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Investigating the Effect of Hard Template Synthesis Method and Diameter Uniformity of Nanocomposite Nanowires based on Polyaniline/Silver Nanoparticles on Electrical and Electrochemical Properties

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ABSTRACT

This research investigated the electrochemical properties of nanocomposite nanowires consisting of polyaniline and silver nanoparticles synthesized by the hard template method. The constant and uniform diameter of the nanowires in this method results in better conditions for the use of this nanocomposite. Polyaniline/silver nanoparticles nanocomposite was prepared by the hard template method. The structural characteristics of the prepared sample were examined by scanning electron microscope and elemental analysis and it was proved that the resulting nanocomposite has a nanowire structure with a size of 80-100 nm. The absorption behavior of this nanocomposite was revealed by ultraviolet-visible spectroscopy to be formed at wavelengths of 320 and 610 nm. Electrochemical experiments were performed to evaluate the conductivity, charge storage capacity and electrochemical stability. The results showed that this nanocomposite exhibits good electrical reversibility at a scanning speed of 50 mVs^{-1} and is almost unchanged even up to 100 cycles. The highest electrical conductivity of the nanocomposites under study was discovered to be approximately 55 Scm^{-1}. The electrochemical behavior of this nanocomposite with high charging capability makes it suitable for use in rechargeable batteries. The production sample in this study was able to perform better than previous similar samples.

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Introduction

Biometric recognition or biometric authentication refers to the automatic identification of a person based on anatomical (fingerprint and iris) or behavioral (signature) characteristics [1].

Electrochemical properties are important in many fields including energy storage and conversion, corrosion protection, and other applications [2]. Understanding and using these features has led to significant progress in many industries. In the case of batteries, the electrochemical approach is the study of chemical reactions that result from the transfer of electrons between species and usually occur at the interface between the electrode and the electrolyte [2].

Conductivity is a crucial property of the electrochemical process, which is the ability of a substance to conduct electric current. Materials with high conductivity are desirable for applications with efficient charge transfer coefficients such as batteries, fuel cells, and supercapacitors [3]. In addition, electrochemical characteristics such as charge storage capacity and electrochemical stability are important factors in determining the performance and lifetime of energy storage devices [4].

In the realm of advanced materials, investigating and determining the electrical and electrochemical properties of nanocomposites is considered a promising frontier in research [4]. A correct understanding of the electrical and electrochemical properties of nanocomposites is essential due to their potential applications in advanced technologies because this can revolutionize energy storage, sensor technology and electrochemical devices and open new horizons in this field [5; 6].

Among various materials, conductive polymers have emerged as an interesting group of materials with unique electrochemical properties because their special properties have made them attractive for various applications[6]. Conductive polymers have a higher electrical conductivity than common insulating polymers [6]. The unique structure and active oxidation/reduction pair of these polymers allow them to store and release charge efficiently by reversibly doping charge carriers [7]. Reversible doping refers to the process of intentionally introducing or removing charge carriers (such as electrons or ions) in a substance, which can be reversed. In the field of polymers, reversible doping allows for the storage and release of charge, which enables the material to act as a rechargeable energy storage system, such as a battery. This process can be repeated multiple times without any significant decrease in performance or degradation [7].

To study and describe the electrochemical properties of conductive polymers, various techniques such as cyclic voltammetry and electrochemical impedance spectroscopy are used to investigate the oxidation and reduction behavior, charge

storage capacity, and charge transfer kinetics of conductive polymers [8; 9]. These approaches provide valuable information on the behavior and electrochemical performance of these materials.

Among the existing conductive polymers, polyaniline has attracted much attention due to its properties such as ease of synthesis, controllable electrical conductivity, simplicity in doping, chemical stability, good environmental stability, and mechanical flexibility [9]. One of the challenging issues in the development of supercapacitors and high-performance electrical devices is to improve the electronic conductivity of the polyaniline electrode through charge transfer doping and protonation [10]. Extensive research has focused on increasing the electronic conductivity of electrodes using metal doping [10-12], and silver nanoparticles are one of these doping agents considered due to their unique optical, electrical, electrochemical and catalytic properties [13]. According to scientific research in the field of nanocomposites, a nanocomposite composed of polyaniline and silver nanoparticles has many applications in various fields [13]. A research team developed an electrode made from polyaniline/silver nanoparticles for use in supercapacitors [14]. The results of the investigations of these researchers showed that for the polyaniline film with a concentration of 0.9% by weight of silver, the specific capacity is 512 Fg^{-1} at the scanning speed of 5 mVs^{-1} and the energy density is 50.01 Wh kg^{-1} in 1 mA cm^{-2} [14]. In another study, researchers prepared a three-component composite based on carbon nanotubes, polyaniline, and sulfonated graphene nanosheets. According to the cyclic voltammetry results, the introduction of sulfonated graphene nanosheets led to a 37% increase in electrochemical capacity. It was reported that the capacity of the polyaniline/carbon nanotube nanocomposite increased from 305 F/g to 419 F/g in the sulfonated polyaniline/carbon nanotube/nanographene nanocomposite [15]. The three-component composite was scanned using electrochemical impedance spectroscopy at a speed of 5 mV/s. The results showed that the energy density is higher than 209 Wh/kg and the power density is approximately 381 W/kg [15]. Another group of researchers developed an easy and stable approach to fabricate low-cost, highly flexible and high-performance supercapacitors based on electrodes coated with polyaniline nanowires and silver nanoparticles [16]. The electrical conductivity in this research was reported as 211 Scm^{-1}. In addition, the fabricated supercapacitor showed a specific capacity of 613 Fg^{-1}, an electron density of 85.13 Wh kg^{-1} and a cycle stability of 90% [16].

Based on a review of sources, this research aimed to investigate and determine the electrical and electrochemical properties of polyaniline/silver nanocomposite nanorods. The innovation of this research is the use of polyaniline/silver nanoparticles nanocomposite nanorods through the hard pattern method, which

results in nanowires with a fixed diameter range. Unlike previous research that investigated different structures, the present research specifically focuses on the electrochemical properties of this nanowire morphology synthesized using the hard template approach. It is important to note that prior to this research, no previous studies had examined the electrochemical properties of this particular morphology. By exploring this domain, the objective is to uncover the distinctive properties and potential applications of these nanowires.

Methodology

Materials

In this research, silver nitrate (Merck, Germany) was used for the synthesis of silver nanoparticles, aniline monomer, hydrochloric acid, and ammonium persulfate; and an AAO template (Merck, Germany) was used for the synthesis of poly-aniline. Acetonitrile and lithium perchlorate (Floka company) were used to prepare the electrolyte and acetone (Merck company) was used to wash the substrate. 5% phosphoric acid (Merck) was used as a solvent to separate polyaniline from the membrane, and N-methylpyrrolidone (Merck) was used as a solvent.

To investigate the electrochemical behavior of the prepared nanocomposite, cyclic voltammograms were drawn in a digital potentiostatic device manufactured by Sama Iran-Isfahan. For performing cyclic voltammetry, the following electrodes were utilized: a GC electrode as the working electrode, an Au electrode as the auxiliary electrode, and an Ag/AgCl electrode as the control electrode. The nanocomposite structure was characterized using a TESCAN MIRA3 scanning electron microscope. To measure electrical conductivity using an ohmmeter, the following equipment was employed: a Megatek-30V 12V switching power source, an analogue and digital multimeter (Modern Digital Multimeter GDM-356/451), a digital oscilloscope (Digital Storage Oscilloscope GDS-1000AUSeries), and a signal generator (GDS-3000 Series). The rotary coating machine was employed as a conventional method for generating thin polymer films on the substrate.

Preparation of polyaniline/silver nanoparticles nanocomposite by hard template method

Initially, a solution of 0.015 M silver nitrate was prepared by combining 0.5 M aniline in hydrochloric acid (1 M). The resulting solution was then stirred for 30 minutes. Subsequently, a solution of 0.25 M ammonium persulfate was prepared in 1 M hydrochloric acid. An AAO mould with a hole diameter of 100 nm and an oxide layer thickness of 100 μm was positioned in the center between two reactors. Subsequently, 10 ml of a preprepared solution of ammonium persulfate was

introduced on one side, while 10 ml of a silver nitrate solution, together with aniline, was added to the opposite side of the membrane. After 2 hours, the AAO membrane was removed and placed in 5% phosphoric acid for 24 hours to dissolve the membrane and settle the nanopolymers [17].

Subsequently, the plastic component of the membrane was extracted from the solution, and the resultant mixture was subjected to a 15-minute centrifugation with distilled water to lower the pH to 7. Finally, the obtained nanocomposite was dried in an oven at 60°C for 24 hours. Figure 1 shows the preparation steps of the resulting nanocomposite [17].

Determining the electrochemical behavior of polyaniline/silver nanoparticles nanocomposite using cyclic voltammetry method

Oxidation was carried out in a single cell equipped with an electrode (GC) and gold and Ag/AgCl electrodes as working, auxiliary and control electrodes, respectively. The electrolyte environment comprised 20 ml of acetonitrile, with sodium perchlorate serving as the solvent and carrier electrolyte at concentrations of 0.1 M and 0.01 M, respectively. Initially, the solution was agitated and purged of oxygen using a flow of Ar gas for 10 minutes. Then, by stabilizing the potential between two ranges relative to the Ag/AgCl electrode in accordance with the oxidation and reduction of the polymer, its scanning occurred between 10-100 times on the surface of the electrode [18]. Subsequently, the electrode, coated with a polymer, was once more subjected to potential scanning within two distinct ranges. The difference in applied potentials (-1800, -1700) at the electrode's surface was varied, and this process was repeated at various scanning speeds. Eventually, the mentioned voltammograms were generated by the device. To achieve this, the resulting nanocomposite was suspended in N-methylpyrrolidone solvent, and then it was deposited onto the GC electrode. After a waiting period of 2 days, the intended solvent was allowed to evaporate, leaving the nanocomposite adhered to the electrode. Cyclic voltammetry experiments involved a three-electrode system, utilizing a glassy carbon electrode coated with a polyaniline nanocomposite containing silver nanoparticles as the working electrode, a gold electrode as the auxiliary electrode, and a saturated Ag/AgCl electrode as the reference electrode.

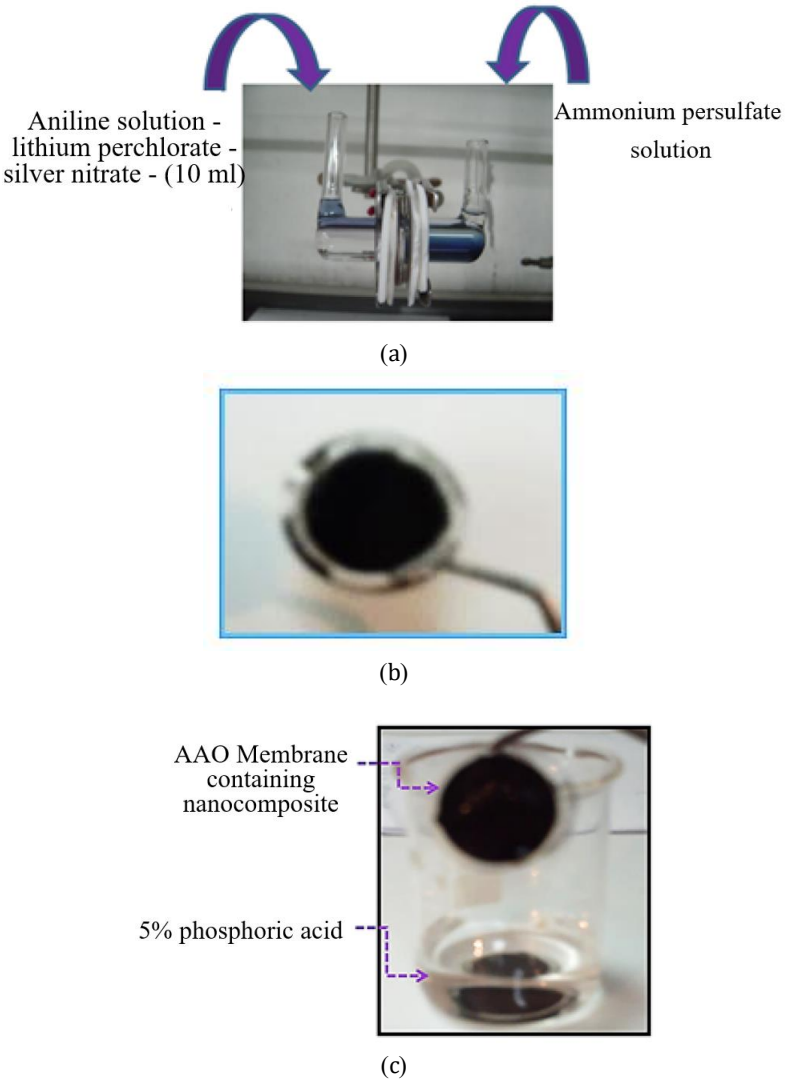


Figure 1. Summary of the preparation steps of polyaniline/silver nanoparticles nanocomposite by hard template method. a) reaction vessel for the synthesis of nanowires. b) polyaniline/silver nanoparticles nanocomposite on AAO membrane. c) dissolving the membrane in phosphoric acid 5 %.

Results and discussion

The results of microscopic analysis

Figure 2 displays images resulting from the electron microscope analysis of the polyaniline/silver nanoparticles nanocomposite. The images clearly reveal the presence of a nanorod structure in the resulting nanocomposite. Notably, the

polyaniline surface exhibits a porous and non-uniform nature, featuring nanorods with a diameter of approximately 100 nm, while the silver nanoparticles have a size ranging from 80 to 100 nm. The diminutive size of the particles enhances the surface-to-volume ratio and positively impacts multiple parameters. Additionally, the findings from the elemental analysis reveal a silver content of 1.21 per cent by weight, whereas it was initially assumed to be 0.02 per cent during the synthesis process.

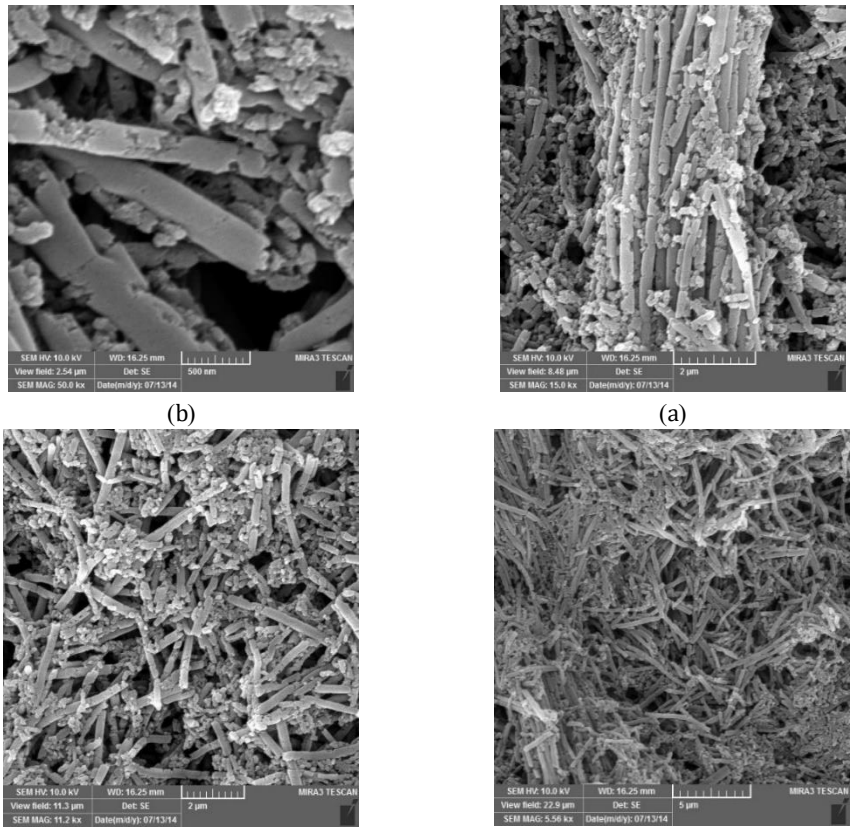


Figure 2. Scanning electron microscope micrographs of polyaniline/silver nanoparticles nanocomposite with nanowire structure in different magnifications.

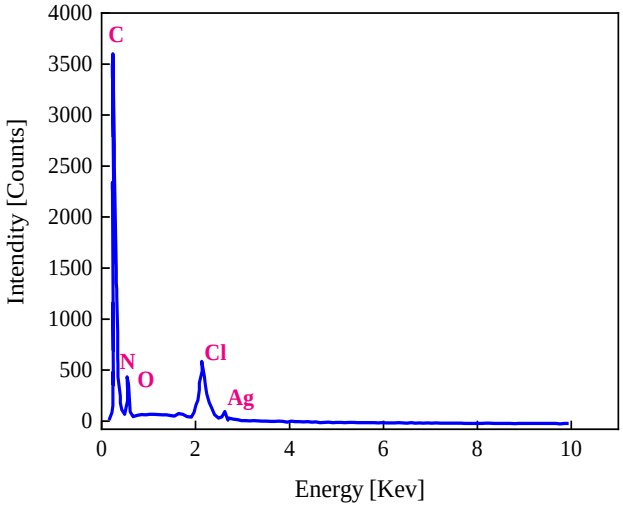


Figure 3. EDX spectrum of polyaniline/silver nanoparticles nanocomposite structure.

Table 1. Weight percentage of elements in polyaniline/silver nanoparticles nanocomposite obtained from EDX spectroscopy.

ELt	Line	Int	Error	K	Kr	W%	A%	ZAF	Qx%	Pk/Bg
C	Ka	1565.3	18.9961	0.8228	0.4788	62.04	67.93	0.7717	0.00	200.21
N	Ka	105.9	18.9961	0.0788	0.0458	22.63	21.25	0.2025	0.00	40.01
O	Ka	155.7	18.9961	0.0559	0.0325	12.06	9.91	0.2697	0.00	31.94
Cl	Ka	77.2	5.2991	0.0288	0.0168	2.06	0.76	0.8145	0.00	5.37
Ag	La	15.4	5.2991	0.0137	0.0080	1.21	0.15	0.6553	0.00	2.55

Analyzing the absorption spectrum

In Figure 4, the absorption spectrum of the polyaniline/silver nanoparticles nanocomposite is shown in the range of 200-1200 nm. In this illustration, the presence of a peak at approximately 320 nm and another peak at around 610 nm can be observed. The absorption peak at 320 nm frequently signifies $\pi \rightarrow \pi^*$ transitions within the benzoid structure, while the absorption at 610 nm is linked to the creation of an exciton in the ring [19].

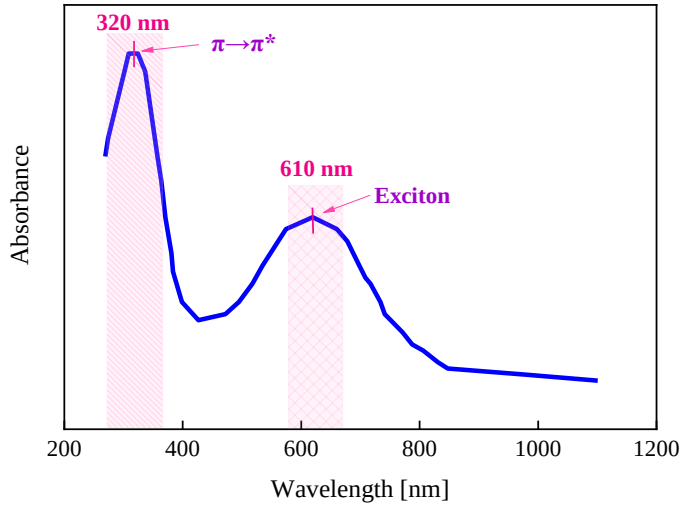


Figure 4. Absorption spectrum diagram of polyaniline/silver nanoparticles nanocomposite.

Investigating the electrical conductivity of polyaniline/silver nanoparticles nanocomposite with nanorod structure

Electrical conductivity is one of the most important parameters for evaluating conductive polymers. This parameter depends on the concentration of charge carriers and their mobility. In conductive polymers, the concentration of these carriers depends on the level of oxidation or doping. Therefore, an enhancement in molecular arrangement, contingent on the specific conductive polymer manufacturing technique employed, will result in an elevation of carrier mobility and, consequently, an augmentation in electrical conductivity [20].

To assess the electrical conductivity of the polyaniline/silver nanoparticles nanocomposite, a two-point and four-point probe device was employed. This allowed for the examination of its electrical conductance. The results showed that the highest electrical conductivity obtained for this nanocomposite after several tests were with the four-point device at 55 Scm^{-1} and with the two-point device at 0.44 Scm^{-1}.

Investigating the electrochemical behavior of polyaniline/silver nanoparticles nanocomposite using cyclic voltammogram

In the cyclic oxidation/reduction process, the oxidation and reduction behavior of different compounds on the electrode can be evaluated. In the mentioned nanocomposite, the oxidation occurred within a potential range of -1500 to -1700 mV when compared to the Ag/AgCl electrode. This was observed at various scanning speeds ranging from 25 mV/s to 200 mV/s in the previously described

cells. The electrolyte medium used consisted of an acetonitrile solvent and a 0.5 M lithium perchlorate.

Figure 5 illustrates the cyclic voltammograms of the prepared nanocomposite. Within the peak corresponding to a scanning speed of 25 mV/s, the reduction peak is evident within the range of -0.4. However, the oxidation peak is not clearly visible due to the low scanning speed. At scan speeds of 75 and 50 mV/s, it can be observed that the peaks related to oxidation are slightly visible. The peak at the end of the graph is the peak of the wall. One peak is observed in the range of 1.4 and another peak in the range of -0.4. In the voltammograms displayed at various scanning speeds, it is evident that during the initial scan, an anodic peak is observed around 300 mV concerning the Ag/AgCl reference electrode. During the reverse scan, which involves transitioning from the maximum potential of 1700 mV to -1500 mV, a reversible reaction is observed to take place. In the second and following scans, a secondary peak emerges at approximately 1300 mV, and its intensity progressively rises from one scan to the next. This phenomenon is associated with the oxidation of the polymer occurring on the electrode's surface. In addition, the amplitude of the peaks is associated with the increase of the current peak due to the increase in electron yield and scanning speed, which is evident in the diagrams.

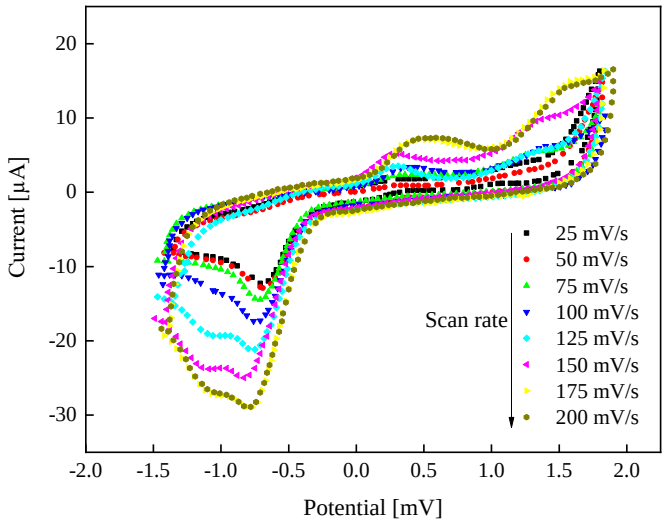


Figure 5. Cyclic voltammogram of polyaniline/silver nanoparticles nanocomposite with nanowire structure in the potential range of -1500-1700 mV compared to Ag/AgCl in 0.1 M solution of acetonitrile/lithium perchlorate with different scanning speeds mV/s 200, 175, 150, 125, 100, 75, 50, 25.

Furthermore, the cathodic peak currents have an almost linear behavior with respect to different scanning speeds, and the polymer film on the working electrode is stable during the oxidation and reduction scanning.

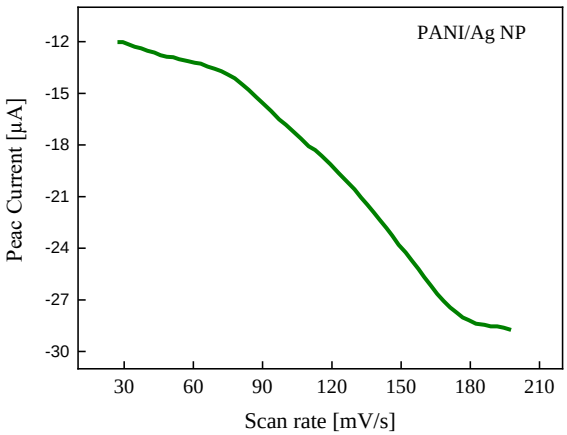


Figure 6. Diagram of anodic and cathodic flow in relation to scanning speed of polyaniline/silver nanoparticles nanocomposite.

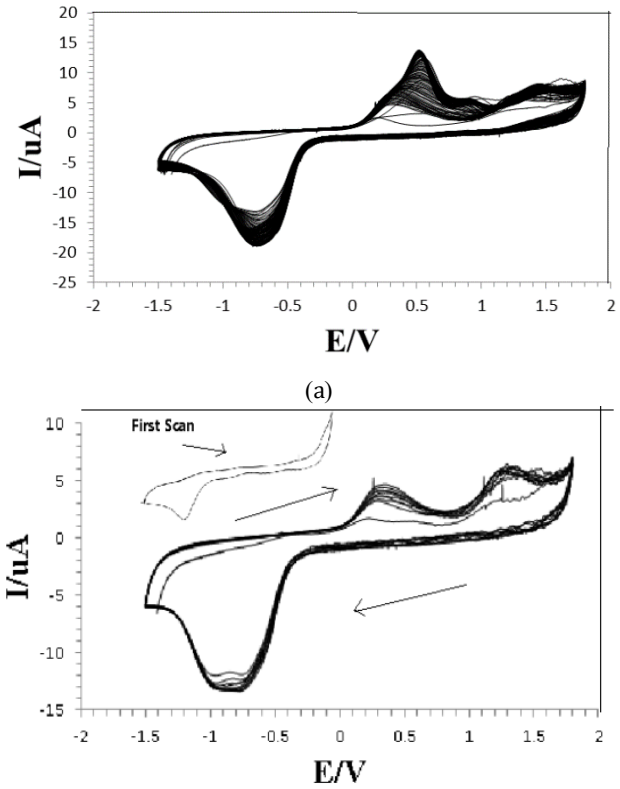
The charging capability

The area under the CV diagram can be attributed to the charge capacity function related to the electrochemical activity of nanocomposites. One of the key applications involves employing nanocomposites in polymer batteries. Consequently, by utilizing the cyclic voltammogram tool, it becomes feasible to conduct a comparative assessment of the utilization of these substances in battery structures. Therefore, the cyclic voltammogram of the prepared nanocomposite was performed up to 100 cycles. In the process of oxidation/reduction on this nanocomposite during 100 cycles and 10 cycles, no significant changes were observed in the oxidation/reduction potential and the electrochemical behavior of this nanocomposite does not change. Hence, it can be inferred that the fabricated nanocomposite has demonstrated excellent performance in both electron extraction (discharge) and electron donation (charge) processes, indicating a strong charging capability. Finally, this feature confirms the usability of polyaniline/silver nanoparticles nanocomposite with nanorod structure in rechargeable batteries.

In general, combining polyaniline and silver nanoparticles in nanocomposite offers several advantages. Silver nanoparticles possess outstanding electrical conductivity and catalytic characteristics, contributing to the enhancement of the nanocomposite's overall electrical conductivity and electrochemical performance.

Furthermore, the ample surface area of silver nanoparticles facilitates electrochemical reactions, resulting in improved charge transfer kinetics [21].

In this review, the focus is on the nanorod structure of metal nanoparticles/polyaniline nanocomposite for energy storage applications. Due to the high percentage of atoms on the surface of metal nanoparticles and stabilization by the final polymer, these nanocomposites have been considered in catalytic applications, showing the combined properties of polymer and metal clusters. Exceptional electrical conductivity and a substantial surface-to-volume ratio enable their utilization in diverse applications. Additionally, employing rod-shaped nanostructures via the challenging pattern synthesis technique, which ensures compound diameter uniformity, enhances the performance of this nanocomposite in comparison to similar samples [21].



(a)

(b)

Figure 7. Cyclic voltammogram of polyaniline/silver nanoparticle nanocomposite with nanowire structure in the range of 1700-1500 mV compared to Ag/AgCl in 0.1 M solution of acetonitrile/lithium perchlorate with different scanning speed of 50 mV/s.) during 10 cycles b) during 100 cycles.

Figure 8 illustrates the cyclic voltammogram of the prepared nanocomposite in the range of -1700-1800 mV compared to Ag/AgCl in 0.1 M acetonitrile/lithium perchlorate solution with different scanning speeds.

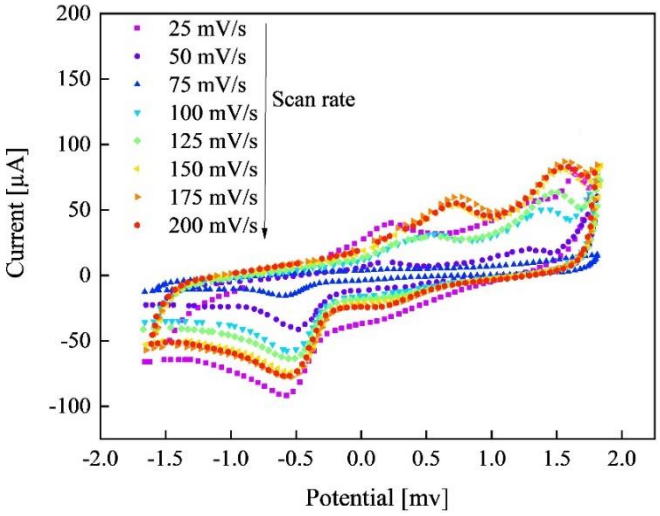
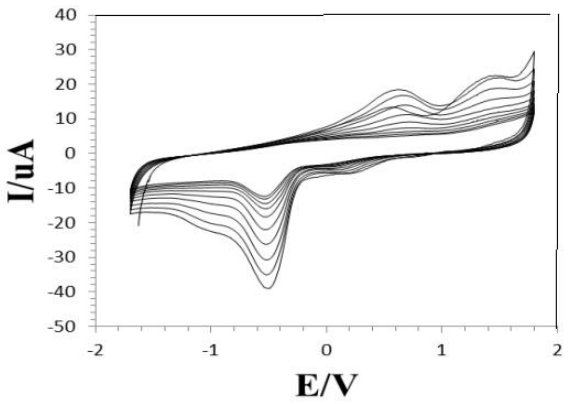
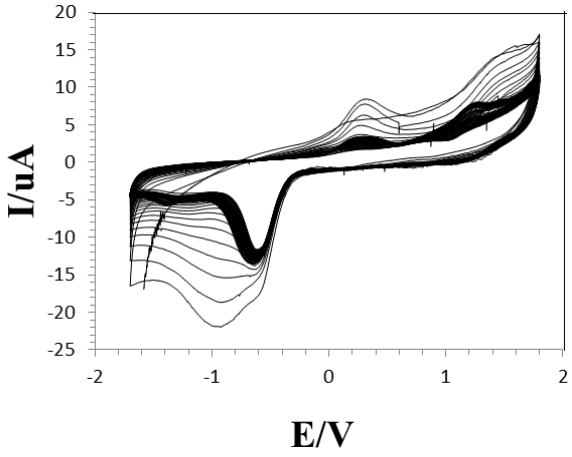


Figure 8. Cyclic voltammogram of polyaniline/silver nanoparticles nanocomposite with nanowire structure in the potential range of -1700-1800 mV compared to Ag/AgCl in 0.1 M solution of acetonitrile/lithium perchlorate with different scanning speeds of 25, 50, 75, 100, 125, 150, 175, 200 mV/s.



(a)



(b)

Figure 9. Cyclic voltammogram of polyaniline/silver nanoparticles nanocomposite with nanowire structure in the potential range of -1700-1800 mV compared to Ag/AgCl in 0.1 M solution of acetonitrile/lithium perchlorate with different scan rates of 50 mV/s. a) during 10 cycles. b) during 100 cycles

Calculation of HOMO and LUMO energy difference of nanocomposite

Using oxidation/reduction information from the cyclic voltammogram diagrams of nanocomposites, it is possible to calculate the energy levels of HUMO and LUMO and finally achieve the energy difference of these two orbitals, which will be the band gap. Hence, employing the subsequent three equations, the energy gap of the polyaniline/silver nanoparticle nanocomposite featuring a nanowire structure was determined using cyclic voltammetry, yielding an approximate value of 2.7 eV for the band gap. Due to the very important applications for the prepared nanocomposite, the high band gap limited its use in applications such as solar cells.

$$HOMO = -[\varphi^{ox}{}_{onset} + 4.71]\ (eV) \quad (1)$$

$$LUMO = [\varphi^{rea}{}_{onset} + 4.71](eV) \quad (2)$$

$$E^{g}{}_{ec} = (\varphi^{ox}{}_{onset} - \varphi^{rea}{}_{onset})(eV) \quad (3)$$

In Table 2, the results obtained from this research are compared with other similar samples.

Table 2. Comparison of the results with previous similar samples.

Nanocomposite	Morphology	Method	Electrical conductivity (Scm^{-1})	Charging capacity F/g	Reference
Pure polyaniline	Nonowire	Hard Template	20	700	[22]
Polyaniline/SiO_2	Nanofiber	Hard Template	50	417	[23]
Polyaniline/TiO_2	Nanowire	Electrochemical deposition	36.5	732	[24]
Polyaniline/AgNP	Nanowire	Hard Template	55	786	In research

Conclusion

In the present research, polyaniline/silver nanoparticle nanocomposite with nanowire structure was prepared by the hard template method. The electrochemical behavior of the prepared nanocomposite was evaluated by cyclic voltammograms. The results obtained are as follows:

- The maximum value of electrical conductivity of this nanocomposite using the four-point probe method was approximately 55 Scm^{-1}.
- Evaluation of the charge capacity of nanocomposite during 10 and 100 cycles indicated the optimal charging capability of this nanocomposite and the possibility of using it in rechargeable batteries.
- Band gap energy for this nanocomposite was obtained according to the oxidation/reduction potential of HUMO and LUMO energy levels of 2.7 eV.
- This review emphasizes the electrochemical properties of nanorods synthesized using the hard template method. Unlike the previous research that investigated various structures, the current research specifically considers nanocomposite nanorods made of polyaniline and silver nanoparticles. This synthesis approach produces nanowires with a constant and uniform diameter range. Furthermore, via a thorough comparison with similar samples, the present study highlights the superior performance achieved using this synthesis method and investigation of this particular morphology. Moreover, a significant improvement in electrical conductivity and charge capacity was observed in the prepared nanocomposite compared to other samples.

Disclosure statement and funding

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