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Physioelectrochemical investigation of supercapacitive performance of ternary nanocomposite by common electrochemical methods and fast fourier transform voltammetry

JavadShabaniShayeh[a*], Ali Ehsani[b], AydaNikkar[c], Parviz Norouzi[a, d*], Mohammad RezaGanjali[a, d] and Mateusz wojdyla[e]

a. *Center of Excellence in Electrochemistry, University of Tehran, Tehran, Iran.*
b. *Department of Chemistry, Faculty of science, University of Qom, Qom, Iran.*
c. *young Researches and elite club, Islamic Azad University Tabriz branch, Tabriz, Iran*
d. *Endocrinology & Metabolism Research Center, Tehran University of Medical Sciences, Tehran, Iran.*
e. *Poznan University of Technology, Institute of Chemistry and Technical Electrochemistry, Piotrowo 3, 60965 Poznan, Poland*

Corresponding Authors E-mail:
shabanijavad@ut.ac.ir
pnorouzi@gmail.com

Abstract

Ternary nanocomposite materials of polyaniline as supercapacitor electrodes with remarkably high specific capacitance are electro synthesized on a glassy carbon electrode (GCE). Scanning electron micrographs clearly revealed the formation of nanocomposites on the surface of the working electrode. The supercapacitor properties of composite films are investigated by using cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS), galvanostatic charge/discharge andfast fouriertransformconsecutive cyclic voltammetry (FFTCCV) technique in acidic solution. Very high specific capacitance of 303 F/g is obtained from PANI in the presence of reduced graphene oxide and Au nanoparticles in acidic electrolyte. Furthermore,FFTCCVtechnique is considered as the powerful technique in characterization of new composite materials as supercapacitor. By using this technique, electrochemical behavior of the system is monitored momentarily. The absolute capacitive charge changes (ΔQ_n) calculated from three-dimensional (3D) CVs of composite electrode at the scan rate of 50 mV/s and computer program algorithm. The present study introduces new nanocomposite materials for electrochemical redox capacitors with advantages including long life cycle and stability due to synergistic effects of each component**.**

1 Introduction

Nowadays a great attention has been attracted by supercapacitors (SCs) due to their application in many technologies such as electric vehicles, uninterruptible power supplies, DC power systems, and mobile devices[1-3]. There are two assortment about charge storage mechanism, electrical double layer capacitors (EDLC) and psudocapacitors which store charge by non-faradic and faradic reactions, respectively[4-7].Conductive polymers (CPs), metal oxides and carbon materials are the active materials that used for SCs[8-10].Charge storage mechanism in CPs proceeds by formation of polycations, causing the anions in the solution to intercalate into the CP in order to maintain electro neutrality. Repeating charge-discharge process leads to accumulation of stress on polymer, which is related to poor cycle life. One of the solutions for this problem is using nano materials to synthesize nanocomposites[11-14].Polyaniline (PANI), Polypyrrole, polythiophene are some of most famous CPs[15]. Many reports have been published about PANI nano material composites[16].

Graphene is one those nano materials which due to its unique electronic and mechanical properties become a very promising material in this field[17]. Using noble metal to get composite lead to enhance the electrical properties of graphene[18]. Recently ternary composites which consist PANI/graphene/noble metals are very attractive for researchers. Increasing the electrochemical performance of composite material compare to binary composite achieved by scientists[19-21].One of the critical case in study of SCs is the electrochemical methodwhich used by researchers. There are some famous methods like as CV, CD and EIS which have been used in all papers. Using new electrochemical method for study of SCs can release new data about the nature of materials. FFTCCVis one of new electrochemical techniques which used for study the SCs[15]. 3D CVs, diffrentiative CVs and stability of electrodes are some features of this techniques which can be used for describe the capacitive performance of electrochemical systems. In this work, we followed the electrochemical properties of Polyaniline/reduced graphene/AuNPs(PANI/rGO/AuNPs) as good candidates for active materials in supercapacitors by FFTCCV technique. Our goals in this paper is increase the capacitance and stability of composite electrode compare to PANI one. Furthermore by using FFTCCV technique we plan to illustrate some new electrochemical performance of ternary composite.

2 Experimental

2.1 Reagent and materials

All the chemical materials used in this work, obtained from Merck Chemical Co. with the analytical grade and used without further purification. Double distilled water was used throughout the experiments. Aniline (ANI) was doubly distilled and the resulting colorless liquid was kept in the dark at 5°C.

2.2 Apparatus

Electrochemical experiments were carried out by an Auto lab General Purpose System PGSTAT 30 (Eco-chime, Netherlands). A conventional three electrode cell with an Ag/AgCl reference electrode (Argental, 3 M KCl) was used in order to carry out the electropolymerisation of the PANI. A platinum wire with a diameter of 0.5 mm and an exposed area of 0.65 cm^2 was used as the counter electrode. A glassy carbon electrode with an area of 0.03 cm^2 was used as the working electrode. A wide frequency range of 10 mHz to 100 kHz was used in EIS. Morphological investigations of the polymeric films were carried out by using SEM (Philips XL 30).

2.3 FFTCCV technique

The FFT experimental data collection was performed with the help of the following equipment; a setup of a computer, equipped with a data-acquisition board (PCL-818HG, Advantech. Co. And a custom-made potentiostat described in our previous works. A computer program that was developed in Delphi6® environment used for data acquisition and data processing. The signal Calculation in this method is established based on the integration of net current changes over the scanned potential range. It must be noted that in this case, the current changes at the voltammograms can be caused by various processes, which take place at the electrode surface. In detail, a CV of the electrode was firstly recorded. Then, the existing high frequency noises were indicated by applying FFT on the collected data. With the help of this information, the cut off frequency of the analogue filter was set at a certain value where the noises were removed from the CV[15, 22-28].

2.4 Preparation of graphene oxide (GO) and rGO/Au NPs

Graphene oxide was synthesized from commercial graphite by modified Hummers method[29]. Graphite (10 g) and concentrated H_2SO_4 (230 mL) were stirred at constant temperature (below

20°C). After that $KMnO_4$ was added to suspension and reaction temperature was changed to 40 °C and the mixture was stirred for 1h. Then 500 ml deionized water was added to the reaction cell by increasing the temperature to 100 °C. After this step H_2O_2 (2.5 ml) was added slowly, followed by addition of deionized water (500 mL). Then, the solution was washed with HCl (200 mL) and deionized water until the filtrate became neutral and the remaining impurities were removed. The product, graphiteoxide, was exfoliated in deionized water in an ultrasonic bath to form graphene oxide (GO) Nano sheets.The GO powder was added to water dispersed Au nanoparticles and uniformly dispersed by sonication for 1 h. This suspension was then stirred 24 h at 40 °C. Afterwards, the mixture was filtered using a Buchner funnel and washed with deionized water three times. The final product was dried at 50 °C for 12 h[30].

2.5 Synthesis of PANI and PANI/rGO/AuNPs composite electrodes

PANI/rGO/AuNPs composite was synthesized electrochemically by cyclic voltammetry in 1 M H_2SO_4 solution contained aniline monomer (0.03M), rGO/AuNPs (0.2% wt) and sodium dodecyl sulfate (0.005 M) that dispersed in solution by sonication. PANI electrode was synthesized in same solution without rGO/AuNPs and sodium dodecyl sulfate. Electropolymerisation were conducted by 10 cycles at the sweep rate of 50 mV/s. The mass of PANI films was approximated assuming a current efficiency for the electropolymerisation process of 100%, using Faraday's law . SEM images of PANI and PANI/rGO/AuNPs film are shown in Figure 1.

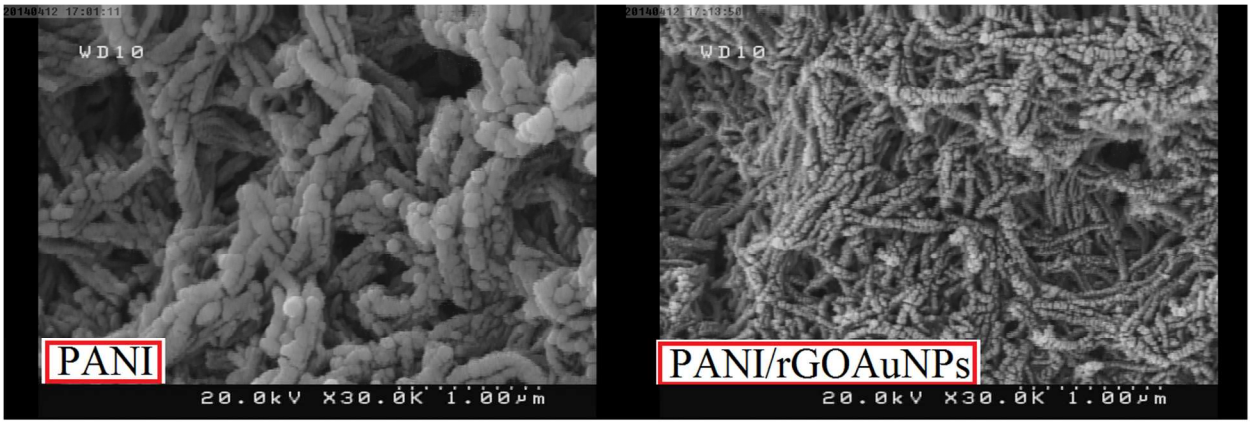

Figure 1. SEM images of PANI and PANI/ rGO/Au NPs.

3 Results and discussion

3.1 Cyclic voltammetry studies

PANI and PANI/rGO/AuNPs electrodes were prepared by CV technique and then placed in 1M H_2SO_4 solution for electrochemical studies. The electropolymerization process conducted by 10 CVs at the scan rate of 50mV/s in solution contained aniline monomer, rGO/AuNPs and SDS for composite electrode and aniline monomer without any additive for PANI electrode.Figure 2 presents the CVs of PANI and PANI/rGO/AuNPs electrodes in 1 M H_2SO_4 solution at the sweep rate of 25 mV/s. A redox peak in the potentials between 0.5 – 0.6 V is attributed to transition of PANI from emeraldine form to the pernigraniline form[31].As observed, the capacitance of ternary electrode is about two times greater than that of PANI electrode, whichshows using rGO/AuNPs in polymer matrix enhances the capacity of electrode.

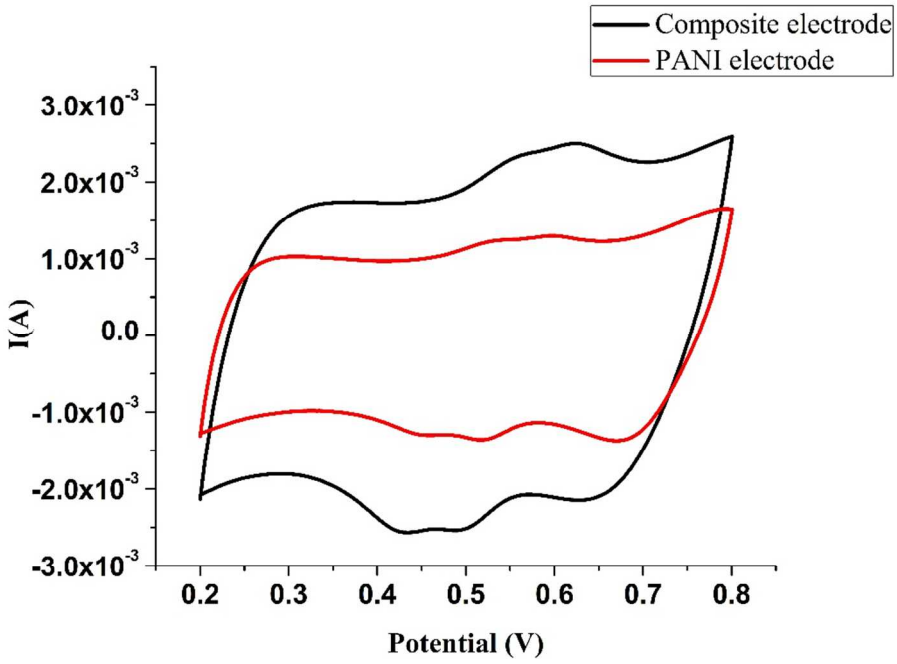

Figure 2.Cyclic voltammograms of PANI and PANI/rGO/AuNPs electrodes in 1M H_2SO_4 at the sweep rate of 25 mV/s in the potential window of 0.2 – 0.8 V.

The CV of PANI/rGO/AuNPs electrode (Figure 2) exhibits nearly symmetrical rectangular shapes in potential window, that shows the incorporation of rGO/AuNPs in PANI matrix not

only increase the capacitance of composite electrode but also save itsideal capacitive behavior.Enhancement in capacitance of composite electrode compare PANI could attributed to accrue the electric double-layer capacitance produced by graphene and pseudo capacitive behavior of PANI.

Specific capacitance of electrodes is calculated from CV curves according to following equation:

$$C = \frac{I}{mv} \qquad (1)$$

Where I is the current, m is the mass of reactive material and ν is the potential scan rate. Thespecific capacitance of PANI and PANI/rGO/AuNPs electrodes were found to be 190 and 303 F/g, respectively. Compare with some reports that used reduced graphene oxide[32-34], this ternary composite electrodehas more advantage than others due to very low magnitude of nano material (0.2wt%). This results show that using electrochemical method for synthesis of composite electrodes is more suitable than insitu process.Figure 3, shows CV curves of PANI/rGO/AuNPs electrode at various scan rates in 1 M H_2SO_4 media. As presented, excellent capacitive performance of the PANI/rGO/AuNPs electrode is also verified from these curves. According to CV results, by increasing scan rate, the current response of the composite filmincreased. This behavior can be related to an idealcapacitive of PANI/rGO/AuNPs electrode.

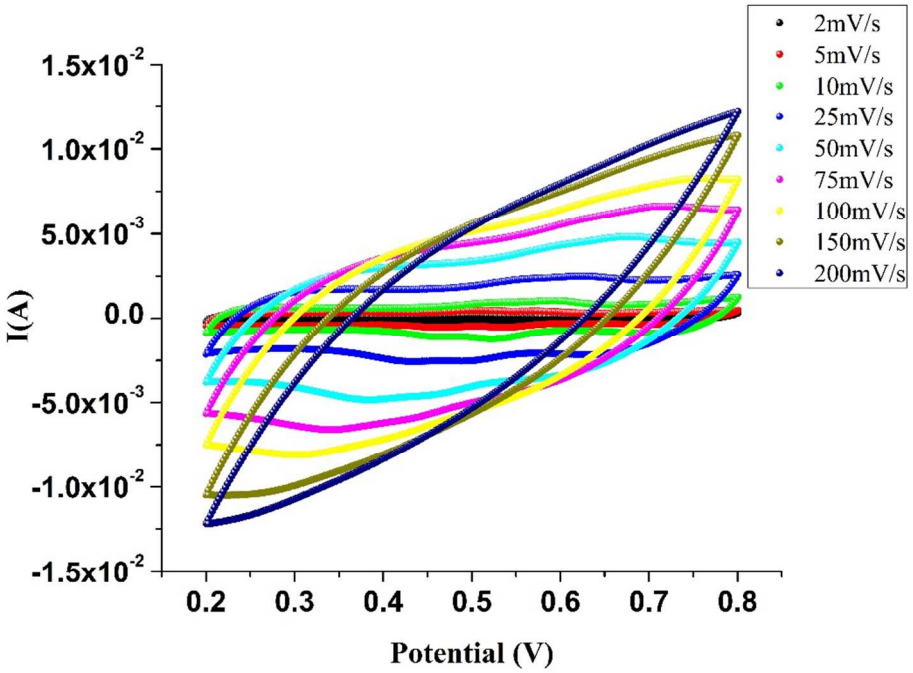

Figure 3. CVs of PANI/rGO/AuNPs electrode at different scan rates in 1 M H_2SO_4 in the potential window of 0.2 – 0.8 V.

Good rectangular shape of PANI/rGO/AuNPs electrode CVs, remains constant at the scan rate of 100 mV/s. The deviation from rectangularity of CVs becomes obvious as scan rate increases. This phenomenon can be attributed to the electrolyte and film resistance and distortion is dependent on scan rate. By increasing the sweep rate, active sites will not have enough time for reaction on the surface of the electrode.Figure 4, shows the calculated specific capacitance of PANI/ rGO /AuNPs electrode as a function of scan rate. PANI/ rGO/AuNPs composite electrode shows specific capacitances of 340 and 120 F/g at the scan rate of 2 and 200 mV/s, respectively, whereas specific capacitances of the PANI electrode decreases from 190 to 60 F/g at the scan rate of 2 and 100 mV/s, respectively. As observed, the capacitance of two electrodes decays over the entire range of scan rate, because in fast sweep rates just outer porosity are use and deeper those are not accessible for dope/ undope process. As can be seen in high scan rates the composite electrode losses it's capacitance with a reduction pattern same as PANI electrode that shows rGO/AuNPs doesn't block the porosities of polymer network. If this phenomena occurred, the reduction of specific capacitance in composite electrode must be more than PANI one. Chemical methods for synthesis of composite could block some of porosities of polymer film and this would decay the supercapacitive behavior of PANI. Therefore, by using electrochemical method, the pseudo capacitive behavior of PANI electrode could be completely sustained.

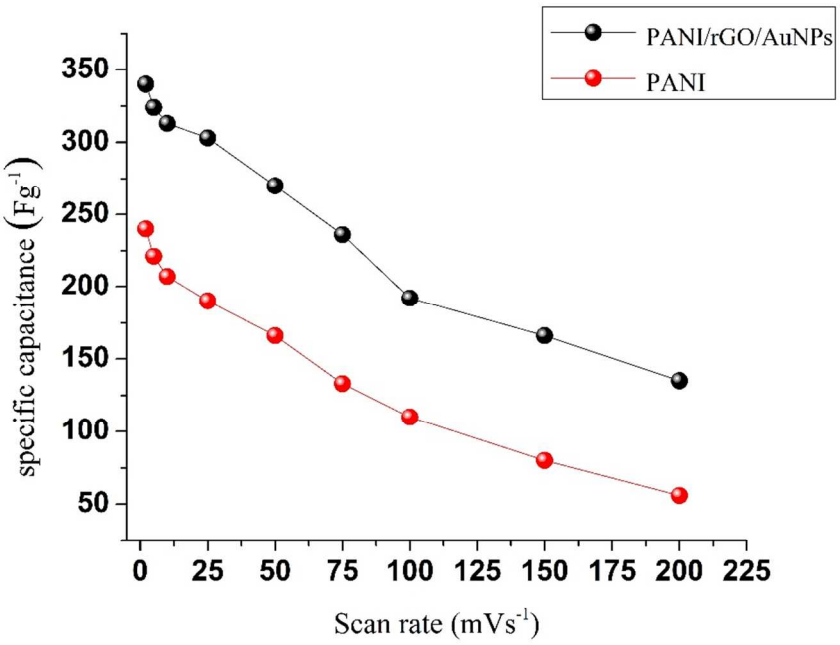

Figure 4.Variations of the specific capacitance for PANI/rGO/AuNPs electrode as a function of the scan rate in 1M H_2SO_4 solution.

3.2 Galvanostatic studies

Galvanostaticcharge/discharge method has been used to highlight the capacitance characteristic of PANI/rGO/AuNPs composite electrode. Figure 5, shows the charge/discharge behavior of PANI and PANI/rGO/AuNPs electrodes in the potential range from 0.2 to 0.8 V at the current density of 2.0 A/g (inset fig).As can be seen, a triangular shape between this potential ranges is observed that indicating good columbic efficiency and ideal capacitive behavior of PANI/rGO/AuNPs as electrode for application in supercapacitors. Furthermore, using rGO /AuNPs caused the voltage drop (iR) decrease from 0.06V for PANI to 0.03 V for PANI/rGO/AuNPs composite electrode. This is related to the internal resistance and appears in the curves during the change of current sign and vice versa. Figure 5 (main figure), presents the charge−discharge curves of PANI/rGO/AuNPs electrode at various specific currents of 0.8, 1.2, 1.4, 1.6, 2.0, 2.4 and 2.8 A/g.Here, specific capacitance has been measured according to the charge/discharge curves, using Eq. (2).

$$Specific\ Capacitance = \frac{i}{\left(-\frac{\Delta E}{\Delta t}\right)m} \qquad (2)$$

In this equation i is the applied current; (-ΔE/Δt) is the slope of the discharge curve after the voltage drop at the beginning of each discharge (ESR); and *m* is the mass of reactive material.

The highest SC for the composite electrode was obtained when the current density for the charge/discharge process was0.8 A/g.specific capacitances were calculated 390, 310, 240, 190, 160 and 145 F/grespectively, for 0.8, 1.2, 1.6, 2.0, 2.4 and 2.8 A/g currents respectively. As illustrated, by enhancing the specific current, specific capacitance values decrease due to intercalation of ions at the surface of the active materials in the electrode/electrolyte interface. On the other hand, when low specific current is used, the specific capacitance increases because there is enough time for insertion and deinsertion of the ions at the surface and deeper porosities of the active materials in the electrode/electrolyte interface. This phenomena can concluded from this data that in low density currents the voltage range in charge−discharge curves decreased.

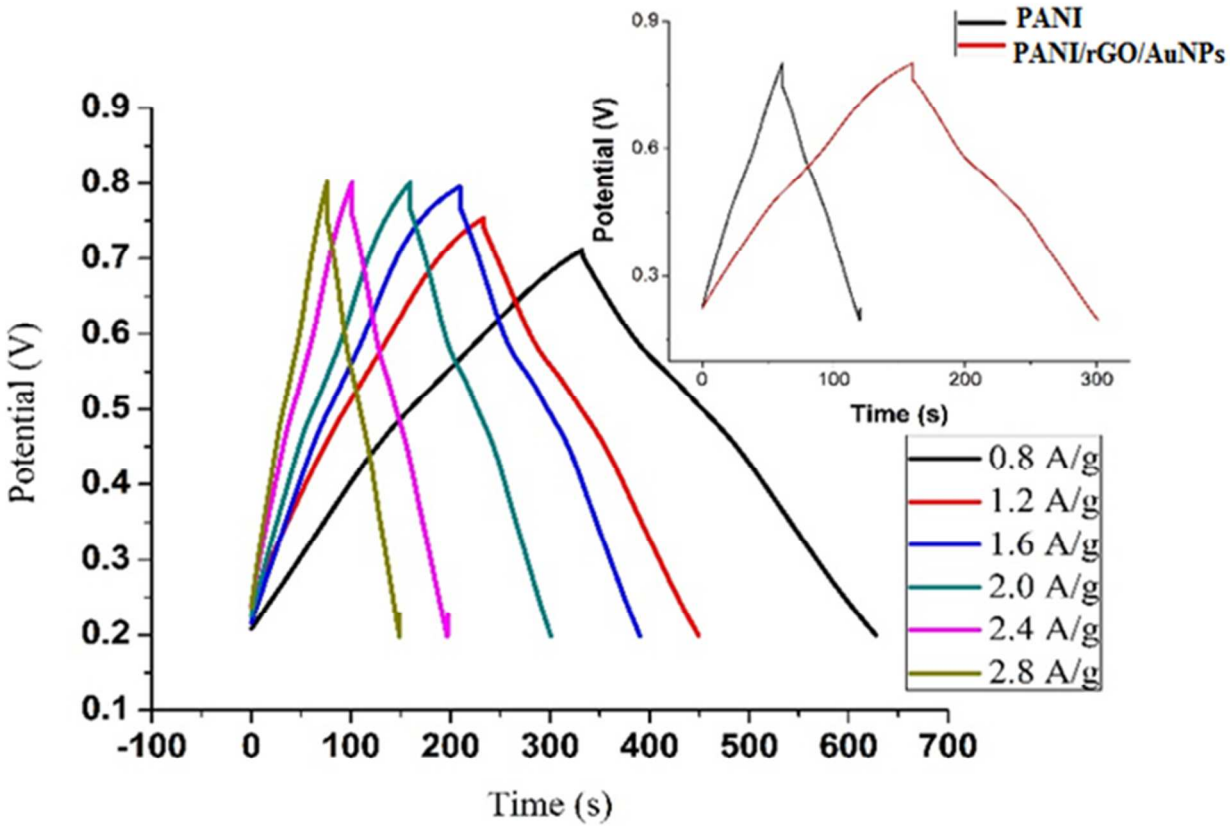

Figure 5. Galvanostatic charge and discharge measurements of PANI and PANI/ rGO /AuNPs electrode at the current density of 2A/g (inset figure) charge−discharge curves of PANI/rGO/AuNPs electrode at various specific currents of 0.8, 1.2, 1.4, 1.6, 2.0, 2.4 and 2.8 A/g (main figure)in 1M H_2SO_4 solution

3.3 Impedance spectroscopy studies

EIS is an appropriate technique to investigate the electrochemical behaviors of various electrode type[35-40]. Figure 6, shows Nyquist plots of PANI and PANI/rGO/AuNPs electrodes at open circuit potential. Intercept of the Nyquist Plots is related to the equivalent series resistance arising from contributions of electronic and ionic resistances. As can be seen, both plots havea semicircle in high frequencies which is related to the charge transfer resistance (R_{ct}) caused by the Faradic reactions and the double-layer capacitance (C_{dl}) at the contact interface between electrode and electrolyte solution. A resistance with the slope of the 45° that appearedin the low frequency region, both electrodes exhibit a nearly linear branch, indicating a decreased diffusion resistance of the electrolyte ions in the electrode, as expected for a capacitor. The low frequency capacitance (C_{lf}) of each film was determined using Eq. (3) in that (Z") is the imaginary component of impedance at low frequency and (f) is the lowest magnitude of frequency.

$$C_{if} = (2\pi f Z'')^{-1} \quad (3)$$

It can be seen that PANI/ rGO /AuNPs electrode has more capacitance than PANI electrode. The SC for PANI and PANI/ rGO /AuNPs electrode were calculated 180 and 310 F/g respectively. These results also confirmed the data obtained by CV and charge-discharge methods.

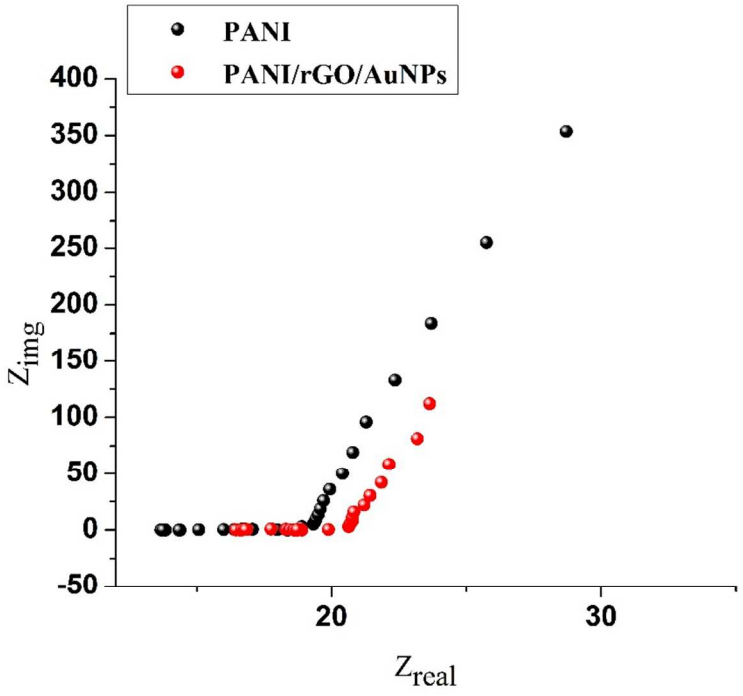

Figure 6. Nyquist plots recorded from 10 kHzto 0.01 Hz with an ac amplitude of 5 mV for PANI and PANI/rGO/AuNPs electrode.

3.4 FFTCCV studies

For examination the changes in the CVs and charge storage of a capacitor during the time, FFTCCV technique could be considered as the best tool[15]. By using this technique one can study the behavior of electrochemical system momentarily. Three-dimensional (3D) CV of PANI/rGO/AuNPs electrode at the scan rate of 50 mV/s was shown in Figure 7a. As can be seen ternary composite electrode losses its symmetrical shape and therefore its capacitance. Figure 7b. illustrated the diffrentiative CVs of composite electrode at 50 mV/s that obtained by FFTCCV technique. In fact, the details of the occurring changes can even be seen well, when the current at recorded CVs subtracted from the current at reference voltammogram, where the reference voltammogram is obtained by averaging 5 voltammograms in the beginning of the measurement. The absolute capacitive charge changes (ΔQ_n) calculation for this operation is as follows equation:

$$\Delta Q_n = \int_{E_1}^{E_2} \Delta i_{(n,E)} dE - ave \left[\int_{E_1}^{E_2} \Delta i_{(m,E)} dE\right] \text{For n>0} \quad (4)$$

Or

$$\Delta Q_n = Q_n - Q_{ave} \quad (5)$$

Where Q_{ave} and Q_n are the calculated average charges at the selected potential range, E_1 to E_2, from m cyclic voltammograms and the calculated charge at the same potential range from subsequent nth cyclic voltammogram, respectively. The computer program algorithm for calculating the detector response was:

$$\Delta Q(n\tau) = \frac{\Delta E}{v}\left(\sum_{E=E1}^{E=E2} i_{(n,E)} - \sum_{E=E1}^{E=E2} i_{(n_r,E}\right) \quad (6)$$

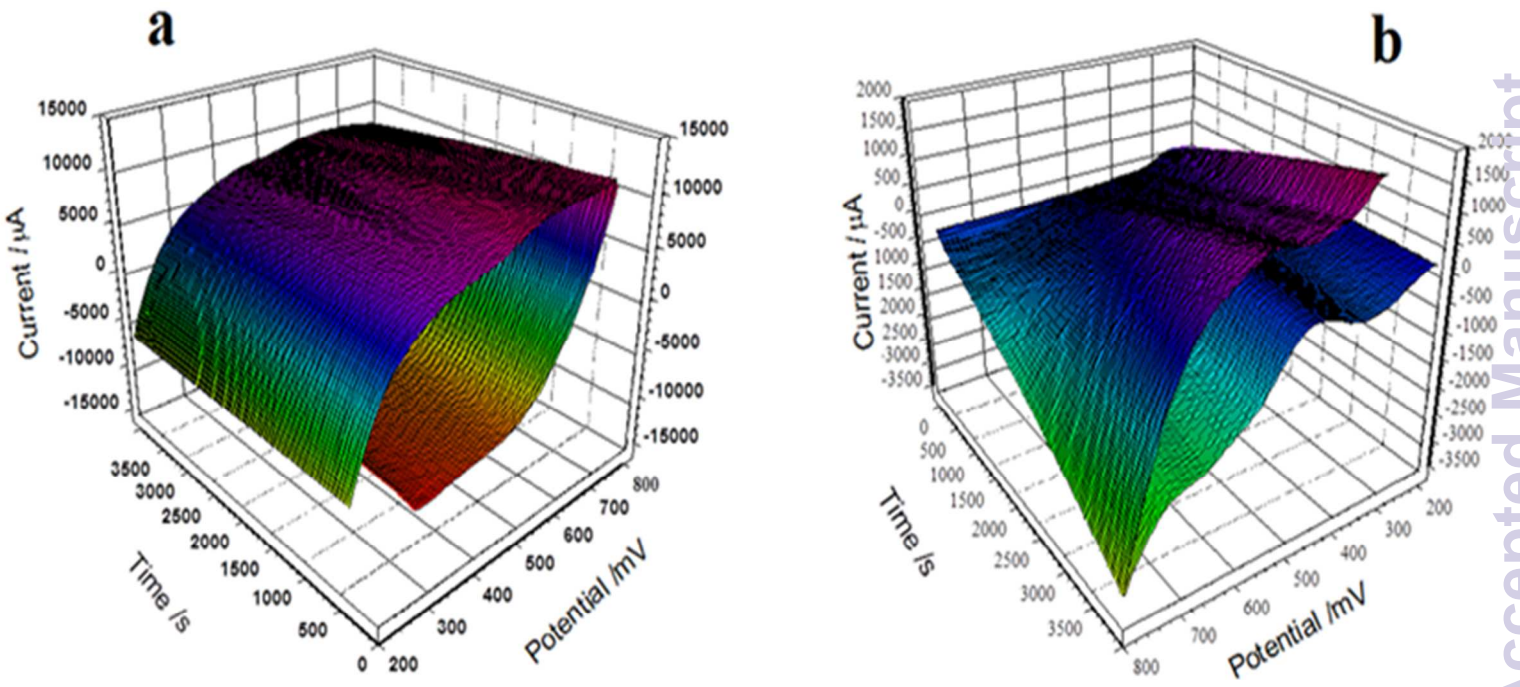

Figure 7. 3D CVs of PANI/rGO/AuNPs electrode as function of Time measured at 50 mV/s (a), diffrentiative cyclic voltammograms of PANI/rGO/AuNPs measured at 50 mV/s.

In this equation, t is the time period between subsequent scans, v is the scan rate, ΔE is the potential difference between two subsequent points on the cyclic voltammogram,$i_{(n,\,E)}$ represents at the recorded cyclic voltammogram during the n^{th} scan and $i_{(nr,\,E)}$ is the reference current at stored cyclic voltammogram in the computer memory. The reference cyclic voltammogram was obtained by averaging a few recorded cyclic voltammograms at the beginning experiment. As can be seen in the Figure 7b there are two peak in the potential range of 0.4 to 0.6V, which related to the oxidation of PANI. As shown in this figure there is a deceleration of current in the high positive potential range. This deceleration shows that the changes in oxidative state are more than reductive one. Figure 8, shows the electrochemical stability of PANI and PANI/rGO/AuNPs electrodes that examined in 1M H_2SO_4 solution by FFTCCV technique after continuous cycles for 20000 seconds with the scan rate of 50 mV/s.

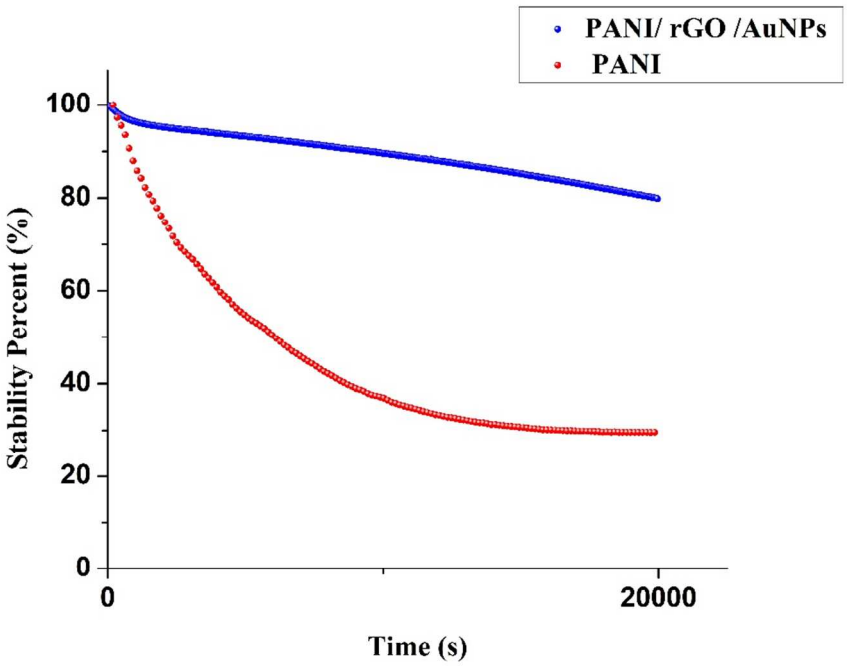

Figure 8.stability of two electrode after consequtive cycles at 50mV/s for 20000 s.

After sequence cycles because of degradation of conductive polymers, the stability of polymer electrode decreased. As can be seen PANI/rGO/AuNPs composite electrode has more stability than PANI electrode, whereas after 20000 seconds PANI electrode just has 30% of its capacitance related to first cycle, the PANI/rGO/AuNPs composite electrode loss just 20% of its capacitance and preserved stable. Using FFTCCV technique one can following the change of CVs, cycle by cycle for study the capacitive behavior of composite electrode. This data processing operation was carried out simultaneously with data acquisition during experiments.

4 Conclusion

Polyaniline/reduced graphene/Au nanoparticles composite electrode was synthesized by electrochemical method. Results showed that reduced graphene oxide and Au nanoparticles have a good effect on the pseudo capacitive behavior of polyaniline electrode. The specific capacitance of composite electrode is much higher than polyaniline electrode. Using Au nanoparticles in composite electrode resulted to increase the conductivity of composite electrode, which appears in electrochemicalimpedance experiments. Furthermore,fast fourier transform continues cyclic voltammetrytechnique is considered as the powerful technique in characterization of new composite materials as supercapacitor. By using this technique, electrochemical behavior of the system is monitored momentarily. The absolute capacitive charge changes calculated from three-dimensional cyclic voltammogram of composite electrode at the scan rate of 50 mV/s and computer program algorithm.Polyaniline/reduced graphene/Au nanoparticlescomposite electrode exhibited more highly stable capacitance retention during charge/discharge cycling and is therefore a promising candidate for long-term applications in high-performance supercapacitors.

Acknowledgements

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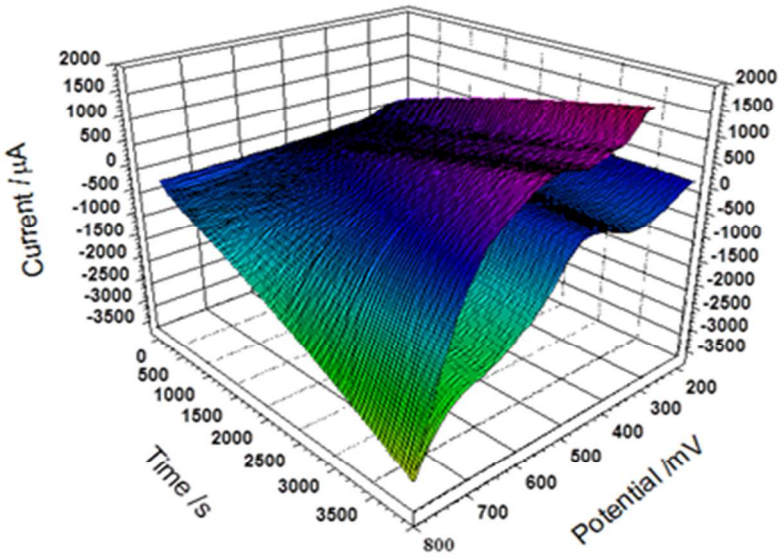

Fast fourier transform continues cyclic voltammetry as a new technique for explanation of composite electrode for supercapacitors.