surface with Au-metal particles.

### 3.2 Scanning electron microscopy (SEM)

For confirmation of experimental results, the PANINFs and Au-PANINF composite was subjected to scanning electron microscopy. Fig. 2[A] shows the SEM micrograph of synthesized PANINFs. Fig. 2[B] is the magnified image of PANINFs. From these

micrographs, it is clear that PANINFs shows fibrous network with porous structure. Fig. 2 [C] show that Au-particles were uniformly dispersed on the PANINFs surface indicates the coupling of Au-particles with the surface of PANINFs. The Au-particles appear as white spots on PANINFs and dense network of Au-PANINF is clearly observed as shown in Fig. 2 [D] (magnified image). Moreover, the image shows homogeneous distribution of Au-particles and sky-scraping porosity, which indicate their reliability using as a sensing material. This type of growth of Au-PANINFs composite on ITO substrate reflects good mechanical behaviour, compactness and hence ensures its stability. The average diameter of these fiber ranges from 180 nm-210 nm. It is clear from the images that there is identical growth rate of PANINFs. Nanofibers seem to be quite flexible in nature as they are curved. Various researchers also found flower, lotus leaf like, rose like, spherical shell, cauliflower like morphology in polymer nano fibres synthesized by different methods [25-26]. It is well reported in literature that the diameter of polyaniline nanofibers are strongly affected by the oxidization ability of oxidants, in which higher oxidation potential generated larger diameter. The present investigation reveals that Au-PANINFs composite is affluent of gaps and cleavages, which explore their possibility to be used for various applications. The results indicate that the Au-PANINFs composite synthesised by the above discussed method has interesting fiber network of PANI with uniformly dispersed Au-particles.

The presence of Au-particles on the surface of PANINFs was ascertained by EDAX results. The Au-peak strongly indicates that Au-particles are decorated on the PANINFs surface. The EDAX measurements were performed in a complete area of square millimetre range to ensure the uniform distribution of Au-particles.

### 3.3 *RAMAN spectroscopy*

The structural analysis of PANINFs and Au-PANINFs composite was carried out by Raman spectroscopy. Fig. 3[A] shows Raman spectrum of PANINFs. The bands in the wavenumber



range of 1100 to 1700  $\text{cm}^{-1}$  corresponds to the stretching modes of different bonds. The benzene C–H bending deformation mode lies at 1140 to 1190  $\text{cm}^{-1}$  for the reduced semiquinone and quinoid ring structure. The different C–N stretching mode (amines, imines, and polarons) lies between 1210 and 1520  $\text{cm}^{-1}$ . Raman band at 1586  $\text{cm}^{-1}$  is attributed to the C–C stretching vibration of benzene ring [27-28]. The band at 1218  $\text{cm}^{-1}$  can be assigned to the C–N stretching mode of the polaronic units. The band at 1327  $\text{cm}^{-1}$  corresponds to the C–N<sup>+</sup> stretching modes of the delocalized polaronic charge carriers [29]. A higher intensity band at 1491  $\text{cm}^{-1}$  corresponds to the bending deformation of the N<sup>+</sup>–H unit. The bands at 1320, 1340, 1478 and 1495  $\text{cm}^{-1}$  are assigned to C–N, protonated C–N<sup>+</sup>, C=N and protonated C=N<sup>+</sup> according to literature [30]. The band at 1165  $\text{cm}^{-1}$  represents the C–H bending vibration in the aromatic ring [31]. The observed Raman bands for PANI film are in good agreement with the literature [32].

It is interesting to compare the spectra of PANINFs with Au-PANINFs composite. The Raman shifts and relative intensity are observed in the Au-PANINFs composite film (Fig. 3 [B]). This shifts in Raman bands and intensity shows the interfacial strapping amalgamation of Au-particles with the polymer film. The bands at 530, 598, 1124, 1165 and 1610  $\text{cm}^{-1}$  show increase in Raman intensity. The characteristics peaks of PANINFs at 1491 and 1586  $\text{cm}^{-1}$  appear at 1482 and 1610  $\text{cm}^{-1}$  in the composite film. The shifts in the Raman bands clearly credited to the deposition of gold particles on the surface of PANINFs. Baibarac et al. [33-35] have investigated the surface enhanced Raman spectrum (SERS) of PANI thin film on Au, Ag, and Cu substrates and verified that SERS spectra of PANI depends on the oxidising properties metallic surface which initiates chemical reaction at the polymer/metal interface. Since the PANINF is partially oxidised state i.e. emeraldine salt and is treated more conductive state. The small Raman band at 1327  $\text{cm}^{-1}$  gets shifted towards higher frequency

number  $1338\text{ cm}^{-1}$  which appears more prominent after reduction of Au-particles. These bands correspond to asymmetrical-N stretching [36-37]. The peaks located at higher frequency side in the spectrum range of  $1122\text{-}1165\text{ cm}^{-1}$  are assigned C-H in plain deformation quinoid rings and vibration of bipolaronic forms [38]. Peak at  $806\text{ cm}^{-1}$  assigned to the amine deformation (C-N-C bending) [39]. In the spectrum, the small band centred at  $954\text{ cm}^{-1}$  is assigned to the ring deformation associated with the dication [40]. The Raman bands at  $2102$  and  $2165\text{ cm}^{-1}$  shows the significant fraction of strong C=C chains i.e. creation of unsaturated bond making it more conjugated, which is a significant factor for composite to be in a good conducting state. These conjugated system of p-orbitals with delocalised electrons causing lattice distortion which results the creation of polaron and bipolaron.

### 3.4 X-ray diffraction

Fig. 4[A] shows the XRD spectrum of PANINFs. The broad peak at about  $2\theta = 25.26^\circ$ , which is the characteristic of PANI [41-43]. XRD pattern of Au-PANINFs composite film is shown in Fig. 4[B]. The composite film exhibit the well-known peaks in the  $2\theta$  values of about  $38^\circ$  and  $43^\circ$ , which are ascribed to the (111) and (200) planes of standard cubic faces of gold, respectively and were in line with the reported data, showing the existence of Au particles in the Au-PANINFs composite[43-44]. Also, the broad peak in PANINFs at  $2\theta = 25.26^\circ$  slightly reduced and shifted to lower angle ( $2\theta = 25.26^\circ$ ) after deposition of Au particles. The small peak at  $23.5^\circ$  is ascribed to the periodicity parallel to the polymer chains of PANI fiber [42]. The peak tends to disappear after deposition of Au-particles which may be due to the decrease in the amorphous nature of PANINF in Au-PANINFs composite [45].

### 3.5 I-V characteristics

The electrical conductivity of PANI and Au-PANI composite film was measured at room temperature by two probe method using Keithley source meter. Fig. 5 shows the I-V

characteristics of PANINFs and Au-PANINFs. The value of the electrical conductivity of Au-PANINFs was found to be  $3 \times 10^{-2} \text{ Scm}^{-1}$ , whereas that of PANI was found to be  $6.2 \times 10^{-3} \text{ Scm}^{-1}$ . It is clear that there was an increase in conductivity of about one order of magnitude upon the decoration of Au particles in the PANINFs film surface. This indicates that incorporation of Au particles in the PANINFs surface enhances the conductivity of composite films. The increase in conductivity is due to the metallic nature of gold. The increase in conductivity reflects good dispersion of Au particles in the PANINFs surface. Feng et al. [21] reported that the electrical conductivity of PANI/Au composite hollow spheres was more than 3 times higher than that of the pure PANI hollow spheres synthesized by using polystyrene/sulfonated polystyrene core/shell gel particle templates. In another study Feng et al. [43] reported that the conductivities of PANI/Au composites synthesized by self assembly process in the presence of camphorsulfonic acid and hydrochloric acid as dopants respectively become higher than that of pure PANI. In a similar study Sivakumar and Gedanken [41] show an increase in conductivity of about one order of magnitude upon the incorporation of Au nanoparticles in the conductive polyaniline, prepared by sonochemical method. These observations are in good agreement with the Raman spectral analysis.

#### **4. Conclusions**

In this article a facile technique for the synthesis of PANINFs and its surface modification by Au particles by using electrochemical technique are reported. The decoration of PANINFs with Au particles results in the enhancement of conductivity. SEM images shows porous and fibrous structure of PANINFs with decoration of Au particles coupled with the surface of PANINFs. XRD and Raman spectrum revealed the formation of composite system. Thus, it is worth mentioning that various nano structure of conducting polymers and their surface modification by metal particles can be exploited using two step electrochemical techniques.

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**Figure Captions:**

**Fig. 1:** The cyclovoltgramm Au-PANINF composite between potential windows -0.4 V to 0.7 V in a solution of  $\text{AuCl}_4+\text{HNO}_3$  in a ratio of 0.0001:0.005M with scan rate of 30 mV/sec for 4-sweep segments

**Fig. 2:** SEM images of PANINF [A-B] and Au decorated PANINFs between potential window -0.4 V to 0.7 V in a solution of  $\text{HAuCl}_4+\text{HNO}_3$  in a ratio 0.0001:0.005 with scan rate of 30mV/sec for 4-sweep segments [C-D].

**Fig. 3:** Raman spectrum of [A] PANI nanofiber and [B] Au-PANINF composite

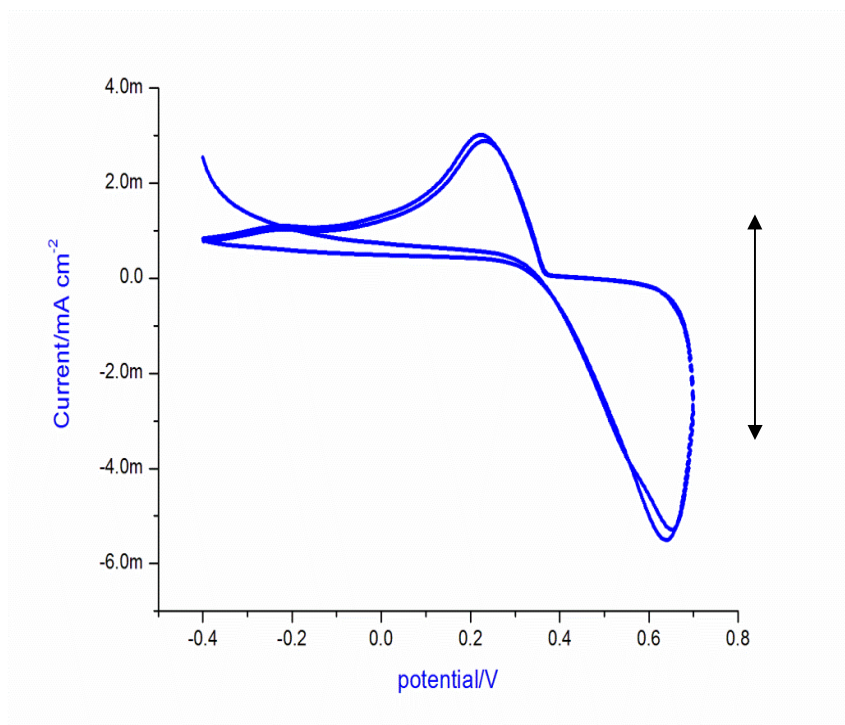
**Fig. 4:** XRD pattern for [A] PANI nanofiber and [B] Au-PANINF composite

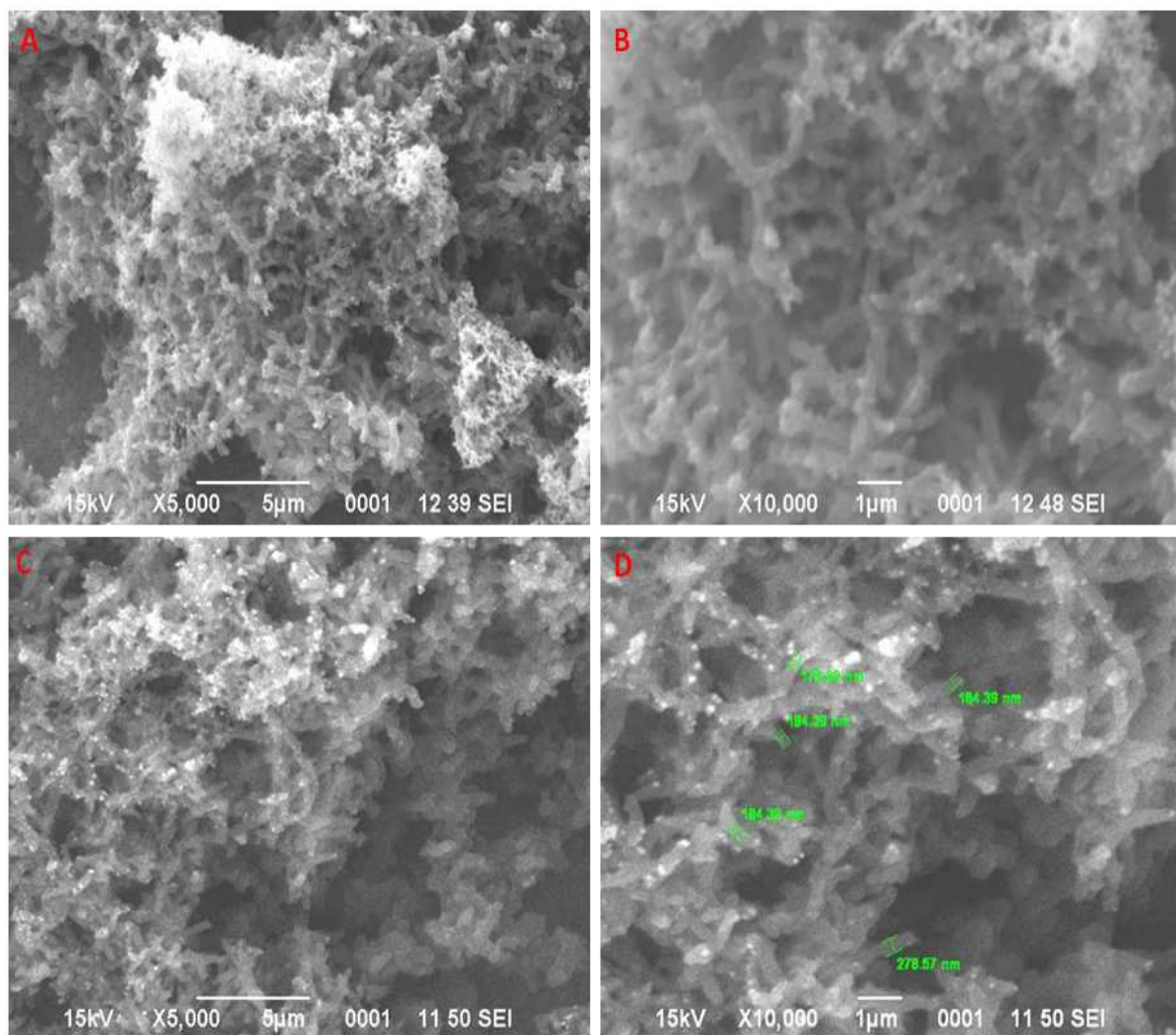
**Fig. 5:** Current voltage plots of PANINFs and Au decorated PANINFs

**Table 1:** Optimization of various process parameters for the fabrication of PANINFs

S. No.	Monomer conc. (M)	Supporting electrolyte conc. (M)	Deposition time (sec)	Current density (mA/cm <sup>2</sup> )
A	0.05	0.3	800	0.9
B	0.08	0.5	800	0.65
C	0.09	0.55	800	0.7
D	0.4	1.7	800	0.7
E	0.7	1.2	800	0.8
F	0.1	0.25	800	0.5
G	0.1	0.5	800	0.65
H	0.1	0.55	800	0.7



**Figure 1**

**Figure 2**

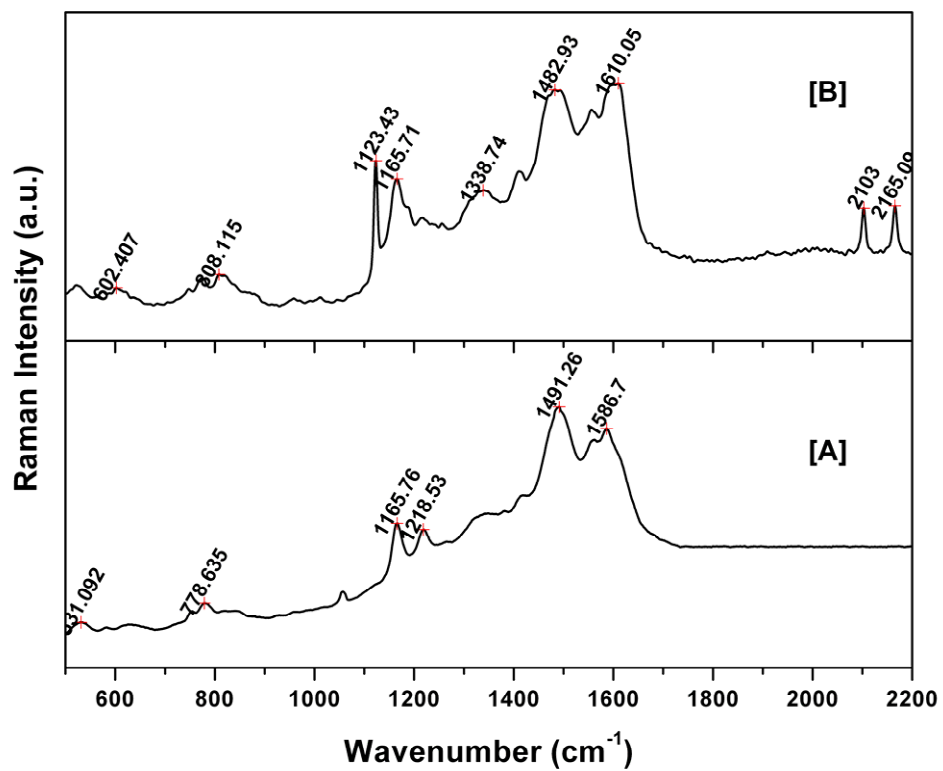


Figure 3

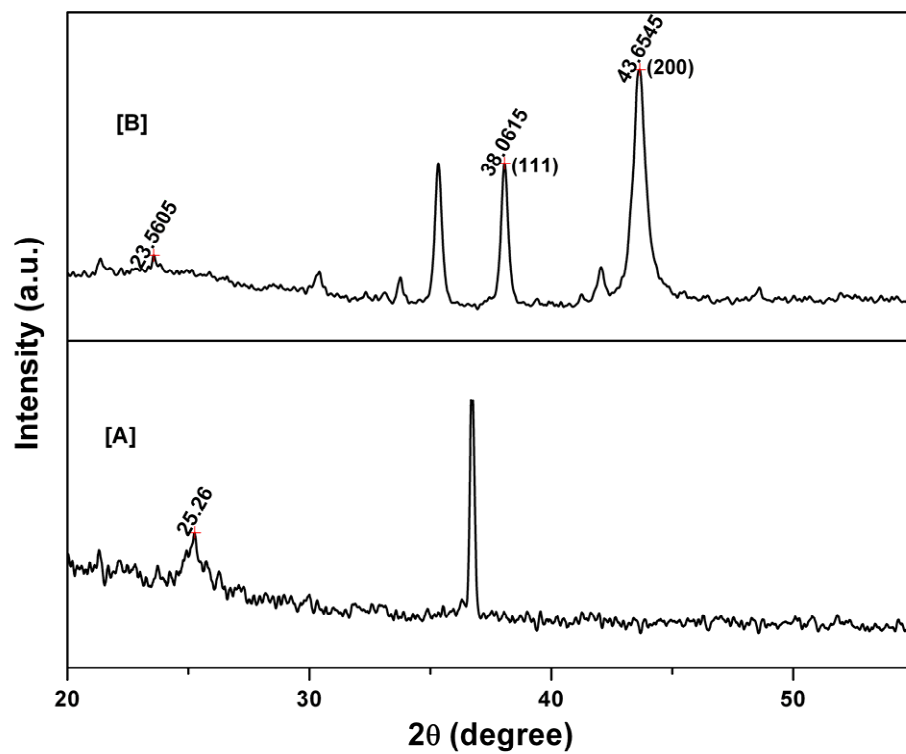


Figure 4

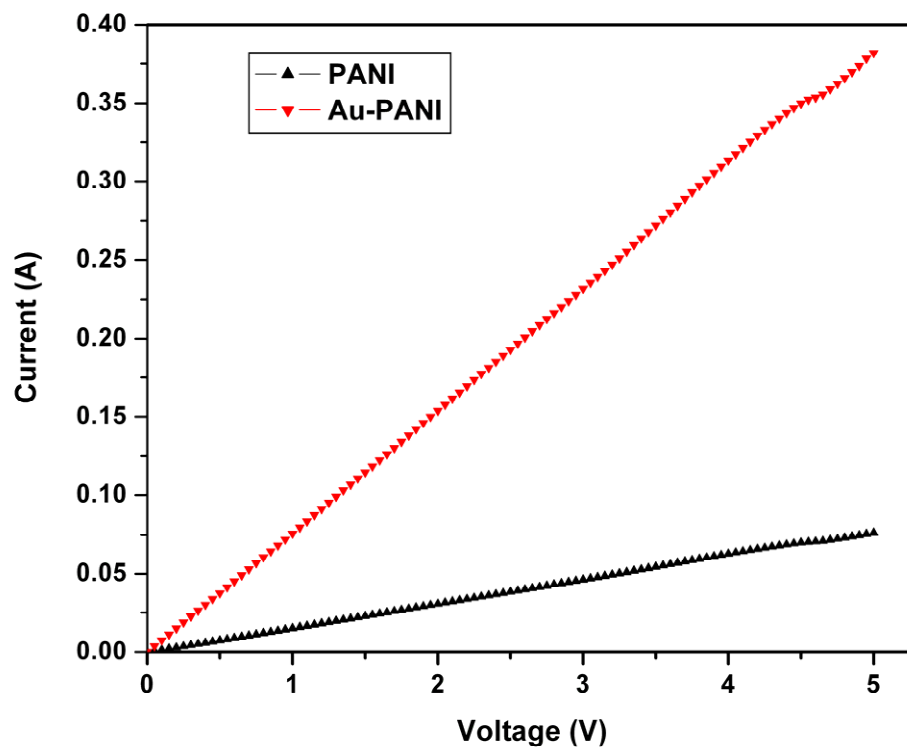


Figure 5

**Research Highlights**

- Decoration of polyaniline nanofibers by gold nanoparticles using CV
- SEM shows good porous and fibrous structure with identical distribution of gold nano-particles
- Structure analysis by using Raman and XRD
- I-V characteristics of Au-PANINFs