Electric Properties of Polyvinyl Acetate (PVA)-Polyol and Prepared Sulfonated Phenol-formaldehyde Resin (SPF) Bulk Samples Composite

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ABSTRACT

Poly vinyl acetate (PVA)/Polyol were blended by weight 3:1. This is mixed with different percentages of prepared sulfonated-phenol formaldehyde resin (SPF). The mixture was prepared on clean glass substrate as a bulk films, at different thicknesses (0.1-0.18) cm. SPF was diagnosed by FTIR spectrophotometer. The current-voltage (I-V) characteristics of the bulk samples were measured at room temperature. These were investigated at time of measurements. The electrical conductivity was investigated and was calculated (10^-7 to 10^-5 to 0.239 to 1.623) S.cm^-1.

Keywords: PVA, Polyol, SPF, Composite, FTIR spectroscopy, I-V characteristics, Electrical conductivity.

INTRODUCTION

Polymers materials in pure state are electrical insulators. However they are filled with specific additives, such as metallic powders or metallic fibres, carbon black, ionic conductive polymer And intrinsically conductive polymer powder[L. I. Soliman and M. W. Sayad (2002)].

Polyvinyl acetate (PVA) is a thermoplastic polymer with chemical formula -(C₄H₆O₂)ₙ. It is normally manufactured by free radical polymerization of vinyl acetate.[Wikipedia (2013)]. PVA is a synthetic resin polymer, which, due its non-polar nature, is insoluble in water, oil, fats or gasoline. On the other hand, it is soluble in alcohols, ketones and esters.[WISEGEEK, (2013)].

A polyol is alcohol containing multiple hydroxyl functional groups. This term means here polymer chemistry, these are compounds available for organic reactions. A molecule with two hydroxyl groups is a diol, one with three is triol and one with four is tetrol and so on.[Wikipedia, (2013)].

Composite are engineering materials made from two or more constituents with significantly different physical or chemical properties which remain separate and distinct on macroscopic level with in the finished structure. One material (the matrix or binder) surrounds and binds together a cluster of fibers or fragments of much stronger material (the reinforcement). For the matrix many modern composites use thermosetting or thermoplastic polymers.[Subita Bhagat, (2013)].
Electric conductivity is one of the most delicate physical properties of polymer composites due to the weakness of the current and size generally small of sample characterized. To a clime a good accuracy measurements of current and voltage across the film, the surface must be large compared with its thickness and high value of applied electric field, thus the geometric shape of the sample almost feasibility of such process; specific preparation of the sample with initial phase during which particular electrodes have to be used.[Bachir Chikh-Bled, etal, (2012)]. Electrically conducting composites based on conducting particles in non-conducting polymer matrix are now being in many practical applications. These filled polymers have a number of advantages in term of absorbing the specific radiation, improving thermal stability, enhancing electrical conductivities and reducing the cost and easy processability to achieve conductivity.[V. S. sangawar, etal, (2006)]. These materials are typically disorder structures consisting of randomly arranged conducting fillers dispersed in polymer medium. Some of the earliest conducting composites were formulated using carbon black or graphite as a filler.[Jamila. Vilcakova, etal, (2007)].

The type of the electric conductivity measurement reported in the literature usually involves simple measurements of current as a function of time, temperature, ambient atmosphere and potential. Attempts are then made to relate the conductivity to physical processes thought to be occurring in the polymer. It is found that electrical conductivity varies exponentially with temperature, is a function of time and may vary with electric field.[M. Serin, etal, (2003)]. Polymeric materials either organic or inorganic are well known insulating materials suitable for many industrial applications such as coating adhesion, coverage, fiber etc. in spite of conflicts and difficulties associated with study of the conduction mechanisms in polymeric materials, some fair results still describe gently the charge carriers migration and its variation with temperature and voltage. Sometimes, it is difficult to prove or disprove the expected conduction mechanism by direct analytical measurements because of low currents implied. Hence, different measurement techniques such as surface conductivity, dc conductivity, thermally stimulated dc current have been employed for studying the relationship between their electrical properties and chemical structure for several purposes such as the high temperature applications because of its thermal and oxidative resistance and high glass transition temperature[Bachir Chikh-Bled, etal, (2012)]. The potential of PVA/polyol blend and SPF mixtures as reinforced fillers rather reflects a significant improvement by reinforcing filler. These significant properties are expected to impart major enhancements in the electric properties of polymer composites. The measured electrical properties gave a motivated considerable interest in the development of polymer composite materials make now this field even more competitive.[Lilian Bokobza (2012)].

Electrically conductive polymers are of great interest as a new class of materials in the field of technology during last two decades, the effect of polymer blending on the electrical conductivity of polymer composite films was investigated such as polypyrrole/copolyester composite films and have studied intensively to improve environmental stability and mechanical properties.[Doo Hyun, etal, (1998)]. Interaction between SPF and PVA/polyol implies a strong interfacial bonding, while functional properties of the composite greatly depend on the conductive structure of SPF.[You zeng, etal, (2010)]. Sulphonated phenol-formaldehyde resin is prepared by the method which was described in experiment. The preparation of PVA/Polyol and SPF polymer composite as conductive polymer sample. Fundamental research’s and potential applications in field of
conductive polymer composites, since the electrical conductivity of conjugated polymers can be increased by many orders of magnitude. [Yun-Ze Long, et al, (2010)].

**EXPERIMENT**

1. **Preparation of Sulfonated Phenol-Formaldehyde Resin**

42.5 moles of phenol was put in clean tri-neck round Flask 500 ml. in capacity, which was emplaced in Isomental heater sort LabHeat BAECO, Germany. The side neck (B19) (mm. inner diameter) of the round flask was closed by stop-fit thermometer and the other side (B19) (mm. inner diameter) was closed by a condenser which is connected to water pump emplaced in ice path, while a stirrer sort Heidolph, Germany is inserted in the middle neck (B24) (mm. inner diameter), the system was run and the phenol was heated to appropriate temperature to dissolve any solid bodies. The system was stopped and 4 moles of sulfuric acid 97% in concentration Thomas Baker India, was added slowly from one side neck by using pipettes. The round flask was closed again as above and the system was operated, while the stirrer was adjusted to appropriate speed and the temperature is raised, which is maintained between 100-120 °C for two hours. The system was stopped and the temperature was cooled slowly, then the round was emplaced in ice path, 12 moles of Formaldehyde Thomas Baker, India. Was added by using pipettes, a fizzing and bubbling have occurred, the temperature is raised and stirring by hand was done using glass rod and the temperature was cooled to 35 °C then below 22 °C. a stirring was continued until a viscose solid mass is obtained. The product was left over night. The PH was examined by using indicator paper which is colored red low PH. NaOH solution was prepared in a separate flask and drops were added until over saturation is reached high PH, a few drops were add of H₂SO₄ for equilibrium until PH=7 was reached. The solution was removed in flask and the precipitate resin was put in a glass plate to be dried at room temperature and the product was collected in plastic container. Fig.(1), shows the setup of instruments used.

![Fig.(1). The setup of instruments used.](image-url)
2. **FTIR Test**

Sample of sulfonated phenol-formaldehyde resin, was examined with KBR disc by Fourier transform Infrared Instrument (FTIR) as in Fig.(2). The peaks at 1128.39 cm\(^{-1}\) and 1175 cm\(^{-1}\) corresponding to C-C-O asymmetric stretch and C-H in plane formulation respectively while the 1000 cm\(^{-1}\) and 748.8 cm\(^{-1}\) peaks belonged to the C-H out of plane vibration. The peak at 1506.37 cm\(^{-1}\) corresponded to the C=C aromatic ring vibration. The above mentioned peaks diminished with increasing reaction time while the absorbance band of hydroxyl groups have increased.[Ida Pojansek and Matgaz Krajnc (2005)].

![Fig.(2). The FTIR spectroscopy of SPF](image)

3. **Preparation of Samples**

Glass substrates were cleaned by rinse with distilled water then by acetone, again with distilled water and dried in oven under vacuum for one hour. PVA-Polyol blend were prepared by weight 3:1 (high molecular weight and low molecular weight) using sensitive electric balance sort Sartorius, Germany and was blended on the clean glass substrates by hand using spatula. Percentages by weight of sulfonated phenol-formaldehyde resin (SPF) were crushed using Pyrex mortar, were added and mixed by hand using spatula until the required mixtures were obtained and to ensure uniform thicknesses. Two copper wires were connected at both ends of bulk films. This is left to dry overnight. The thicknesses and diameter of the electrode were measured using Capilar Certificate Vernier made in China, and micrometer Starret, U.K. respectively, as in Table-1. The samples then undergoing electrical measurements.

4. **Electrical Measurements**

Measurements of current-voltage characteristics of the prepared samples were carried out using the set up in Fig.(1), consists of DC power supply sort, Lybold and Heros, U.K. Amplifier, sort PHYWE, Germany which is connected to a pointer ammeter for low current measurements, sort PHYWE, Germany. Digital voltmeter was used to measure the voltage of the bulk samples. The electrical contact of the wires was made as electrode configuration to reduce any effect of leakage current. The samples were put in side front slide glass of wood box to prevent the effect of the environment. The schematic diagram of...
the circuit is used for measurements as in Fig.(3). The current-voltage was measured at applied electric field 13 volt DC. Table-2. Shows the I-V characteristics of the samples. The equation which, was used for calculating the electrical conductivity from Table-2,

\[ \rho = \frac{RA}{L} \]
\[ \sigma = \frac{1}{\rho} = \frac{L}{RA} \]

where:
- \( L \): is the length of the bulk sample.
- \( R \): is the resistance of the sample.
- \( A \): is the effective area of the electrode of the sample \( A = \pi D^2/4 \) as in Table-1.

![Fig.(3). The electrical measurements circuit](image)

**Table (1). Sample preparations of PVA-Polyol and SPF.**

<table>
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<tr>
<th>PVA/ Polyol gm.</th>
<th>percents of SPF</th>
<th>thickness cm.</th>
<th>Length cm.</th>
<th>Width cm.</th>
<th>effective area cm(^2)</th>
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Table 2. I-V characteristics of the prepared samples.

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Fig. (4). I-V characteristics of PVA and Polyol blend with 5% SPF.

Fig. (5). I-V characteristics of PVA and Polyol blend with 10% SPF.
RESULTS AND DISCUSSIONS

The shapes of plots obtained from Table-2. The I-V characteristics depends on applied field the linearity of the graph obtained in which some of the points were shifted from the straight line for the bulk sample of PVA-polyol with 5% SPF in time of measurements 58 min. as shown in Fig.(4). There is sudden increase in current at voltage 44 mv. Conduction is predominantly achieved through the sample with 5% SPF and was controlled. Because of the instability of low current passes through the sample by the resistance of film against the current which passes through under certain applied field. [Jamila Vilcakova, etal (2000)] work on polyester resin filled with different contents of carbon fibres (0-10wt%) for measurement of DC conductivity depend strongly on filler content. As SPF increased to 10%, the linearity of the graph was rather instable but shows points become more close to the straight line obtained between the current and voltage as shown in Fig.(5)., the system needs to overcome the resistance so that increase the transferred electron and the current is low even with high electric voltage. With SPF 15%, the current passes through the sample was affected by weak Van der Waals interaction so there is no decrease in stability in the composite.[J. N. Coleman (1998)], The electrical conduction depends on the shape geometry and structure of the composite which reflects the graph linearity of current density dependent on the electric field.[D. D. Chung, (2001)]. At the beginning of the measurements and the current increased rapidly with time after 42 minutes, the experiment controlled by the source measurement unit allowing the measurement of the
currents in nA to µA range as in shown Fig.(6). In agreement with work of [Jamila Vilcakova, etal, (2000)]. The current passes through the sample become rather instable at 20% SPF and increased with time of measurements 55 min. in the range indicated from which a straight line was obtained with voltage across the sample as shown in Fig.(7). In the investigation of carbon nanotubes asa dopant material. A semiconjugated, organic polymer was mixed with carbon nanotubes to form a wholly organic composite. This behavior is indicative of percolative character in composite systems. Percolation theory deals with the effect of varying, in random system, the interconnections in this case are the highly conductive polymer composite.[J. N. Coleman (1998)].

Increased current with time of measurement 47 min. with SPF 30% by weight of sample and thickness of the bulk film was 0.17 cm. gave a significant dependant of linear graph obtained on film thickness. At critical concentration of SPF, beyond which the polymer composite becomes conductive is referred to as the percolation threshold at this point the conductive network is formed through composite constituents. This permits the movement of charge carriers in the SPF through the polymer composite constituents, and so the composite achieves a certain degree of electrical conductivity as shown in Fig.(8). The investigation of composite formation from low to high nanotubes concentration increases the conductivity dramatically by ten orders of magnitude indicative of percolative threshold [J. N. Coleman, (1998)].

Electric measurements of polymeric composites, these materials are typically disordered structures consisting randomly arranged filler dispersed in polymer medium.[Jamila Vilcakova, etal, (2000)]. Fig.(9). Shows that the prepared SPF was increased to 40% by weight, which is more convenient of current and voltage measurements across the bulk sample. The current was increased with increasing voltage at time 72 min. The thickness of the sample was increased to 0.18 cm. also reflects linear dependencies of the graph.[Torbio f. Otero and Joes G. Mrtines, (2010)] they have shown in their work on oxidation rates of polypyrrole films on clean platinum electrodes, electron transfer decrease very fast for increasing film thicknesses.

Due to the effects SPF on PVA-Polyol molecular structures on their electrical conductivity, this is increased with increasing SPF. In the work on nanotube filled elastomers, the processing conditions have strongly effect on composite properties especially on electrical properties. The use of nanoscale conducting filler such as carbon nanotubes (CNTs) has proven to be effective reducing the filler content required to achieve the electrical percolation threshold. [Lilian Bokobza, (2012)]. Above certain amount of conductive particle, called the percolation threshold, an interconnecting filler network formed. This is result in the sharp drop of the electrical resistance of the composites.

According to shape geometry of the bulk samples, the current density passes through the samples were plotted as a function of applied field strength. Polymer composite are intensively studied for the new properties which are given by the combination of the properties of both polymer matrix (SPF) and binds together a cluster or fragments of a much stronger material (the reinforcement) respectively. When the concentration of the binds in the composite reaches the percolation value, the continues bulk network structure is formed. If (SPF) binder becomes electrically conductive the composite properties can change from insulator to conductive ones. The investigation on PVC-NI system has showed the dependence of electric conductivity on filler content. Electrical conductivity can change in the magnitude of the several orders. [Ye. P. Mamunya, etal, (2002)]. From Table-2, the calculated electrical conductivities from the reciprocal of the resistivity were increased by many orders of magnitude from $10^{-7}$ to $10^{-5}$ to 0.239-1.623 S.cm⁻¹. Values have been given of electrical conductivity of conjugated polymers can be increased by many orders of magnitude from $10^{-10}$ to $10^{-5}$ S/cm upon doping, such as carbon nanotubes, inorganic
semi-conducting nanotubes/wires, and conjugated polymer nanotubes/wires.[Yun-Ze Long et al (2010)]. The dependence of conductivity on (SPF) shows sharp rise (percolation threshold).[Subita Bhagat, (2013), Jan Lptack, etal (2010)]. The electrical conductivity is a sensitive probe of composite, the percolation behavior in the electrical conductivity of composite at relatively loading percentages of SPF (5 and 10)wt%. in the work on carbon nanotubes (CNTs)-polymer composite. The high aspects ratio is known to be advantageous in making a percolation net work at relatively small loading percentages (of order 1wt%).[E. S. Choi, etal, (2003)].

**CONCLUSIONS**

The investigation was carried out in order to evaluate the electrical properties of PVA-polyol with SPF mixtures. On the basis of obtained results the following conclusions were drawn:

- The effect of SPF on polymer blending PVA-Polyol was investigated. Improved electrical conductivity approached with increasing SPF% by weight. There is jump in current rather than electron transfer.
- Dry SPF can influence the resulting structure in the composite by the effect of the polymer and the SPF network formation is van der Waals interaction between SPF surrounds and binds.
- Thicknesses of bulk samples were given a significant dependant of linear graphs obtained on samples thicknesses rather than the voltage across the sample.
- Effect of external field-electrical, when applied SPF particles agglomerations preferred in the direction of the electric field force lines results to the SPF chains.
- Conductive polymers have many advantages over metallic conductors; they can be easily shaped with low cost technologies: they have light weight; they provide corrosion resistance and they can offer wide range of electrical conductivities.
- At critical concentration of SPF, conductivity around percolation threshold is formed through the polymer composite.
- The increase of SPF wt% will alter the dependencies of current passing through the bulk samples on the voltage, due its effect on PVA-Polyol molecular structures on their electrical conductivity.

**REFERENCES**


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