

Available online at: www.jrrset.com

JIR IF: 2.54 SJIF IF : 4.334 Cosmos: 5.395

Volume 9, Issue 4 – April 2021 - Pages 1-21

POLYMER CARBON NANO COMPOSITES: SYNTHESIS AND **CHARACTERIZATION**

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ABSTRACT

The development and characterization of carbon nanotube (CNT)-based solid polymer electrolytes (SPEs) tailored for various device applications. Through the integration of CNTs into different high molecular weight polymers-Polyvinyl alcohol (PVA) and Polyvinyl pyrrolidone (PVP)-via solution casting technique, we successfully synthesized three novel carbon-based polymer electrolytes: PVP + NH4I+CNT, PVP + NaI + CNT, and PVA + NH4I+ CNT. These SPEs were fabricated into self-standing thin films with varying weight percentages of CNT. Subsequently, comprehensive characterization studies were conducted on these thin films. The investigation encompasses structural, morphological, thermal, and electrochemical analyses to elucidate the properties and performance of the developed SPEs. This research contributes to advancing the understanding and utilization of CNT-based solid polymer electrolytes in diverse device applications.

KEYWORDS

carbon nanotube, solid polymer electrolytes, Polyvinyl alcohol, polymer electrolytes, morphological, Flexural property, Impact property, electrochemical analyses.

Introduction

During last few decades a large number of solids having high ionic conductivity comparable to those of liquid electrolytes (10-1 - 10-5 S cm-1) and nearly zero or negligibly small electronic conductivity have been discovered. Such solids are called as super ionic solids or Fast ion conductors or Solid electrolytes. These are also called as Proton conducting solid electrolytes or Proton conductors. The mobile species in such solids are, H+, OH-, NH4+,



Available online at: www.jrrset.com

ISSN (Post) : 2347-6729 ISSN contant : 2348-3105

JIR IF: 2.54 SJIF IF : 4.334 Cosmos: 5.395

Volume 9, Issue 4 – April 2021 - Pages 1-21

H3O+, H5O2 +, H5N2+ etc. But, in general, their conductivities are found to be very small. Now, the solid electrolytes are broadly classified in to following groups based on their microstructure and physical properties. The word 'Polymer' is derived from the Greek words 'POLY' and 'MEROS' signifying 'many' and 'part' separately. Thus a polymer is a substance which hasmany parts. Some scientists prefer to use the word 'macromolecules' for polymers. Polymers have a marked and very direct practical impact on our everyday life. Actually in the present time we are living in the 'polymer age'. Products made from polymers are all around us. This is why the study 'macromolecules / polymers is one of the most important and rapidly growing branch of science (Singh et al., 2011) In this manner, polymers (or macromolecules) are extensive particles having high molecular weight.

Polymer electrolytes:

Polymer electrolytes are the materials of current enthusiasm because of their worthwhile substance and mechanical properties permitting simple mouldability and manufacture of thin films of desired sizes. The framework adaptability guarantees legitimate terminal electrolyte contacts in various electrochemical gadgets. The overwhelming class of polymer electrolytes involves the normal polar polymers complexed with soluble base metals/divalent change metals/ammonium salts/acids and so on.

The research on polymer electrolytes started with the pioneering work of Wright and his co-workers, who firstly reported the ionic conduction in polymer salt complexed system formed by doping alkali salt in to polyethylene oxide (PEO) polymer matrix in the year 1973 (Fenton et al., 1973; Wright, 1775; Wright et al., 1976) Later, Armand, (Armand et al.,1979; Armand, 1986) explored the potential applications of these materials for device applications. Since then, the subject of polymer electrolytes is being hotly pursued to improve the ionic conductivity of existing polymer electrolytes or to explore new polymer electrolytes with higher ionic conductivity and better mechanical properties being well suited for device applications (Gray, 1991; Abraham et al., 1993). The polymer electrolyte materials can be classified into the following groups.



Available online at: www.jrrset.com

ISSN (Post) : 2347-6729 ISSN contant : 2348-3105

JIR IF: 2.54 SJIF IF : 4.334 Cosmos: 5.395

Volume 9, Issue 4 – April 2021 - Pages 1-21

Basalt Fiber

Basalt fibers are extracted from the igneous rock of lava, which is primarily composed of minerals such as plagioclase, pyroxene, and olivine. They exhibit superior physiomechanical properties compared to other types of synthetic fibers, and are also more costeffective than carbon fibers. The technology behind the production of basalt fibers involves a one-stage process of melting and direct extraction from basalt rocks. At a manufacturing temperature of around 1500, the molten basalt is extruded through small nozzles for continuous fiber extraction. These extruded basalt fibers typically have a diameter range between 10µm to 20µm. Basalt fibers possess a high elastic modulus and are three times stronger than steel.

Thin basalt fibers are utilized as reinforcing materials in the textile industry for producing woven fabrics. Thicker fibers find applications in the manufacturing of large structural products used in aerospace, construction, and shipbuilding industries. Table 2 illustrates the physical properties of basalt fibers [1], while Figure 2 depicts typical images of basalt short fibers, basalt continuous fibers, and mat fibers. Basalt fibers hold significant potential for high-temperature applications and are increasingly being used as a replacement for cement concrete in structural buildings.

Polymer composite

Composites can be characterized as materials that comprise of at least two artificially and physically extraordinary phases isolated by a particular interface. The diverse frameworks are joined sensibly to accomplish a framework with increasingly helpful basic or practical properties non achievable by any of the constituent alone. Composite material made out of at least two unmistakable stages (matrix phase and dispersed phase) have mass properties essentially not quite the same as those of any of the constituents. Matrix phase is the essential phase having a nonstop character. Matrix phase is normally progressively flexible and less hard stage. It holds the dispersed phase and offers a heap with it. Dispersed (strengthening) phase is installed in the lattice in an intermittent shape. This auxiliary stage is known as the dispersed phase. Dispersed stage is normally more grounded than the matrix



Available online at: www.jrrset.com

ISSN (966) : 2347-6729 ISSN (966) : 2348-3105

JIR IF : 2.54 SJIF IF : 4.334 Cosmos: 5.395

Volume 9, Issue 4 – April 2021 - Pages 1-21

phase, along these lines, it is at times called reinforcing phase

Most economically delivered composites utilize a polymer framework material regularly called a resign arrangement. There are a wide range of polymers accessible relying on the beginning crude fixings. PMCs are exceptionally well known because of their minimal effort and basic creation techniques. Utilization of non strengthened polymers as structure materials is constrained by low dimension of their mechanical properties, to be specific quality, modulus, and effect opposition. A critical hypothesis for understanding conductivity inside composite materials, particularly where the polymer network and the filler have altogether different attributes, is the idea of percolation (Roman et al., 1986; Singh et al., 2003; Shahi and Wagner, 1981; Liang,1973). The percolation idea initially managed the spread of theoretical liquid particles through an irregular medium. In a perspective of electrical conduction in a polymer lattice, electrons are allowed to move through conductive filler particles. On the off chance that these filler molecule get in touch with one another, a ceaseless way is framed through the polymer lattice, which is a protecting material, for electrons to go through. This way is known as a conductive system, and the material with the conductive system transforms into a conductive material.

Carbon based polymer electrolyte

On account of their extraordinary structures and numerous valuable properties Carbon nanotubes (CNTs) have pulled in overall intrigue and have turned out to be a standout amongst the most encouraging building-hinders for nanotechnology. As we realized that CNTs are allotropes of carbon with a tube shaped nanostructure. Both the closures or any of the single end of a nanotube might be topped with a half of the globe of the buckyball structure which is additionally an allotrope of carbon. These barrel shaped carbon atoms named CNT have extraordinary novel properties. These epic properties make them possibly significant in various applications in nanotechnology, equipment, optics and diverse fields of materials science, similarly as potential uses inengineering fields.Nanotubes are ordered as single-walled nanotubes (SWNTs) and multi-walled nanotubes (MWNTs) (Harris, 2009)

At the point when an uncommon class of CNT materials are CNT-composites in which the



Available online at: www.jrrset.com

ISSN (7966) : 2347-6729 ISSN (7966) : 2348-3105

JIR IF : 2.54 SJIF IF : 4.334 Cosmos: 5.395

Volume 9, Issue 4 – April 2021 - Pages 1-21

CNTs are installed inside or inside the a host polymer grid. The resultant Polymer composites containing CNTs are of extraordinary intrigue territory in all most all field since they may have a novel blend of electrical, optical and mechanical properties. Polymer grid demonstrates upgraded mechanical properties, warm conductivity and electrical properties when CNT is included into polymer networks, despite the fact that measure of CNTs in these polymer/CNT nanocomposites little when contrasted with host polymer. By and large, customary filled polymers utilize a moderately high substance of great conductive fillers, for example, carbon dark, carbon filaments, graphite, to accomplish a helpful electrical reaction for different responsive applications (Harris,2009;Reich et al.,2004; Awasthi et al.,2008). As of late, polymer/CNT nanocomposites have observed much consideration because of the one of a kind electrical properties of CNTs. Numerous specialists are effectively

Literature Survey

Abrantes et al. [1] contribute to the burgeoning field of solid-state ionics with their investigation into thin film solid-state polymer electrolytes (SPEs) containing silver, copper, and zinc ions as charge carriers. The study builds upon previous research efforts focused on developing novel SPEs for diverse electrochemical applications, leveraging the unique properties of metal ions to enhance the conductivity and functionality of polymer electrolyte systems.

Byrne et al. [2] reported on ceramic nanoparticles, such as oxide and sulfide materials, and their applications in electrochemical devices and energy storage systems. Theoretical frameworks for predicting the influence of nanoparticle size, shape, and surface chemistry on ion transport properties may also inform the development of effective nano-filled polyelectrolyte gels.

Chand et al. [3] investigated the field of materials science with their investigation into the structural and electrical conductivity properties of pure Polyvinyl Alcohol (PVA) and PVA doped with Succinic acid polymer electrolyte systems. This research aligns with the broader interest in developing novel polymer electrolyte materials for various electrochemical applications, including batteries, sensors, and super capacitors.



Available online at: www.jrrset.com

ISSN (Post) : 2347-6729 ISSN contant : 2348-3105

JIR IF: 2.54 SJIF IF : 4.334 Cosmos: 5.395

Volume 9, Issue 4 – April 2021 - Pages 1-21

Devendrappa et al. [4] investigated to the exploration of polymer electrolyte systems by investigating the structural and electrical conductivity properties of pure Polyvinyl Alcohol (PVA) and PVA doped with Succinic acid. This study aligns with the broader research interest in developing polymer electrolytes for various electrochemical applications, including batteries, sensors, and fuel cells.

Ferrari et al. [5] reported the advancement of lithium-ion battery technology with their study on lithium-ion conducting PVdF-HFP composite gel electrolytes based on N-methoxyethyl-N-methylpyrrolidinium bis (trifluoromethanesulfonyl)-imide ionic liquid. This research aligns with the broader interest in developing advanced electrolyte materials for lithium-ion batteries, aiming to improve their safety, stability, and electrochemical performance.

Harris et al. [6] reported that comprehensive exploration of carbon nanotube (CNT) science, covering synthesis methods, fundamental properties, and diverse applications of these nanomaterials. This seminal work builds upon an extensive body of research spanning multiple disciplines, including materials science, nanotechnology, chemistry, physics, and engineering.

Limpan et al. [7] reported the field of food packaging materials with their investigation into the influences of the degree of hydrolysis and molecular weight of poly(vinyl alcohol) (PVA) on the properties of fish myofibrillar protein/PVA blend films. This study aligns with the broader research interest in developing sustainable and biodegradable packaging materials for the food industry, aiming to improve the shelf-life, safety, and quality of food products while minimizing environmental impact.

RESEARCH METHODOLOGY

Primarily focus of this thesis is to study CNT doped polymer electrolyte or polymer- salt-CNT complexed system and studied their properties for various device applications. Four distinct polymer-CNT-salt complexed system dispersion with CN have been developed and characterized for various device applications. Hence this thesis centre on the following four samples of polymer-salt-CNT complexed system.



International Journal on Recent Researches in Science, Engineering & Technology (IJRRSET)

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JIR IF : 2.54 SJIF IF : 4.334 Cosmos: 5.395

Volume 9, Issue 4 – April 2021 - Pages 1-21



Figure 1 Films made with the help of solution cast method (a) PVP: NH4I+0.1wt% CNT, (b) PVP: NH4I+0.2wt%CNT, (c) PVP: NH4I+0.3wt% CNT and (d) PVP: NH4I+ 0.4wt% CNT

CNT synthesis techniques using spray pyrolysis method

In the coarse of Carbon nanotube synthesis, various experimental parameters need to be controlled or optimized during the synthesis process. The main parameters which is needed to be optimized are mainly substrate morphology, synthesis temperature, concentration of precursor, injection rate and time, carrier gas mainly used ammonia, hydrogen, ethylene etc.) flow rate and the grouping of reactant gases in the chamber. (These gases could be smelling salts, hydrogen, water, ethylene, or others.) Fine- tuning of these elements could choose the whole and sorts of CNT advancement similarly as their improvement rate.(Murakami et al.,2003;Dresselhaus et al.,2001;Derycke et al.2001;Bonard et al.,1998). This examination researches the impact of various factors on CNT development by means of shower pyrolysis CVD strategy



Available online at: www.jrrset.com

ISSN (Post) : 2347-6729 ISSN (Oulse) : 2348-3105

JIR IF : 2.54 SJIF IF : 4.334 Cosmos: 5.395

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Figure 2 Spray pyrolysis set-up

EXPERIMENTAL METHODOLOGY

The opening of the inner tube is about, ~ 0.5 mm in dia while that of the outer tube is such that are can connect a rubber pipe with inner diameter 4mm.

The solution reservoir was made from a borosil glass 250ml flask. The lower end of the flask was elongated and connected to a stopper to control the flow of solution in the vertical arm of the quartz atomizer. The coupling to the solution reservoir is arranged though B19 joint.

Figure 2.1 exhibits the schematic diagram of the spray pyrolysis set-up and the inset shows the arrangement of the shower gush. The spray gun includes a quartz gush having an internal and outside broadness 0.5 and 2.0 mm, separately. The internal cylinder is chosen for n-heptane oil and ferrocene blend supply though the external cylinder coordinates the bearer gas stream towards the spout. This shower spout is settled to the primary quartz tube (500 mm) length and inward distance across (25 mm) by methods for a standard B-29 joint. The delta of the inward container of the sprayer is joined to an isolating pipe which contains the blend of the n-heptane oil and ferrocene. The quartz tube alongside the splash spout framework is put in a 300 mm long cylindrical electrical Quartz were cleaned in CH3)2CO utilizing ultrasonicator pursued by de-ionized water. It is at long last dried utilizing nitrogen blower.



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Available online at: www.jrrset.com

ISSN (Paul) : 2347-6729 ISSN codust : 2348-3105

JIR IF: 2.54 SJIF IF : 4.334 Cosmos: 5.395

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Figure 3 Experimental spray pyrolysis set-up at RTDC for CNTs synthesis

At the point when furnace achieved the ideal union temperature, cock was opened to pass the blend of reactant gasses of n-heptane oil and ferrocene by means of shower spout. Stream of argon gas was kept up to atomize the fluid. 3–4 drops/min was the stream rate which was settled so as to splash the fluid in 60 mins. After the affidavit, the furnace was turned off and permitted to cool ordinarily at encompassing rate.



Figure 4 Unpurified CNTs

Figure 5 Purified CNTs



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Physical and chemical properties of PVP

Molecular weight: Polyvinylpyrrolidone can be set up with an assortment of sub-atomic loads subordinate upon the techniques used to blend the polymer. The atomic weight territory from 2,500 to 2,900,000. Since the polymer comprises of a progression of various chain length polymers, the atomic weight is communicated as a normal of the different sub-atomic loads of the diverse chain length units that include the polymer.

Viscosity : Viscocity does not change considerably over a wide pH go, but rather increments in concentrated HCI. Solid harsh arrangements encourage the polymer, yet this hasten arrangement redissolves on weakening with water.

Solubility: PVP is promptly solvent in virus water and the focus is constrained just by thickness. It is conceivable to get ready free-streaming arrangements of PVP K-30 in fixations up to 60% with just moderate impact on thickness. PVP K-60 and K-90 are accessible monetarily as 45 and 20 percent watery arrangements, individually.

Polycrystalline sample with grain boundary effects

In polycrystalline samples or samples with extremely rough surface, grain boundaries are also present. Due to these grain boundaries the conductivity may either increase or decrease depending upon whether the activation energy for the grain boundary conduction is less or more than that of the bulk conduction.

Using formula 2.5, the bulk ionic conductivity (σ) of a prepared polymer electrolytefilm was evaluated from complex impedance / admittance plot with the help of 'CHinstrument model 604D, U.S.A.' in the frequency range 102 Hz to 105 Hz using thecell configuration Steel / Polymer electrolyte / Steel.A light spring loaded sample holder as shown in Figure 2.11 was used for all the measurement..



ISSN contant : 2348-3105

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Figure 6 Experimental arrangement for electrical conductivity measurement

Wagner's polarization method

This strategy depends on a straightforward rule wherein either the ionic or electronic transport is stifled in a blended conductor by an appropriate decision of the terminal sallowing the commitment of non-smothered species to be resolved. In this method, the sample is sandwiched between two electrodes- one is reversible (non- blocking)which can exchange both ions and electrons with electrolyte and the other is blocking(non-reversible) which can exchange only electrons with the electrolyte. To polarize the sample, a small dc potential is applied across the cell such that the mobile ionicspecies move towards the blocking electrode as shown in Figure.



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Figure 7 (a) Experimental arrangement for measuring transference number byWagner's polarization method, (b) Current vs. Time plot showing the polarizationbehavior

CNT DISPERSED PVP+NH4I POLYMER ELECTROLYTE

Conductivity measurement

As detailed in Chapter 2, conductivity of NH4I doped PVP samples have been obtained from the impedance (Nyquist plot) measurements. Table 1. lists the obtained conductivity values for each sample and pictorial representation is given as Figure

Composition	Conductivity(S/cm)
90:10	2.24 * 10-5
80:20	2.63 * 10-5
70:30	1.09 * 10-4
60:40	3.70 * 10-4
50:50	7.55 * 10-5

Table 1. Conductivity values of PVP:NH4I films at room temperature

Optical microscopy

To make sure our observation, due to CNT dispersion, polarized optical micrographs (POMs) of the CNT-dispersed polymer electrolyte films have been taken. The pictures at



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JIR IF : 2.54 SJIF IF : 4.334 Cosmos: 5.395

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10x magnification are shown as Figure.



Figure 8. Optical micrograph of the (a) PVP: NH4I+.01wt%, (b) PVP: NH4I+.02wt%,(c) PVP: NH4I+0.3wt% and (d)PVP: NH4I +0.4wt% of CNT with 10X magnification

CNT DISPERSED PVP+NaI POLYMER ELECTROLYTE

Conductivity measurement of PVA:NaI

The conductivity of NaI doped PVP samples have been obtained from the impedance (Nyquist plot) measurements. Table 2 lists the obtained conductivity values for each sample and pictorial representation is given as Figure.

Composit	Conductivity(S/cm)
ion	
90:10	3.82 *
80:20	10-6
70:30	4.36 *
60:40	10-6
50:50	8.44*
40:60	10-6
	1.80 *



International Journal on Recent Researches in Science, Engineering & Technology (IJRRSET) ISSN (Heat) : 2347-6729 ISSN (Make) : 2348-3105

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JIR IF : 2.54 SJIF IF : 4.334 Cosmos: 5.395

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10-5
7.16 *
10-5
1.02 *
10-4

Table 2. Conductivity values of PVA:NaI films at room temperature

The conductivity of the polymer electrolyte films are found to increment gradually with the expansion of sodium salt. A sharp rise in the conductivity could be seen while approaching the 60:40 composition. Maximum conductivity was obtained at 60:40 composition at which the conductivity value approaches 1.80 x 10-5 S/cm beyond which it drops down.

The increase in the ionic conductivity can be due to the mobility or number of mobile charge carriers whereas the decrease in the conductivity above 60:40 composition can be due to the formation of non conducting ion pairs or the formation of ionic multiples. This type of behavior can be explained in terms of the ion association theory. Initially when the salt was added, it gets dissolved in the polymer and ions of the salt get dissociated. The dissociated ions then contribute to the ionic conductivity. The initial jump from 0% salt to 10% salt has not been shown in the Figure. However, as the concentration of the ions was increased, due to ion association, immobile charge multiplates are formed which do not contribute to the ionic conductivity. As a result, the conductivity does not increase upon addition of salt. However, on further addition of salt, higher charge multiplates are formed which start contributing to the conductivity and hence conductivity reaches to its maximum value. Beyond this composition, the dipole-dipole interaction dominates and the conductivity drops down.

The maximum ionic conductivity was obtained at 60:40 composition at which the conductivity value approaches $1.80 \times 10-5$ S/cm. It may be noted that, for any device application the desirable conductivity should be in the range 10-3 to 10-2 S/cm. Therefore, to further enhance the conductivity multiwall CNTs synthesized with the help of spray



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JIR IF : 2.54 SJIF IF : 4.334 Cosmos: 5.395

Volume 9, Issue 4 – April 2021 - Pages 1-21

pyrolysis method have been added in the highest conducting (60:40) composition

Optical microscopy



Figure 9. Optical micrograph of the (a) PVP: NaI+0.1wt%, (b) PVP: NaI+0.2wt%, (c) PVP: NaI+0.3wt% (d)PVP: NaI+ 0.4wt% (e) PVP: NaI+0.5wt% (f) PVP: NaI+0.6wt% of CNT with 10X magnification

It can be seen in the literature that pure PVP film shows semicrystalline nature with large interconnected spherulites. Spherulites are the crystalline regions of the polymer/ polymer electrolyte films which occur due to the coiling of the polymer chain. In general, these appear as a strained structure with a center (may not be symmetrical) and stretched towards its periphery. Due to addition of NaI into PVP matrix the spherulite size becomes smaller whereas amorphous region (black portion) increases (Singh and Chandra,2002). It is observed that addition of CNT into polymer electrolyte matrix (PVP:NaI) results to more amorphicity. The dark portion increases drastically. This result shows sensible agreement with our DSC and ionic conduction measurements.



Available online at: www.jrrset.com

JIR IF : 2.54 SJIF IF : 4.334 Cosmos: 5.395

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CNT DISPERSED PVA+NH4I POLYMER ELECTROLYTE

Conductivity measurement of PVA: NH4I

conductivity of NH4I doped PVA samples have been obtained from the impedance (Nyquist plot) measurements. Table 3 lists the obtained conductivity values for each sample and pictorial representation is given as Figure.

Composition	Conductivity(S/cm)
90:1	2.63 *
0	10-5
80:2	1.09 *
0	10-4
70:3	7.55 *
0	10-4
60:4	4.85 *
0	10-4
50:5	7.45 *
0	10-5

Table 3. Conductivity	y values of PVA: NH4I	films at room temperature
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Optical microscopy

To make sure our observation, due to CNT dispersion, polarized optical micrographs (POMs) of the CNT-dispersed polymer electrolyte films have been taken. The pictures at 10x magnification are shown as Figure



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Available online at: www.jrrset.com

ISSN (hut) : 2347-6729 ISSN (huto) : 2348-3105

JIR IF : 2.54 SJIF IF : 4.334 Cosmos: 5.395

Volume 9, Issue 4 – April 2021 - Pages 1-21



Figure 10 Optical micrograph of the (a) PVA: NH4I +0.1wt%, (b) PVA: NH4I +0.2wt%, (c) PVA: NH4I +0.3wt% ,(d)PVA: NH4I +0.4wt% (e) PVA: NH4I +0.5wt% (f) PVA: NH4I +0.6wt% of CNT with 10X magnification

It can be seen in the literature that pure PVA film indicates semicrystalline nature with large interconnected spherulites. Spherulites are the crystalline regions of the polymer/ polymer electrolyte films which occur due to the coiling of the polymer chain. In general, these appear as a strained structure with a center (may not be symmetrical) and stretched towards its periphery. It is observed that addition of CNT into polymer electrolyte matrix (PVA:NH4I) results to more amorphicity. The dark portion increases drastically. This outcome indicates sensible agreement with our DSC and ionic conduction measurements.

CONCLUSIONS

This Investigation is primarily concerned with the development and characterization of carbon nanotube based solid polymer electrolyte used for multiple device applications. We have successfully prepared three new carbon based polymer electrolytes viz. PVP + NH4I+CNT, PVP + NaI + CNT and PVA + NH4I+ CNT by doping CNT into different high molecular weight polymers viz. Polyvinyl alcohol (PVA) and Polyvinyl pyrrolidone (PVP) using solution casting technique. These SPEs were obtained in the form of self



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JIR IF : 2.54 SJIF IF : 4.334 Cosmos: 5.395

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standing thin films of different wt% composition of CNT which were further used in the characterization

Carbon based solid polymer electrolyte (PVP+ NH4I + x wt% CNT) where x = 0.1, 0.2, 0.3, 0.4, 0.5, 0.6

The maximum ionic conductivity was obtained at PVP: NH4I is at 60:40 concentration where conductivity value approaches at $3.70 \times 10-4$ S/cm. It may be noted that, for any device application the desirable conductivity should be in the range 10-3 to 10-2 S/cm. Therefore, to further enhance the conductivity multiwall CNTs added in the highest conducting (60:40) composition.

The conductivity initially increases with the addition of CNT in PVP: NH4I (60:40) system . It attains a maximum value of $\sigma = 8.23 \times 10-2$ S cm-1 at 0.4 wt% CNT composition.

The optical microscopic examinations accommodate the complexation of CNT salt into PVP: NH4I have polymer network and furthermore propose that amorphicity of the host polymer matrix PVP in the polymer strong electrolyte films increments as convergence of CNT increments.

Carbon based solid polymer electrolyte (PVP+ NaI + x wt% CNT) where x = 0.1, 0.2, 0.3, 0.4, 0.5, 0.6

The maximum ionic conductivity was obtained at PVP: NH4I is at 60:40 concentration where conductivity value approaches at 1.80 x 10-5 S/cm. It may be noted that, for any device application the desirable conductivity should be in the range 10-3 to 10-2 S/cm. Therefore, to further enhance the conductivity multiwall CNTs added in the highest conducting (60:40) composition.

The conductivity initially increases with the addition of CNT in PVP: NHaI (60:40) system. It attains a maximum value of $\sigma = 9.04 \times 10-3$ S cm-1 at .5 wt% CNT composition.

The optical microscopic studies conform the complexation of CNT salt into PVP: NH4I



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host polymer matrix and also suggest that amorphicity of the host polymer matrix PVP in the polymer solid electrolyte films increases as concentration of CNT increases.

Carbon based solid polymer electrolyte (PVA+ NH4I + x wt% CNT) where x= 0.1, 0.2, 0.3, 0.4, 0.5, 0.6

The maximum ionic conductivity was obtained at PVA: NH4I is at 70:30 concentration where conductivity value approaches at 7.55 x 10-4 S/cm. It may be noted that, for any device application the desirable conductivity should be in the range 10-3 to 10-2 S/cm. Therefore, to further enhance the conductivity multiwall CNTs added in the highest conducting (70:30) composition.

The conductivity initially increases with the addition of CNT in PVA: NH4I (70:30) system . It attains a maximum value of $\sigma = 6.22 \times 10-3$ S cm-1 at 0.5 wt% CNT composition.

The optical microscopic studies conform the complexation of CNT salt into PVA: NH4I host polymer matrix and also suggest that amorphicity of the host polymer matrix PVA in the polymer solid electrolyte films increases as concentration of CNT increases.

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