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**ABSTRACT**

A nano composite is a multi phase solid material where one of the phases has dimensions of less than 100 nano meters. Nano composites have different properties for specific applications. In this work PVP based polymer nano composite has been prepared by using nano materials such as Aluminium Oxide, Aluminium Nitride, Nickel Oxide, Barium Titanate and Zinc Oxide. FTIR Spectroscopy has been used in the present work to assessing purity of polymer nano composite fabricated by solution cast technique FTIR Spectra of various composition of polymer nanocomposite has been determined and discussed in detail highlighting the features FTIR Spectra of different polymer nano composite system

**Keywords:** Polymer, Nano composites, FTIR, Nano materials, FTIR Spectra

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**INTRODUCTION**

A nanocomposite is a multiphase solid material where one of the phases has dimensions of less than 100 nanometres. Nano composites have different properties for specific applications. These materials can be in form of gels, copolymers and porous materials. Polymer nano composites are materials containing inorganic materials that have nano-dimensions.

The incorporation of inorganic nano particles as dopants into polymer systems results in polymer nano composites which has excellent characteristics suitable for application. The doping at the nano scale level can improve properties of polymer composite for the requirements of different applications. Moreover synthesis methods, morphology, nanoparticle, surface treatments are important factors. By optimizing these factors significant improvement have been noticed.

In this work PVP based polymer nanocomposite has been prepared by using nano materials such as Aluminium Oxide, Nickel Oxide Alumina and Zinc Oxide and FTIR Spectroscopy studies are carried out.

Feng-Chih Chang, et al [1], prepared PVP based polymer electrolyte by dissolving desired amount of the PVP and the vacuum-dried LiClO<sub>4</sub> salt. After continuous stirring for around 4 hours at room temperature, the solutions were allowed to stand at 40-50 °C for 24 hours to facilitate the evaporation of the solvent, and then further dried under vacuum at room temperature for additional 12 hours. All these complexes were stored at inert atmosphere to avoid contact with moisture from air.

Many complex salts have been developed; fast ion conducting ceramics are being used as an additive to increase mechanical strength as well as to increase ionic conductivity. Suriani Ibrahim, et al [2], have prepared polymer electrolyte with complex salt like lithium hexafluorophosphate. They prepared Polymer electrolyte by standard solution-casting techniques. PEO (MW = 600,000, Acros Organics) was used as host polymer matrix, lithium hexafluorophosphate (LiPF<sub>6</sub>) (Aldrich) as the salt for complexation and ethylene carbonate (EC) (Alfa Aesar) as plasticizer. CNT was prepared by chemical route at low temperature. Prior to use, PEO was dried at 50°C for 48

hours. Appropriate quantities of ingredients were dissolved separately in Acetonitrile and stirred well for 24 hours at room temperature to form a homogeneous solution.

Shiao-Wei Kuo, et al [3], prepared PVP based polymer electrolyte. They prepared Zinc salt/PVP blends having varying zinc content by dissolving the desired amounts of the polymer and  $Zn(ClO_4)_2 \cdot 2H_2O$  in DMF. After stirring continuously for 12 hours, these solutions were maintained at 500°C for 24 hours on Teflon plates to facilitate desolution, and then dried further under vacuum at 900°C for 2 days.

Chong Min Koo, et al [4], prepared PVP/MMT nanocomposite by two methods to investigate the effect of mixing condition on the properties. One is simple stirring. The other is more rigorous mixing by using attrition ball milling, in which zirconium balls with diameter of 1 mm was used at 300 rpm in the zirconium jar. MMT was dispersed for 2 weeks in water for sufficient wetting before adding PVP. The concentration of solute was kept below 7 wt%. PVP/MMT/H<sub>2</sub>O suspensions were prepared by stirring for 1 week after adding PVP. Nanocomposite films were obtained by casting the PVP/MMT/H<sub>2</sub>O suspensions.

E.F. Spiegel, et al [5], experimented with Lithium trifluoromethane sulfonated and PEO polymer electrolyte. Experiment was set-up in the dry box in an argon atmosphere using a 250 ml three-neck round bottom flask. Polymer, lithium salt and plasticizer solvent were weighed and transferred to the round bottom flask followed by addition of solvent. Upon completion of transfer of reactants the flask was capped to avoid introduction of oxygen and removed from the dry box. Solution was stirred for 24 hours to ensure that dissolution had occurred. The temperature was varied between 35°C and 500°C depending upon solution composition.

Ashuthosh Verma, et al [6], conducted experiments PVP to study the electrical characteristics. No dopant was used in this research. Thin PVP films were grown from a suitable solution by the isothermal immersion technique. PVP (2.4 g) had been dissolved in 30 ml of chemically pure chloroform at room temperature and continuously stirred for about 60 min by means of a Teflon coated magnetic stirrer. Thereafter, it had been stirred and heated at 50°C to yield a homogeneous solution. The PVP films were grown on metal coated thoroughly cleaned glass substrates. After films deposition, the glass slide has been taken out and out gassed in air at 40°C for 24 hours to remove all the traces of solvent. This was followed by room temperature out gassing at  $10^{-5}$  Torr for a further period of 24 hours to reduce the memory effects and enhance the repeatability of the measured currents. Metallic electrodes of area 1.33 cm, are vacuum deposited on interfacial layers.

R.J.Sengwa, et al [7], conducted research on different Mw PVP polymers mixed with glycerol. The polymer investigated in this work was poly (vinyl Pyrrolidone) (PVP; average molecular weight  $M_w = 24000, 40000, 360000$  g mol<sup>-1</sup>). The Laboratory grade PVP40000 was obtained from Loba Chemie, Mumbai, and PVP 24000 and PVP360000 were purchased from S D Fine Chem, Mumbai. Glycerol of spectroscopic grade was purchased from Hi Media Laboratories, Mumbai. The blends of PVP24000-Gly, PVP40000-Gly and PVP360000-Gly were prepared in the range 0-30, 0-25 and 0-20 wt% PVP.

U. Sasikala, et al [8], study on the ion transport property studies on plasticized Na<sup>+</sup> ion conducting PEO/PVP blended solid polymer electrolyte (SPE) membranes, 95[35PEO:35PVP:30NaF] : 5x, where x = PEG200, PEG400, PEG600 were reported. SPE films were prepared using solution cast technique. The solid polymer electrolyte film, (35PEO:35PVP:30NaF), also prepared and identified as the highest conducting composition at room temperature.

A Chandra, et al [9], carried out a study on the ion transport property studies on Ag<sup>+</sup> ion conducting PEO–PVP blended solid polymer electrolyte (SPE) membranes, (1 –x)[90PEO : 10AgNO<sub>3</sub>] : x PVP, where x = 0, 1, 2, 3, 5, 7, 10 (wt%), are reported. The conventional solid polymeric electrolyte (SPE) film, (90PEO: 10AgNO<sub>3</sub>), also prepared by the hot-press method and identified as the highest conducting composition at room temperature on the basis of PEO–AgNO<sub>3</sub>-salt concentration.

SK Tripathi, et al [10], carried out a study on the Polymer blend electrolytes composed of poly(vinylidene fluoride-co-hexafluoro-propylene), poly (methylmethacrylate) and 1.0 M NaI as salt have been synthesized using solution cast technique by varying the PVdF(HFP)–PMMA blend concentration ratio systematically. XRD studies reveal

complete complexation of the salt in the polymeric blend systems. The dependence of conductivity on temperature has been performed and it is observed that it obeys the Arrhenius behaviour.

S Tachikawa, et al [11], carried out a study on the Optical properties of ZnO nano particles capped with polymers were investigated. Polyethylene glycol (PEG) and polyvinyl Pyrrolidone were used. They found the time of adding materials have the effect on the properties.

Andres Bernal, et al [12], studied on the PVA and PVP blended films obtained by casting method. The fillers like lactic acid, Glutaraldehyde and hydrochloric acid were added to investigate their effect on mechanical properties of the blends with the aim to find possible material properties for medium or long term implants. Combination of all three additives proved as the best choice for films production with respect to examined properties important for any medical application.

Z L S Seow, et al [13], carried out a study on several important parameters like precursor concentration, rate of evaporation and reaction time. They determine the growth of ZnO nanostructures and these parameters can be optimized and tuned to produce a variety of nanostructures.

P.M. Sirimanne, et al [14], carried out a study on the ZnO films with different morphologies were synthesized electrochemically. They found that addition of selected precursors into the electrolyte changes the morphology of ZnO films and yields hybrid organic-ZnO films.

## POLYMER COMPOSITES

The various system of polymer nano composites are as considered below

PVP +KI, PVP +KI+Al<sub>2</sub>O<sub>3</sub> and PVP+KI+AlN system

PVP+NaI, PVP+NaI+ZnO and PVP+NaI+BaTiO<sub>3</sub> system

PVP+KOH and PVP+KOH+NiO system

PVP+NH<sub>4</sub>I and PVP+NH<sub>4</sub>I+NiO system

## FTIR STUDIES

The term Fourier transform infrared spectroscopy originates from the fact that a Fourier transform (a mathematical process) is required to convert the raw data into the actual spectrum. Goal of FTIR is to measure how well a sample absorbs or transmits light at each wavelength. The Figure 1a shows FTIR spectrometer and the typical FTIR spectra are shown in the Figure 1b. FTIR Spectroscopy is used for identifying chemical bonds in a molecule with help of an infrared absorption spectrum. The spectra produce different profile depending on the sample, molecular fingerprint that can be used to screen and scan samples for many different components.

When a material is subjected infrared radiation, absorbed radiation u excites molecules into a vibration state. The absorbed light wavelength by a molecule is a function of the difference in energy of rest and excited vibration states. The particular wavelengths absorbed depends characteristic structure of the molecule.

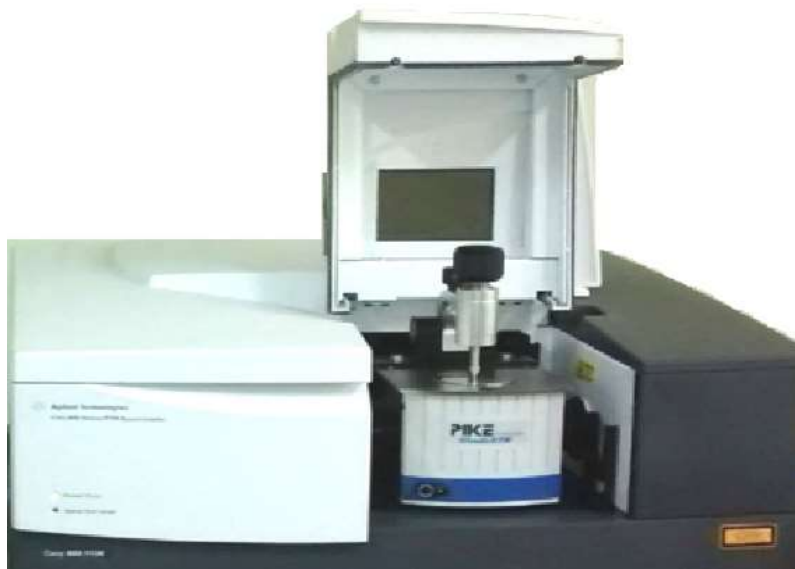


Figure 1a Agilent Technologies Cary 600 series FTIR spectrometer.

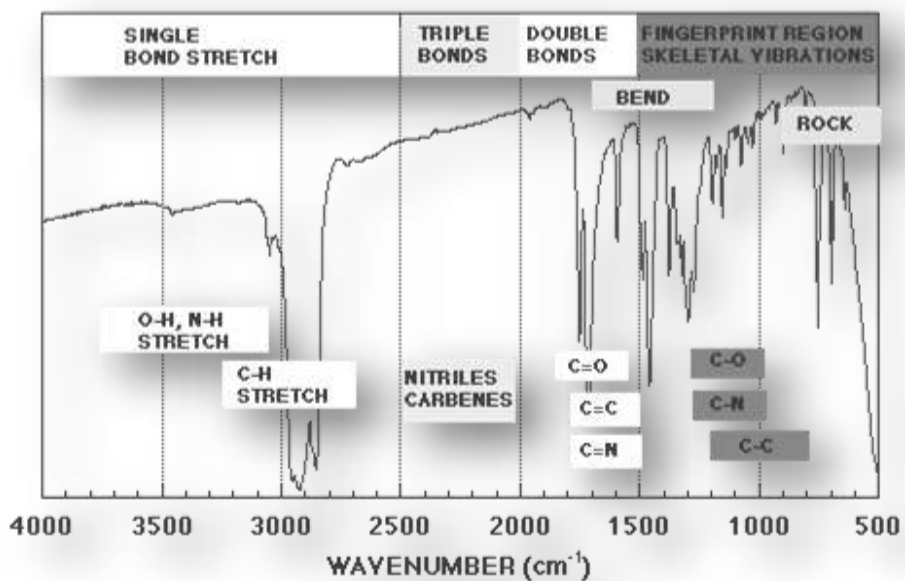


Figure 1b Typical FTIR spectra

The following steps were carried for FTIR

- a) Agile Technologies FTIR spectrometer 600 series was used to conduct FTIR test.

b) This spectrometer simultaneously collects spectral data in a wide spectral range from 400 to 4000  $\text{cm}^{-1}$  range of wavelength.

c) It was used to determine percentage (%) transmittance of intensity at various spectral ranges of wave lengths.

d) Specimen was placed in the specimen holder and the percentage (%) transmittance was noted at different range of wave length.

e) Plotting of data to obtain FTIR using ORIGIN software.

Agile Technologies FTIR spectrometer 600 series was used to conduct Fourier Transform Infrared Spectroscopy. The spectral data are collected by spectrometer simultaneously in a wide spectral range from 400 to 4000  $\text{cm}^{-1}$  range of wavelength. It was used to determine percentage (%) transmittance of intensity at various spectral ranges of wave lengths. Specimen was placed in the specimen holder and the percentage (%) transmittance was noted at different range of wave length.

## RESULTS AND DISCUSSIONS

The FTIR Spectra of various nano composite systems are as follows

### FTIR Spectra of PVP+KI, PVP+KI+Al<sub>2</sub>O<sub>3</sub> and PVP+KI+AlN System

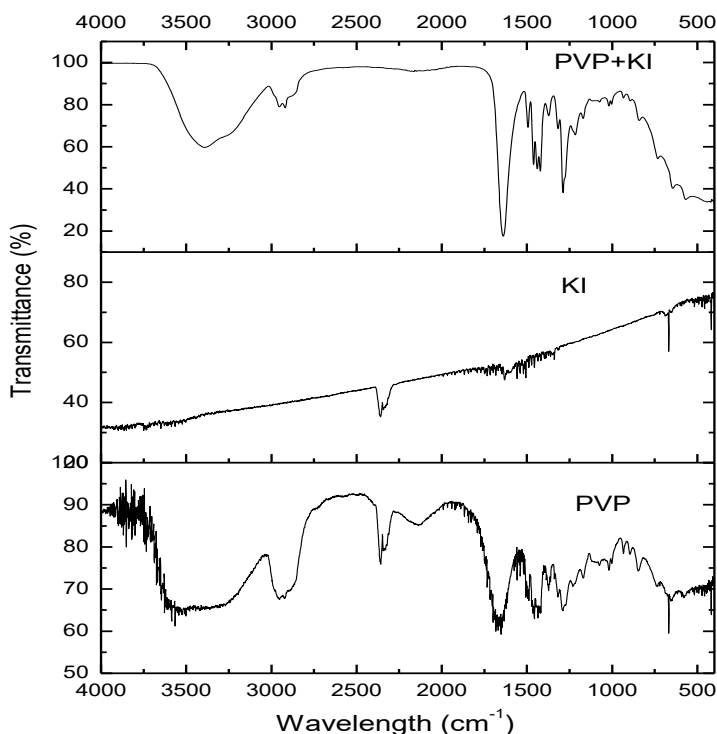


Figure 2a FTIR Spectra of PVP+KI system

Figure 2b shows FTIR Spectra of pure PVP KI and PVP+KI system. The vibration bands observed at 758  $\text{cm}^{-1}$  and 1474  $\text{cm}^{-1}$  are ascribed to CH<sub>2</sub> rocking and C-H deformation of cyclic CH<sub>2</sub> group of pure PVP, respectively. The vibration peaks at 844  $\text{cm}^{-1}$ , 1018  $\text{cm}^{-1}$  and 2135  $\text{cm}^{-1}$  in the FTIR spectrum are attributed to CH<sub>2</sub> bending, C-C

stretching and C-N stretching vibration of pure PVP, respectively. The peaks at  $2135\text{ cm}^{-1}$  and  $1018\text{ cm}^{-1}$  are present and the vibration peak observed at  $896\text{ cm}^{-1}$  assigned to CH<sub>2</sub> rocking. The CH<sub>2</sub> wagging vibration peak is at  $1328\text{ cm}^{-1}$  is attributed. The  $1653\text{ cm}^{-1}$  broadband is assigned to the carbonyl group C=O and that of around  $2860\text{ cm}^{-1}$  is due to aliphatic CH stretching and the broad bands around  $3370\text{ cm}^{-1}$  correspond to O-H stretching in the pure PVP. This shows good arrangement with literature.

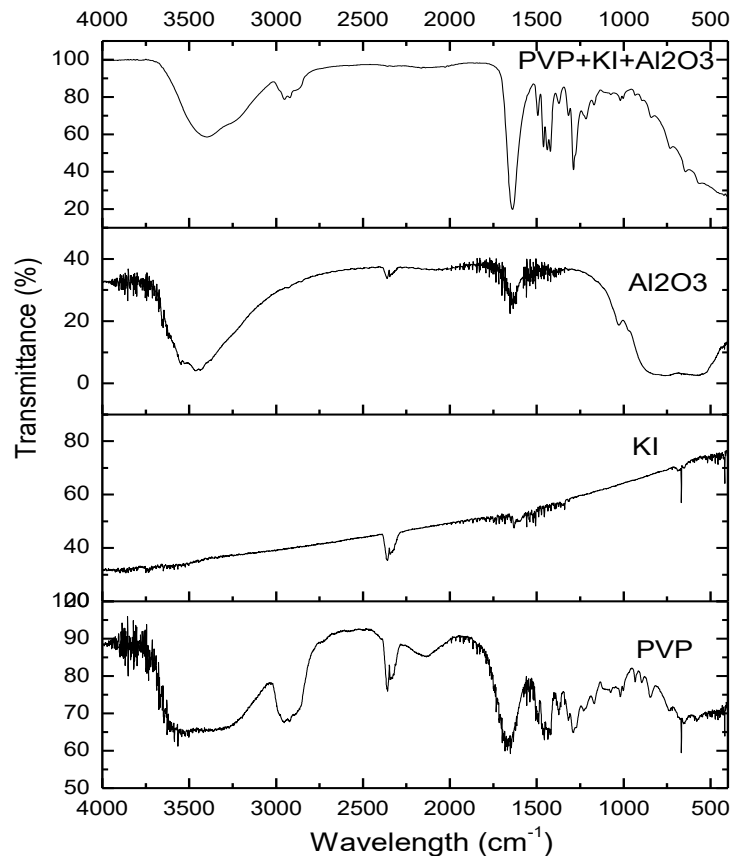


Figure 2b FTIR Spectra of PVP+KI+ Al<sub>2</sub>O<sub>3</sub> system

Figure 2b shows FTIR Spectra of PVP, KI, Al<sub>2</sub>O<sub>3</sub> and PVP+KI+Al<sub>2</sub>O<sub>3</sub> system. The broadening band at  $3800 - 3000\text{ cm}^{-1}$  occurs due to the hydrogen bond between the various hydroxyl groups. The stronger broadening band  $1000 - 400\text{ cm}^{-1}$  correspond to Al-O vibration existed in Al<sub>2</sub>O<sub>3</sub>. Since there is no change in peak position of the (PVP+KI), nor there is any new peak, so it is confirmed that the system is a composite (PVP+KI) + Al<sub>2</sub>O<sub>3</sub>.

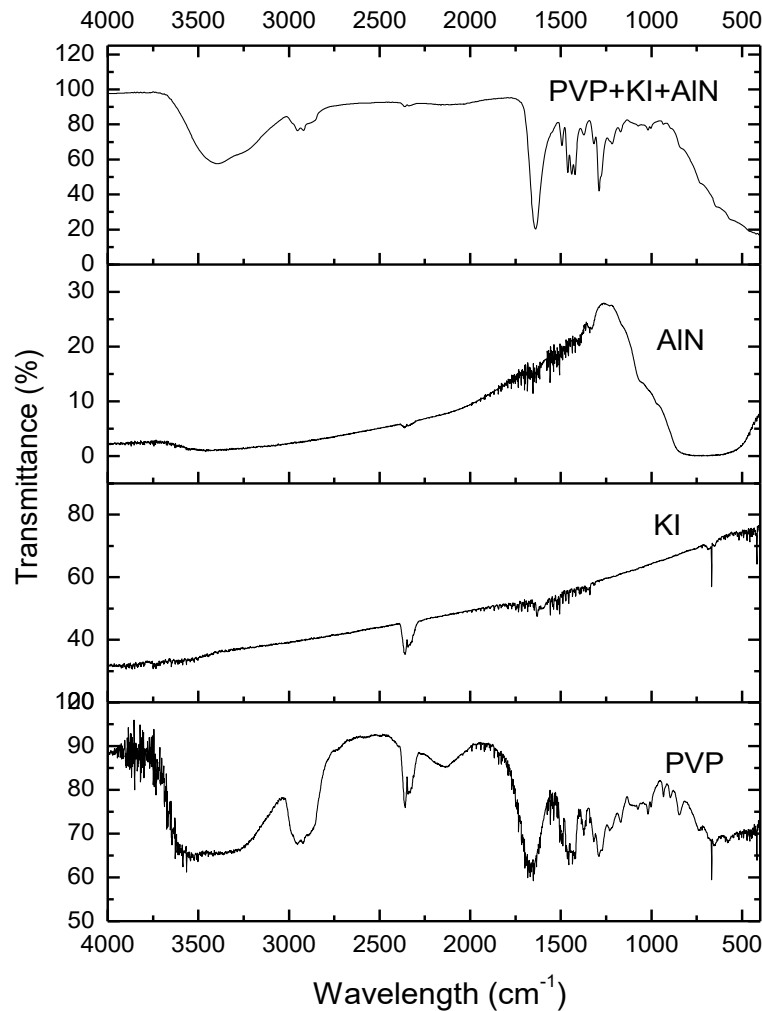


Figure 2c FTIR Spectra of PVP+KI+AlN system

Figure 2c shows FTIR Spectra of PVP, KI, AlN and PVP+KI+AlN system. In the FTIR broad band spectrum at 690  $\text{cm}^{-1}$  is characteristic of the aluminium nitride transverse optical mode. The adding KI is added in PVP forms a complex. The complex is due to a bond with  $\text{K}^+$  and PVP chain. The adding of AlN does not make any bond with the polymer. There is no change in peak position of the (PVP+KI), nor there is any new peak, so it is confirmed that the system is a composite (PVP+KI)+ AlN.

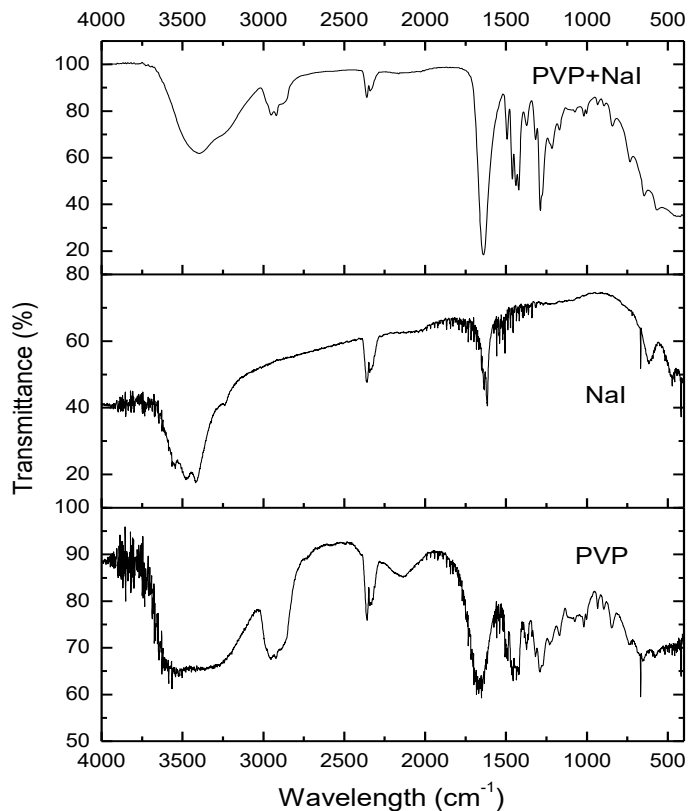


Figure 3a FTIR Spectra of PVP+NaI system

Figure 3a shows FTIR Spectra of PVP, NaI and PVP+NaI system. Broad spectrum is visible around 1000cm<sup>-1</sup> in NaI. The absorption is observed PVP+NaI complex around 3500cm<sup>-1</sup>. Figure 3b shows FTIR Spectra of PVP, NaI, ZnO and PVP+NaI+ZnO system. In FTIR Spectra the peak at 473.33 cm<sup>-1</sup> is the characteristic absorption peak of Zn–O bond and also authenticates presence of ZnO. The adding NaI is added in PVP forms a complex. The complex is due to a bond with Na<sup>+</sup> and PVP chain. Therefore, at least one peaks of PVP will shift and a new peak comes. The nano ZnO added does not make any bond with the polymer. There is no change in peak position of the PVP+NaI, nor there is any new peak, so it is confirmed that the system is a composite PVP+NaI+ZnO. Figure 3c shows FTIR Spectra of PVP , NaI , and PVP+NaI+BaTiO<sub>3</sub> system. The peaks at 3396 cm<sup>-1</sup> and 1586 cm<sup>-1</sup> may be attributed to OH group and stretching vibrations are at 3396, 1586, 1351 and 583 cm<sup>-1</sup>. The peak 1351 cm<sup>-1</sup> may be assigned to carbonate group whereas peak at 583 cm<sup>-1</sup> be attributed to BaTiO<sub>3</sub>. When BaTiO<sub>3</sub> is added, it does not make any bond with the polymer nanocomposite. Since there is no change in peak position of the PVP+NaI, nor there is any new peak, so it is confirmed that the system is a composite PVP+NaI+ BaTiO<sub>3</sub>.



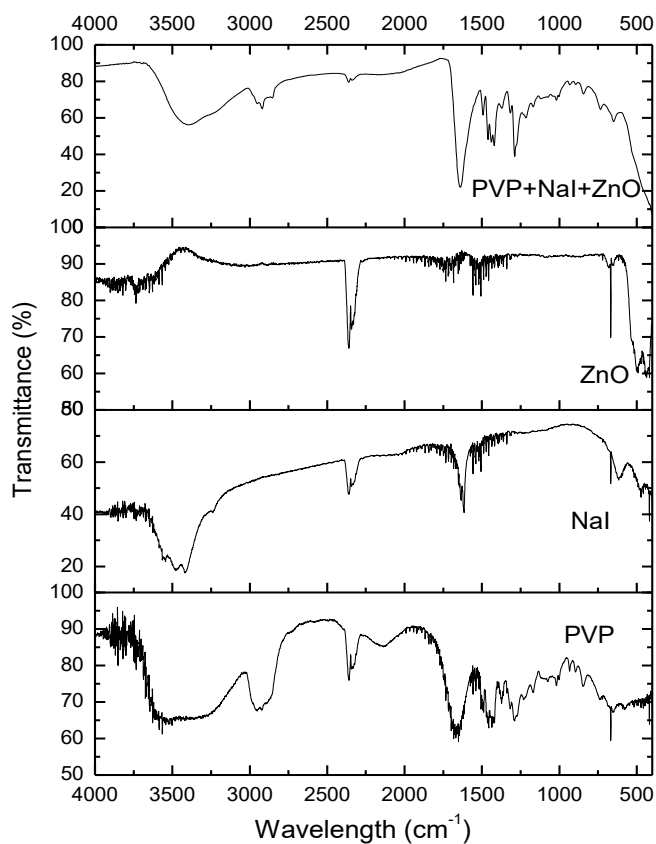


Figure 3b FTIR Spectra of PVP+NaI+ZnO system

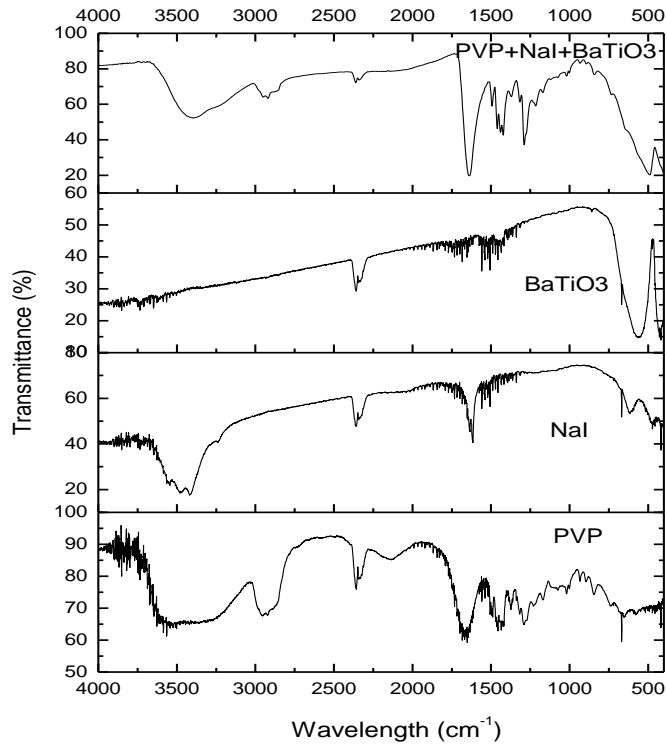


Figure 3c FTIR Spectra of PVP+NaI+BaTiO<sub>3</sub> system

**FTIR Spectra of PVP+KOH and PVP+KOH+NiO system**

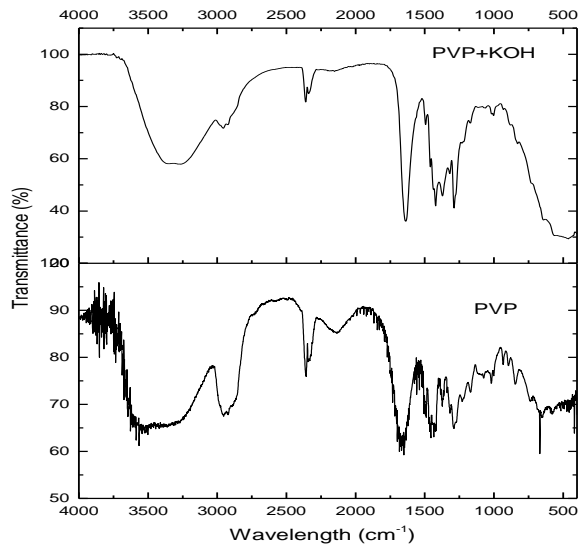


Figure 4a FTIR Spectra of PVP+KOH system

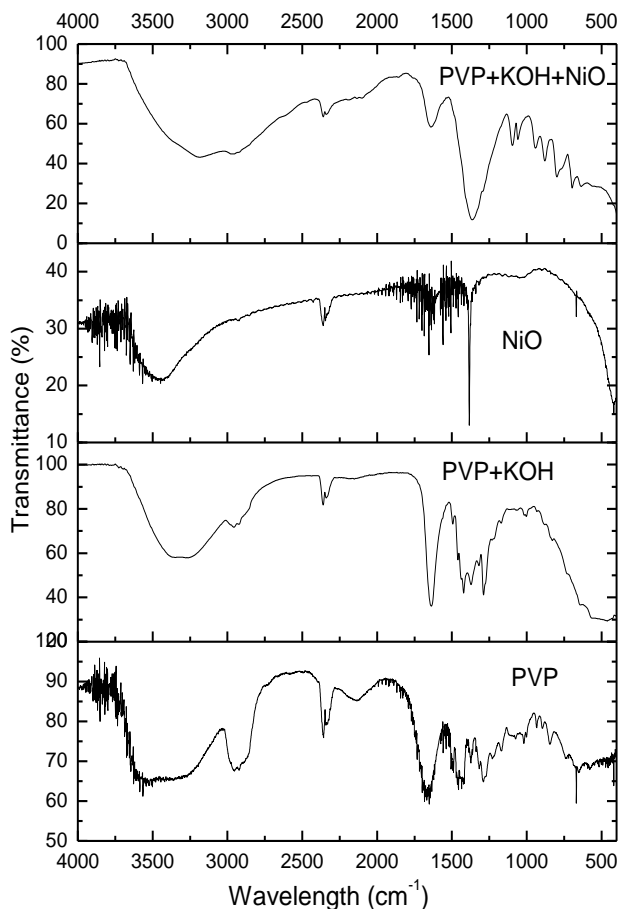


Figure 4b FTIR Spectra of PVP+KOH+NiO system

Figure 4a shows FTIR Spectra of PVP, KOH and PVP+KOH system. The absorb peak is observed at  $2400\text{cm}^{-1}$  both in complex as well as in PVP. Figure 4b shows FTIR Spectra of PVP, KOH and PVP+KOH+NiO system. The broad band in the region of  $600\text{--}700\text{cm}^{-1}$  is observed. The broad band at  $3440\text{cm}^{-1}$  is present in the complex. The added KOH does not make any bond with the polymer. Since there is no change in peak position of the (PVP+ KOH), nor there is any new peak, so it is confirmed that the system is a composite (PVP+ KOH)+NiO.

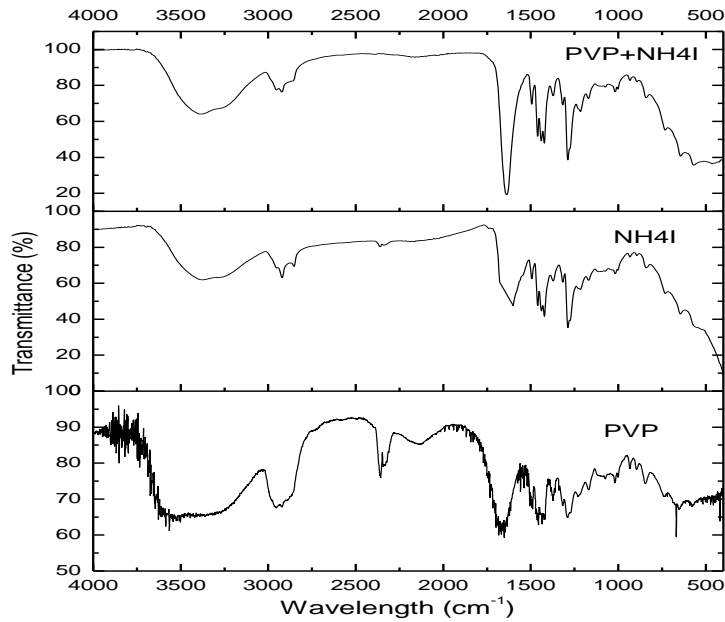


Figure 5a FTIR Spectra of PVP+ NH<sub>4</sub>I system

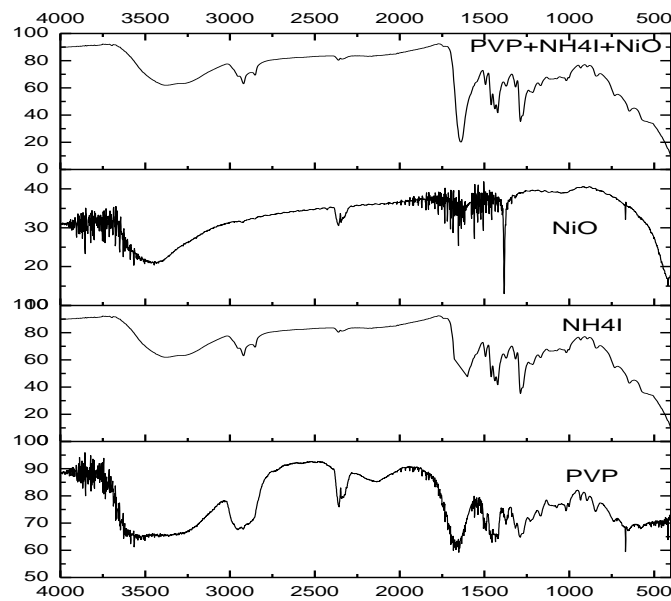


Figure 5b FTIR Spectra of PVP+NH<sub>4</sub>I+NiO system

Figure 5a shows FTIR Spectra of PVP, NH<sub>4</sub>I and PVP+NH<sub>4</sub>I system. The peaks corresponding to NH<sub>4</sub>I between 1200 to 1700 cm<sup>-1</sup> are observed in complex. Figure 5b shows FTIR Spectra of PVP, NH<sub>4</sub>I, NiO and PVP+NH<sub>4</sub>I+NiO system. The broad band in the region of 600–700 cm<sup>-1</sup> is assigned to Ni–O stretching vibration mode; the broadness of the band indicates that the NiO powders are nanocrystals. The broad band centered at 3440 cm<sup>-1</sup> is attributable to the band O–H stretching vibrations and the weak band near 1635 cm<sup>-1</sup> is assigned to H–O–H bending vibrations mode were also presented due to the adsorption of water in air while preparing the specimen. Since there is no change in peak position of the (PVP+ NH<sub>4</sub>I) nor there is any new peak, so it is confirmed that the system is a composite (PVP+ NH<sub>4</sub>I)+NiO.

## CONCLUSIONS

When salt is added in PVP polymer, a complex is formed. The complex is due to a bond with salt ion and PVP chain. Therefore, at least one peak of PVP will shift and new peaks may appear. After adding nanoparticle, it does not make any bond with the polymer. Since there is no change in peak position of the (PVP+Salt) composite, nor there is any new peak. So it is confirmed that the system is a polymer nano composite (PVP+Salt+nano particles). The FTIR has been used for Identifying Polymer composition, Additives, Organic contaminants and unknown material if any. FTIR Spectra of various composition of polymer nanocomposite has been determined and discussed its features.

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