

# Fabrication and Characterization of PVP: Aluminium Nitride Polymer Nano Composites

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**Abstract**— The purpose of this study is to enhance the electrical, thermal and mechanical properties of PVP polymer composites by the addition of nano particles. The mechanical properties of polymer nano composites such as tensile strength, micro hardness number and degree of swelling have been measured and the results are presented. Tensile test results revealed the Young's modulus and strength increasing with percentage of AlN in polymer nano composites. Micro hardness number has been increased from 24 to 48 kgf/mm<sup>2</sup> for polymer nano composites that shows increase in resistance to deformation than Pure PVP. The XRD study reveals the amorphous nature of the polymer Nano composites. SEM results reveal that homogenous distribution of AlN nano particle in polymer nano composites.

**Keywords**—Poly vinyl pyrrolidone (PVP), Aluminium nitride (AlN), Scanning Electron Microscope (SEM), tensile test, X-Ray Diffraction (XRD).

## I. INTRODUCTION

Polymers are generally produced as powders, pellets, and liquids. In order to produce polymeric products with desired shapes, thermoplastics must be melted and cool to a final product shape whereas thermosets must undergo further polymerization to complete cross linking reactions to finally solidify into the designed shape. These operations are called processing. Many factors influence the processing operations. Those include viscosity, orientation of heterogeneous phases, rate of reactions, and volatile formation. Among these factors, the viscosity consideration is by far the most dominant factors in processing. Viscosity is strongly influenced by temperature, shear rate, molecular weight and its distribution, molecular structure of the polymeric chains and heterogeneity of materials. Thus, the study of flow behavior of polymeric materials, which is called rheology, is very important. A composite is a heterogeneous substance consisting of two or more materials which does not lose the characteristics of each component. This combination of materials brings about new desirable properties. The main objective of this study was to investigate the effect on ionic conductivity and mechanical properties of poly vinyl pyrrolidone (PVP) with potassium iodide (KI) doping salt upon addition with and

without the nanosized Aluminium Nitride inorganic filler. The analyses are made; X-ray diffraction, scanning electron microscopy, Differential scanning calorimetry, Optical microscopy, Thermo gravimetric analysis, Tensile Test, Micro hardness Test.

## II. MATERIALS & FABRICATION PROCESS

The different materials used to prepare the polymer nano composites specimen are Poly vinyl pyrrolidone (PVP), Potassium Iodide (KI) and Aluminium nitride (AlN).



Fig 1: PVP, Potassium Iodide & Aluminium nitride powder

**Poly vinyl pyrrolidone** has drawn a special attention amongst the conjugated polymers because of its good environmental stability. PVP is a potential material having a good charge storage capacity and dopant dependent electrical and optical properties. Chemically PVP has been bound to be inert, nontoxic and it displays a strong tendency for complex formation with a wide variety of smaller molecules.

**Potassium iodide** is ionic KI. It crystallizes in the sodium chloride structure. It is produced industrially by treating KOH with iodine. A white crystalline solid, with a strong bitter taste, soluble in water, ethanol, and acetone. It may be prepared by the reaction of iodine with hot potassium hydroxide solution followed by separation from the iodate by fractional crystallization.

**Aluminium nitride** is a nitride of aluminium, a semiconductor material, giving it potential application for deep ultraviolet optoelectronics.

There are different methods of preparation of polymer electrolytes. For the present study, simple and most

commonly used solution casting method is used. This method includes the following steps:

- Polymer is dissolved in the solvent and allowed to dissolve.
- Appropriate amount of salt is added to that polymer solution.
- Mixing by means of magnetic-stirrer for complexation.
- Casting the mixture on a glass plate/Petridish/substrate,
- Drying in vacuum or in an inert atmosphere.

For the preparation of specimen, 0.5g of PVP was taken in a conical flask and 2 ml Triple distilled water is added to the polymer. After dissolving the polymer in the water medium, appropriate amount of KI was added in that solution. Then the solution was stirred for 2 hours by means of magnetic-stirrer. The obtained solution was then casted on separate Petridish and allowed to evaporate slowly at room temperature. The process is repeated for different % of potassium iodide. (10%, 20%, 30%, 40%).

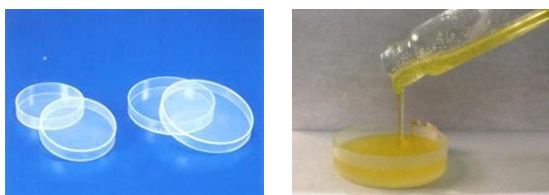


Fig 2: Petridish & Casting process

Later, the samples were cut into a proper size for testing mechanical properties and structural behavior of the polymer composite.



Fig 3: Specimens with different percentage of KI

To obtain the polymer nano composites, the solution casting process is repeated with the mixture of PVP+KI+AlN. The samples were then cut into a proper size for testing mechanical properties and structural behavior of the polymer nano composite.



Fig 4: Polymer nanocomposites

### III. CHARACTERISATION & TESTING

In the current study, several tests have been conducted to characterize the micro-structural, conductivity and mechanical properties of prepared polymer and polymer nano composites.

**Scanning electron microscopy (SEM)**, is a versatile tool to characterize microstructures of the samples SEM makes use of point to point scanning of the solid surface, which produces a clear image of specimens, and provides information about their size which lies in the range of micro-meter.

**X-ray diffraction (XRD)**, is a powerful tool in the identification of atomic structure of crystalline substances. For eg: minerals in rocks and soils. BRUKER D8 XRD instrument is used to understand to crystallinity of polymer nano composites.

**Micro hardness** is an indentation hardness tests used to determine the hardness of a material to deformation. Here the examined material is indented until an impression is formed; these tests can be performed on a macroscopic or microscopic scale.

**Tensile testing** is a fundamental materials science test in which a sample is subjected to a controlled tension until failure. The results from the test are commonly used to select a material for an application, for quality control, and to predict how a material will react under other types of forces. In this study, tensile test is conducted to obtain Young's modulus, Poisson's ratio & yield strength of the sample. The specimen for tensile test has been prepared as per ASTM standards. The test was conducted on tensometer instrument.



Fig 5: ASTM D638 Type PVP +KI+AlN nano composite sample

## IV. RESULTS & DISCUSSION

**Scanning electron microscopy** was used to determine size and distribution of particles sample. The SEM images obtained for polymer and nano composite are shown below:

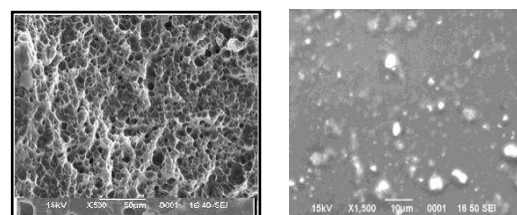


Fig 6: SEM images of PVP+30%KI polymer (a) & PVP+30%KI+12% AlN nano composite (b)

In fig 6(a), the black color dots represent the salt structure and white color shows the polymer structure. The salt completely dissolved in distilled water and uniformly distributed in PVP polymer. Fig 6(b) shows comparative SEM scans at high resolution of a portion of the polymer nano composites which indicates distribution of nano powder in polymer composites which shows in white dots.

**X-ray diffraction**, Fig 7 depicts the XRD patterns of PVP/Aluminium nitride nanocomposites. The peaks are seen at the same 2θ Angles where AlN show peak intensities. The fig 6.14 shows the change in morphology of the polymer. Polymers are always amorphous but due to addition of crystalline nano powder there is introduction of crystallinity in polymer matrix.

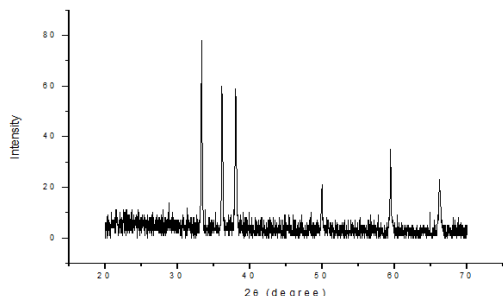


Fig 7: XRD pattern of PVP+30%KI+12%AlN nano composite

The XRD patterns showed the presence of few crystalline peaks and amorphous humps indicating the semi crystalline nature of all the polymer nanocomposites. From the XRD patterns it is observed that, two main peaks are appearing around 34° and 39°. At 50° we can see Sherrer Broadening Effect because the size of the powder is below 100nm.

**Micro hardness** test was conducted on specimens on loads varying from 10gm to 100gm and observed under a microscope with magnification of 100X to 1000X. The test results of hardness test have shown in the table below:

Table 1: Micro Hardness for Polymer Nano composites

Specimen	Load (gm)	d <sub>1</sub> (mm)	d <sub>2</sub> (mm)	d* = (d <sub>1</sub> + d <sub>2</sub> ) / 2 (mm)	Scaling Factor d = d*0.1154	HV = 1.85 4F/d2 (kgf/mm <sup>2</sup> )
PVP	100	0.7412	0.7802	0.7616	0.08790	24
PVP+KI+AlN (12%)	100	0.5934	0.6048	0.5974	0.0689	41
PVP+KI+AlN (16%)	100	0.6121	0.6315	0.6218	0.0717	48

From the hardness values obtained, it is clear that the micro hardness increases with increasing AlN content due to the increase in crystalline of the PVP fraction in composite and higher micro hardness of AlN compared to pure PVP.

**Tensile Test** was conducted on samples with different percentage of salt, i.e. 0%, 10%, 20%, 30%, 40% KI in PVP. The results reveal that the mechanical strength decreases with increase in % of salt in PVP polymer matrix. Stress–strain data, acquired on many samples of the molecular composites, have been used to determine the values for several mechanical properties. A typical stress–strain response for all case of sample of the polymer nano composite made using the ionically modified is shown in figure below:

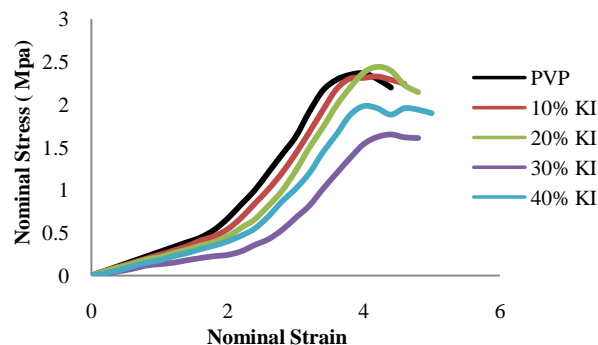


Fig 8: Stress Strain curve for different percentage of KI in polymer

From the graph, it can be concluded that the ultimate stress is maximum where polymer composite has well crystalline.

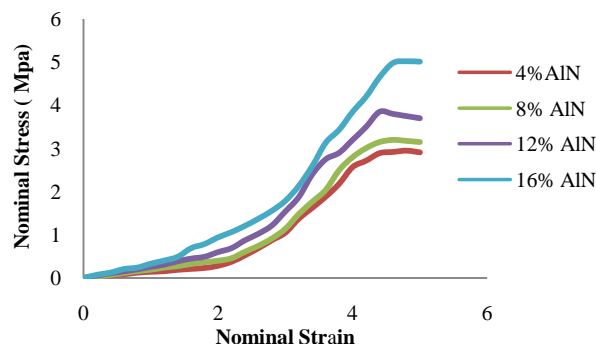


Fig 9: Stress Strain curve for different percentage of AlN in polymer

From the graph, it can be concluded that percentage of nano particle increases the ultimate strength of Polymer composites increases due particles size of which gives strength to composites.

**CONCLUSIONS**

A new type of polymer nano composite was produced using as the polymer matrix. A method was developed to allow to be used as the polymer matrix in polymer nanocomposites. The polymer nanocomposites require only small additions of nano material in comparison to traditional composite materials. The project work reported found that as little as 16 wt% of aluminum nitride nanoparticles effected a change in the mechanical properties of the original polymer. Potassium Iodide and aluminum particles are both effective materials at creating a polymer nano composite. The Potassium Iodide appears to be slightly more effective in increasing conductivity and aluminum nano particles appears to be more effective in increasing mechanical properties. The following conclusions were drawn from the present study:

- X–ray diffraction (XRD) studies implied the higher degree of amorphous nature of the polymer electrolytes by reducing the intensity of characteristic peaks. XRD patterns of PVP polymer nanocomposites doped with AlN showed the decrease of intensity of peaks corresponding to pure AlN with the increase of

nano powder wt% ratio suggesting a decrease in the crystalline of the complex.

- By analyzing the SEM images, the higher porosity revealed the improved ionic migration in the sample. SEM image of PVP/KI composite revealed presence of distinct spirulites in the sample.
- The results of the tensile strength analysis were not as promising as was predicted. Tensile strength was more in pure PVP polymer and tensile strength decreased with increase in percentage of sodium iodide salt.

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