

## Structural and Infrared Spectroscopy of Polyvinylpyrrolidone/Multi-walled Carbon Nanotubes Nanocomposite

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### Abstract

In this work, polyvinylpyrrolidone (PVP)/ Multi-walled carbon nanotubes (MWCNTs) nanocomposites were prepared with two concentrations of MWCNTs by casting method. Morphological, structural characteristics and electrical properties were investigated. The state of MWCNTs dispersion in a PVP matrix was indicated by Field Effect-Scanning Electron Microscopy (FESEM) which showed a uniform dispersion of MWCNTs within the PVP matrix. X-ray Diffraction (XRD) indicate strong bonding of carbonyl groups of PVP composite chains with MWCNTs. Fourier transfer infrared (FTIR) studies shows characteristics of various stretching and bending vibration bands, as well as shifts in some band locations and intensity changes in others. Hall effect was studied to test the type of charge carriers which was shown to be P-type. The electrical conductivity was shown increased for the pure PVP and pure MWCNT from  $(2.047 \times 10^{-5}) (\Omega \cdot \text{cm})^{-1}$  and  $(3.683 \times 10^1) (\Omega \cdot \text{cm})^{-1}$  to  $(2.51 \times 10^2$  and  $2.36 \times 10^2) (\Omega \cdot \text{cm})^{-1}$  for both concentrations of nanocomposites, which indicate the conductivity was enhancement by using the carbon nanotubes.

### Article info.

#### Keywords:

*polyvinylpyrrolidone, Multi-walled carbon, nanotubes, structural properties, Hall Effect.*

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### 1. Introduction

Carbon nanotubes (CNTs) have captivated the interest of researchers and scientists since Ijiman discovered them due to their extraordinary optical, electrical, thermal, and mechanical properties. They have a wide range of applications such as supercapacitors [1], transistors [2], Schottky contacts [3], solar cells [4], gas sensors [5].

Polymer composites are most attractive to be used with carbon nanotubes since the result has many interesting properties [6,7]. However, it is so difficult to get a homogeneous solution of polymer and CNTs. Polyvinylpyrrolidone (PVP) is soluble in water and in many polar solvents such as ethanol which make it easy to be synthesized [8, 9]. PVP is good in a stable environment, of moderate electrical conductivity, high dielectric strength and with good charge storage capacity. Many studies have investigated the effect of MWCNTs system on the polymeric composites. The results show an enhancement in the mechanical, electrical, thermal and optical properties [10-12], and has many important applications in growing pharmaceutical, biomedical, electronic and optical applications [11, 12].

In this work, casting method was used to prepare the PVP/MWCNTs nanocomposite with two different concentrations of MWCNTs: 0.002wt% and 0.005wt%. FESEM, XRD and FTIR were employed to study its structural properties. Also, the Hall Effect was investigated.

## 2. Experimental work

### 2.1. Materials

Polyvinylepyrrolidone (PVP-K30) was supplied by central drug house LTD, with an average molecular weight  $M_w=40000$ g/mol. Multi-walled carbon nanotubes (MWCNTs) have outer diameter 13-18nm with length  $\sim 1-12$   $\mu\text{m}$ , and purity  $> 99$  wt. % were purchased from Neutrino.

### 2.2 Samples preparation

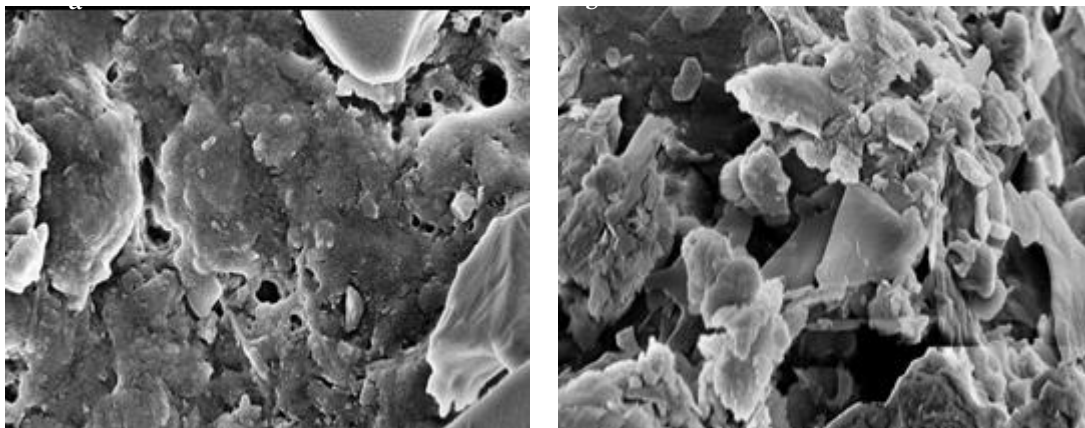
The casting solution method was used in this work. First, 4mg/ml PVP was dissolved in 50 ml of ethanol and mixed with a magnetic stirrer for 30min. then the solution was sonicated for 30 min. Meanwhile, two concentrations of MWCNTs were dispersed in 50 ml of ethanol stirred for about 24 hours at room temperature. The PVP solution and the MWCNTs were mixed together with a magnetic stirrer for about 48 hours. The last step, the mixtures were kept in the ultrasonic bath for 2 hours in order to obtain homogenous solutions.

### 2.3 Characterization

Field Effect-Scanning Electron Microscope (FESEM) images were obtained with a field emission scanning electron microscope (Quanta FEG-250 SEM) operating at an acceleration voltage of 3 kV. The X-ray diffraction patterns were studied using Shimadzu model XRD-6000, the Bragg's angle range of  $5^\circ \leq 2\theta \leq 60^\circ$  and Cu K $\alpha$  radiation of wavelength  $\lambda = 1.5406$  Å with scanning speed of  $5^\circ/\text{min}$ . Shimadzu 8400 FTIR spectroscope was used to record the FTIR spectra of the fabricated composite films in the wave number range  $400-4000$   $\text{cm}^{-1}$  with a resolution of  $4$   $\text{cm}^{-1}$ .

## 3. Results and discussion

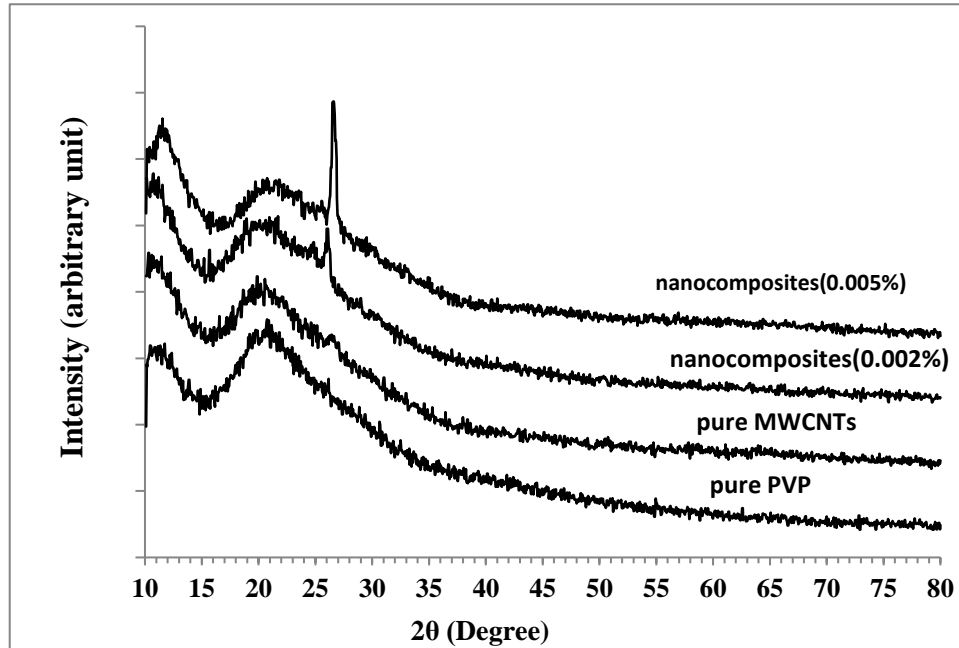
The FESEM was used to study the surface morphology of pure PVP, pure MWCNTs and its nanocomposites. The results showed a uniform dispersion of MWCNTs within the PVP matrix, as shows in Fig.1. This can be attributed to the good compatibility between the MWCNTs and the polymer chains of PVP. Although at the high concentration of the MWCNTs(0.005wt.%), an aggregation and local irregularities were observed which can be attributed to the MWCNTs nature.



**Figure 1: FESEM images for: a) 0.002wt% and b) 0.005wt% PVP/MWCNT nanocomposite.**

The molecular interaction between PVP and MWCNTs in the nanocomposites was characterized by the XRD technique. Fig.2 shows the XRD patterns of pure PVP,

pure MWCNTs and their nanocomposites. For pure PVP, the mean peaks were located at  $2\theta=11, 21$  it shows very broad diffraction peaks. These peaks indicate the amorphous nature of the polymer. This result agrees with that of Zidan et al. [13]. For pure MWCNTs, its peaks are at  $2\theta= 26.15$  corresponding to (020) crystal plane. This result agrees with that of Abdullah et al. [14]. For the nanocomposites, the peak intensity at  $2\theta=26.15$  becomes sharp and of higher intensity. This indicates that the interaction between MWCNTs and PVP leads to an increase in the inter-molecular interactions between the blend chains [15]. Because of its amorphous form, it has a higher ionic diffusivity, resulting in enhanced ionic conductivity.



**Figure2: XRD patterns of all samples for pure PVP, pure MWCNTs and their nanocomposites**

The purpose of FTIR spectroscopy is to examine multi-component functional groups to provide helpful information about the mechanism of interaction and to measure the content of the material phase in a variety of bond types found in all samples. Fig.3 shows the FTIR spectra of the pure PVP, pure MWCNTs and their nanocomposites spectra, they show characteristics of various stretching and bending vibration bands, as well as shifts in some band locations and intensity changes in others. The peaks at:  $3377.61\text{ cm}^{-1}$  corresponds to OH stretching;  $2954.95\text{ cm}^{-1}$  corresponds to C-H stretching;  $1514.12\text{ cm}^{-1}$  corresponds to characteristic vibration of C=N (pyridine ring):  $1514.12\text{ cm}^{-1}$  and  $1224.80\text{ cm}^{-1}$  represent the characteristic vibration of C=N (pyridine ring) and C-C stretching and/or  $\text{CH}_2$  deformation, respectively. The results are in good agreement with that of Alibe et al. [11]. FTIR spectrum for the PVP/MWCNT nanocomposites show that the intensity of the peaks has decreased due to the existence of MWCNTs. The broad peak at  $3477\text{ cm}^{-1}$  is due to OH stretching vibration of the hydroxyl groups. Also, the peak at  $1654.92\text{ cm}^{-1}$  has decreased in intensity and became broader. This exhibits the structural changes of PVP after being filled with MWCNTs. The shift of wave number indicates the change in potential energy distribution along the polymeric chain. These results are in good agreement with those of Zidan et al. [13].

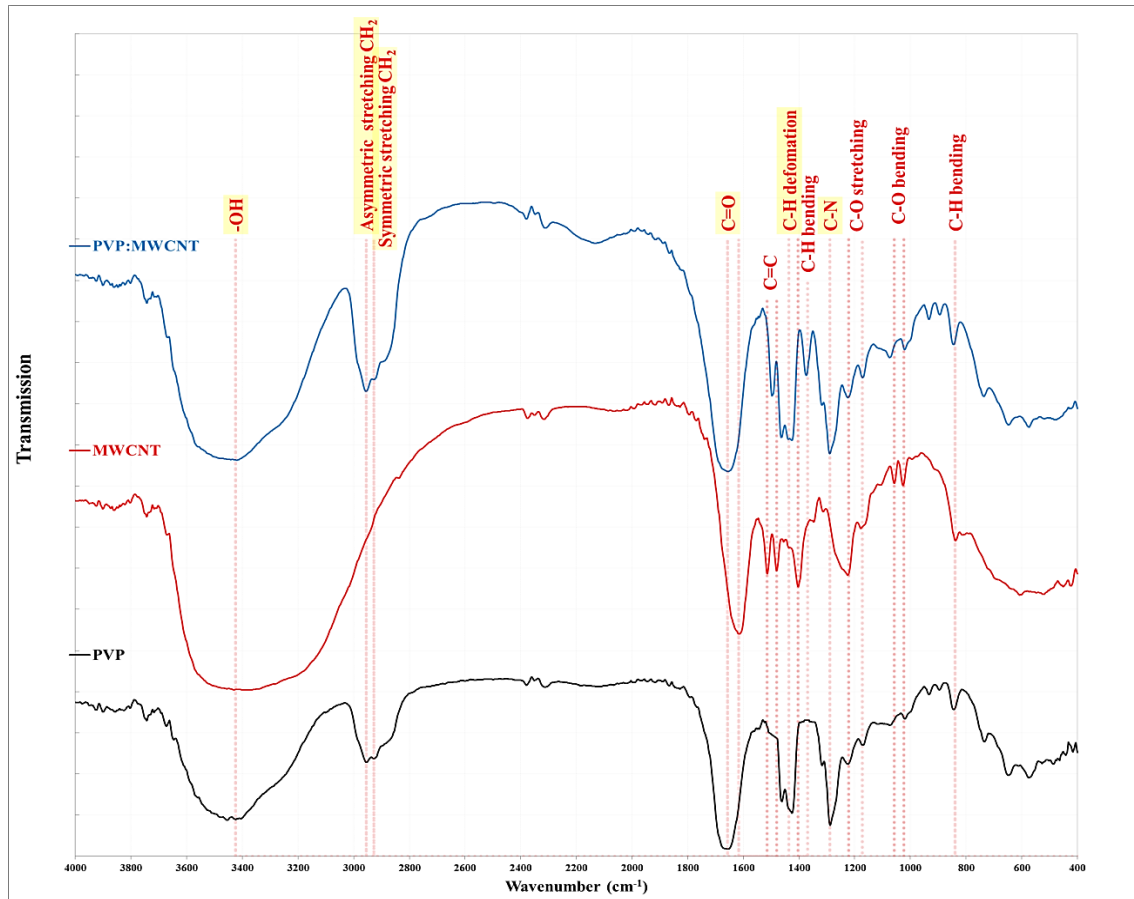


Figure 3: FTIR spectra of pure (PVP, MWCNTs) and their nanocomposite samples.

The Hall measurements of pure PVP, pure MWCNTs and PVP/MWCNTs nanocomposites were studied as shown in Table 1. The electrical conductivity and carrier mobility were observed to be higher in PVP/MWCNTs nanocomposites as compared with pure samples, which indicates the carrier type was found to be positive for all samples which indicates that holes are the majority charge carriers so the samples are p-type semiconductor. The conductivity and mobility were increased for the two composites by comparing with pure PVP and MWCNTs.

Table1: Hall Effect Parameters.

Samples	Charge Concentration [1/ cm <sup>3</sup> ]	Mobility [cm <sup>2</sup> /V.s]	Resistivity [Ω cm]	Average Hall Coefficient [cm <sup>3</sup> /C]	Conductivity (Ω cm) <sup>-1</sup>	Carrier type
PVP	7.381×10 <sup>13</sup>	1.731×10	4.886×10 <sup>4</sup>	8.457×10 <sup>4</sup>	2.047×10 <sup>-5</sup>	P
MWCNTs	1.264×10 <sup>16</sup>	2.753×10 <sup>3</sup>	1.79×10 <sup>-1</sup>	4.937×10 <sup>2</sup>	3.683×10 <sup>1</sup>	P
PVP+MWCNT (0.002%)	7.74×10 <sup>17</sup>	7.23×10 <sup>1</sup>	1.67×10 <sup>-1</sup>	8.065×10	8.97×10	P
PVP+MWCNT (0.005%)	1.11×10 <sup>17</sup>	2.41×10 <sup>1</sup>	1.23×10 <sup>-1</sup>	2.29×10 <sup>2</sup>	2.36×10 <sup>2</sup>	P

## 5. Conclusions

Ordinary casting techniques were used to create pure PVP, pure MWCNTs, and their nanocomposite samples. FESEM suggests that an entangled network of PVP-coated carbon nanotubes can be easily identified. The establishment of an intermolecular contact between the PVP polymer chain and MWCNTs was revealed by FTIR analysis. The nanocomposites had no notable peaks characterizing MWCNTs, according to the X-ray examination. The semicrystalline character of the examined system was also demonstrated. The Hall measurements indicated p-type carrier for all samples.

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## Conflict of interest

Authors declare that they have no conflict of interest.

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## الخواص التركيبية وظيف الإشعة تحت الحمراء للمركب النانوي بولي بيروالدين/انابيب الكربون متعددة الطبقات الجدران

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### الخلاصة

في هذا العمل، تم تحضير مركبات الانابيب الكربونية المتعددة الجدران (MWCNTs) / البولي فينيل بيروالدين (PVP) بتركيزين من الانابيب الكربونية المتعددة الجدران بطريقة الصب. تم التحقيق في حالة تشتت الانابيب الكربونية المتعددة الجدران في مصفوفة البولي فينيل بيروالدين بواسطة المجهر الإلكتروني لمسح تأثير المجال (FESEM) والذي أظهر تشتتاً موحداً للانابيب الكربونية المتعددة الجدران داخل مصفوفة البولي فينيل بيروالدين. يشير حيود الأشعة السينية (XRD) إلى الترابط القوي لمجموعات الكربونيل من سلاسل مركب البولي فينيل بيروالدين مع الانابيب الكربونية المتعددة الجدران. تُظهر دراسات مطيافية الأشعة تحت الحمراء باستخدام تحويلات فورييه (FTIR) خصائص نطاقات الاهتزاز المختلفة للتمدد والانحناء، فضلاً عن التحولات في بعض مواقع النطاقات وتغيرات الشدة في مناطق أخرى. تمت دراسة تأثير هول لاختبار نوع الموصلية التي تبين أنها من النوع P. تمت زيادة الموصلية لـ البولي فينيل بيروالدين النقي و الانابيب الكربونية المتعددة الجدران النقي من  $(2.047 \times 10^{-5})$  (اوم.سم)<sup>-1</sup> و  $(3.683 \times 10^1)$  (اوم.سم)<sup>-1</sup> إلى  $(2.51 \times 10^2)$  و  $(2.36 \times 10^2)$  (اوم.سم)<sup>-1</sup> لكلا تراكيز المركبات النانوية، مما يشير إلى التحسين الذي تم إجراؤه للتوصيلية.