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Improvement of the physical properties of poly vinyl alcohol nanofibers by adding carbon nanotubes

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Abstract

In this study, nano textile fibers were prepared from poly vinyl alcohol (PVA) and multi wall carbon nano tubes (MWCNTs) via electro spinning method. Different proportions of the multi wall carbon nano tubes were used, including 2, 4, and 6 % weight percent. The optical, electrical, and thermal properties were studied. Scanning electron microscopy was used to study the morphology of samples. Fourier-transform infrared spectroscopy (FTIR) analysis was used to study the bonds of the prepared samples. Differential scanning calorimetry (DSC) was used to investigate the thermal properties including the glass transition temperature and melting the point. Ultraviolet spectroscopy was used to study the optical properties. The results showed that the addition of carbon nano tubes in small proportions of 6% wt leads to an improvement in the thermal properties represented by increasing the glass transition temperature value from 85.17 o C of poly vinyl alcohol to reach 107 ^{o}C with a strengthening percentage of 6 weight percent. The electrical conductivity improves with the addition of 6 weight percent multi wall carbon nanotubes from 14.28 $\Omega^{-1}cm^{-1}$ to 20 $\Omega^{-1}.\ cm^{-1}$. The results of the UV spectrum indicate that the inclusion of multi wall carbon nano tubes control the energy band gap. Absorbance and coefficient of absorbance also increase with increasing the carbon nano tubes contents. The best sample had a band energy gap of roughly 1.75 eV which consisted of poly vinyl alcohol with 6 weight percent of multi wall carbon nano tubes.

Keywords: CNTs, Nano composites, PVA, Thin films, UV-spectrum.

Introduction

Modern technological advances depend heavily on polymers. Polymers play a crucial role in creating nanocomposites that are lightweight and simple to form . Although polymer is an insulating material, one of the processing techniques that can change it into a semiconductor is doping and nanocomposite [1,2] . Two phases make up a material called a polymeric nano composite: the matrix phase is a substance with a macromolecule size of polymer , and the reinforcing phase is a material with a dimension of less than 100 nm [3] . Many Nano shapes can be used as reinforcement materials as : nano sphere filler like ZnO and Al_2O_3 nanoparticles [4] , carbon nano tubes [5], nanofibers [6], and natural pigments [7]. There are many applications that are dependent on the electrical as dielectric constant and photoelectrical properties of Nano composites [8]. Considering solar energy is one of the more interesting sources of long-term energy, and is important in the field of renewable energy. Organic nanocomposites solar cells are one type of electronic device that prepared by organic substances such as polymer materials and organic reinforcement materials as carbon nanotubes [9]. With a number of its special qualities, such as a large surface area, high stability, excellent thermal and electrical conductivity, a high anti-static feature, and hydrophobicity, make carbon nano tubes are a very significant substance that is employed as a reinforcement phase for photovoltaics applications[10,11] . On the other hand, there are many techniques are used to prepare thin film nanocomposites such as hand casting, spin coating , and electrospinning techniques , the last method is the best for preparing the homogenous , controlling and uniform thin films as a nanofibers form [12] . Many previous studies performed about the use of carbon nanotubes with polymeric substance for thermos physical applications and solar cell and photovoltaic applications as: Mansoor Farbod et al[13]. published a paper about thermo characteristics of paraffin reinforced with carbon nanotubes, through their study a nanocomposite of paraffin and carbon nanotubes (Pa/CNTs) was created with various CNT lengths to study how the thermos physical characteristics of paraffin phase change . As a result of this study , sulphuric and nitric acids were used to shorten CNTs before further processing. The test findings demonstrated that the weight percentage of carbon nanotubes (CNTs) enhanced the thermal conductivity of the composites. The composite containing 1.2 weight percent of solid paraffin and one hour of refluxed CNTs had the highest rise, at 26.92%. Further, when the weight percentage of CNTs increased, the specific heat decreased and the latent heat of melting and solidification of the composite increased. With a concentration of 0.6 weight percent and an hourly refluxed carbon nanotube (CNT) content, the composite exhibited the highest absorption capacity of paraffin by graphene aerogel, showing a 36.11% increase over pure paraffin. Furthermore, the GA/Pa/CNTs structure's outstanding form stability was found during the leakage test . Flayieh . et al. published on a paper about using the CNTs with polystyrene nanofibers for preparing the solar cell application. They prepared three groups of nanofiber samples using the electrospinning technique, with each group having a different PS concentration (12, 14, and 16 w/v). Four samples containing (PS/MWCNT) elements were produced by adding multi walled carbon nanotubes to each group in one of four ratios. To determine how evenly dispersed nanofibers and multi-walled carbon nanotubes were within polymeric nanofibers, as well as their impact on the final nanofiber's attributes through the absorption peaks, a UV-Visible test was conducted. SEM was used to study the morphological of samples. The outcomes demonstrated that CNTS has a dominant influence on produced nanofibers in the (12 wt.% Polystyrene) group, and both (PS, MWCNT) have a shared effect, greatest stable absorption curves, and uniformly scattered radiation at (14 wt.% PS) group, and random results at (16 wt.% PS). According to FESEM results, beads are more likely to form at groups (12,16 weight percent PS) and when polymer concentration rises, the diameter of nanofibers also increases. The inclusion of CNT lowers the nanofibers' diameter even further, improves their orientation, and boosts their UV absorption as well as their strength and mechanical capabilities in the group of (14 wt.%) Polystyrene . Utilizing polyvinyl alcohol, a water-soluble polymer, and carbon nanotubes for high sensitivity solar cell application, the current work intends to propose a technique to produce solar cells utilizing affordable, environmentally benign, and locally accessible materials . CNTs, as one-dimensional materials, have garnered significant interest in the photovoltaic field due to their exceptional optical and electrical characteristics, together with their consistent chemical and mechanical stabilities. In particular, adding carbon nanotubes (CNTs) to perovskite solar cells has significantly increased the devices' flexibilities, stabilities, and efficiencies[14].

The energy gap of polymers, like polyvinyl alcohol, is roughly 5 eV. By enhancing the material's crystallization and transforming it into nanofibers, the energy gap of polyvinyl alcohol can be decreased to about 3 eV [15] . Carbon nanotubes can be used to form extra levels that serve as crossing points for electrons moving from the valence band to the conduction band, which is necessary for electro-voltaic applications to reduce the energy gap to smaller ranges [16] . Additionally, they serve as centres for the absorption of UV rays, which boosts the effectiveness of electro-voltaic systems [17].

Materials and Methods

The solvent used to prepare the PVA solution is formic acid. The poly vinyl alcohol (PVA) with the chemical formula [C2H4]n. It is a white powder with a hydrolysis rate of 88 mol/mol, a molecular weight (Mw) in the range of 25000 to 40000 g/mol, and a purity of 0.92%. Multi walled Carbon nanotubes with a diameter of 25 nm, and 99.99% purity were used as reinforcing materials, The materials used in this study were employed from Sigma-Aldrich ™. The percentage and contents of the experimental samples used in this study are displayed in Table 1.

The dispersion process was performed under 50 degrees Celsius for 15 minutes for each sample using a 100 ml beaker containing the weight percentages added separately to the dispersion liquid (formic acid). The beaker was then put within the chamber of the direct ultrasonography device. In the ratios indicated in Table 1, dispersed solutions were formed to dissolve polyvinyl alcohol. Electrospinning system was used to prepare the nanofibers of pure PVA and PVA reinforced with CNTs under processing parameters including : (20 kV applied voltage , 15 cm of electrospinning distance, 0.5 ml/hr of flow rate , and rotating cylinder collector with 360 rpm) . Table 2. shows the solution parameters that were mustered and utilized in this study. Fig. 1. Shows the graphical abstract of electro spun nanofibers.

Table 1. contents of nanocomposites samples

No. of samples	Contents
NC0	PVA nanofibers
NC1	PVA + 2% wt CNTs
NC2	PVA + 4% wt CNTs
NC3	PVA+6% wt CNTs

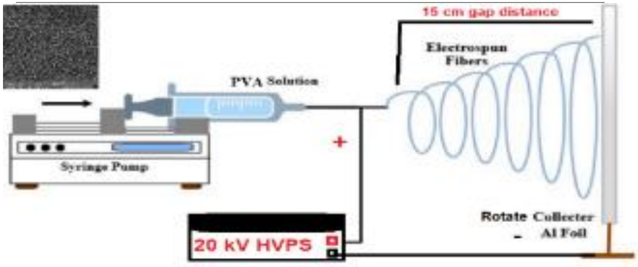


Figure 1. Set up of by electro spinning system for Nano fibers preparation

The greatest concentration, 0.06 weight %, was utilized in this investigation since higher concentrations caused the resultant samples to aggregate and the tests to fail because they were unable to produce homogeneous samples when dispersed.

Results and Discussions

Solution parameters

The relationship between the concentrations of CNTs with the viscosity and electrical conductivity of solutions is displayed in Table 2 and Figs. 2and 3 respectively. Fig. 2 demonstrates that as CNTs concentration was increased, the viscosity of polymeric solutions decreased due to a decrease in the amount of polymer in place. Additionally, CNTs act as a lubricant, which increases the slipping of polymeric Nano fluid solution layers and lowers flow resistance, both of which contribute to a decrease in the viscosity of solutions.

Table 2. PVA/formic acid solution properties

No. of samples	Contents of solutions	Viscosity Cp	Electrical conductivity mS/cm
NC0	PVA	510	0.9
NC1	PVA + 2% wt CNTs	380	3.2
NC2	PVA + 4% wt CNTs	240	4.6
NC3	PVA+6% wt CNTs	170	5.3

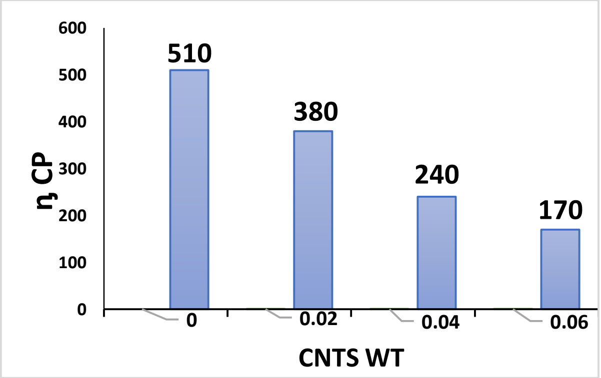


Figure 2. Viscosity of PVA and PVA/CNTs Nanofluid solutions

Fig. 3 shows that the electric conductivity of the PVA solution increased as more CNTs were added. More conductive pathways in PVA were created as the weight ratios of MWCNT were increased. As a result, charge carriers were more mobile and denser, increasing EC. As shown in Fig. 2, the EC for a pure PVA solution was 0.9 mS/cm and increased to 5.3 mS/cm for PVA with 6wt% MWCNT. These findings support earlier research [18] .

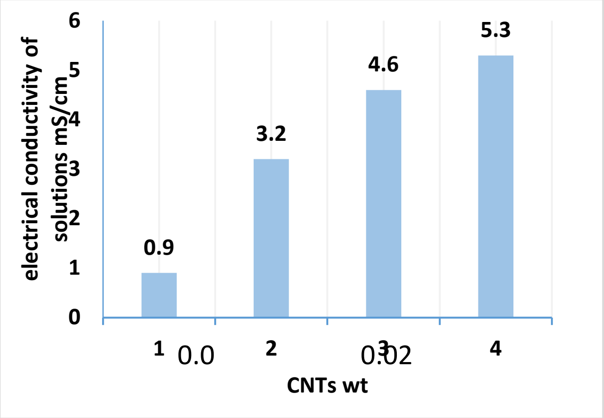
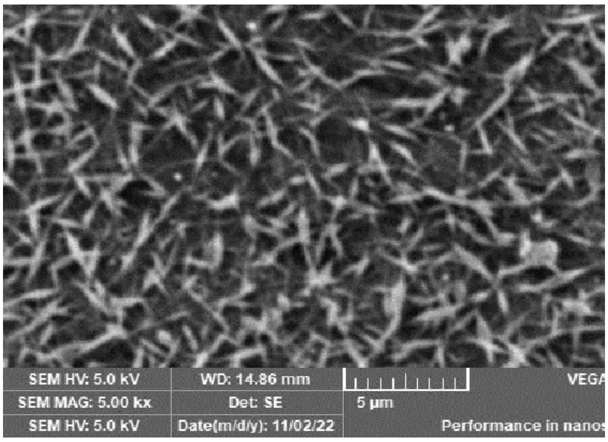


Figure.3. Electrical conductivity of PVA and PVA/CNTs Nanofluid solutions

SEM Images of PVA and PVA/CNTs nanofibers

Figs. 4 a-d show the SEM images of PVA and PVA/CNTs nanofibers



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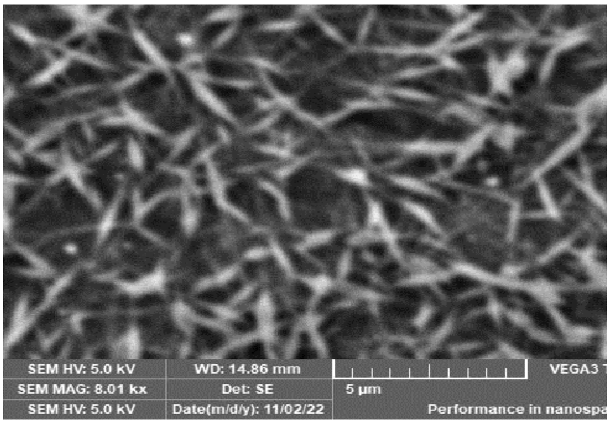

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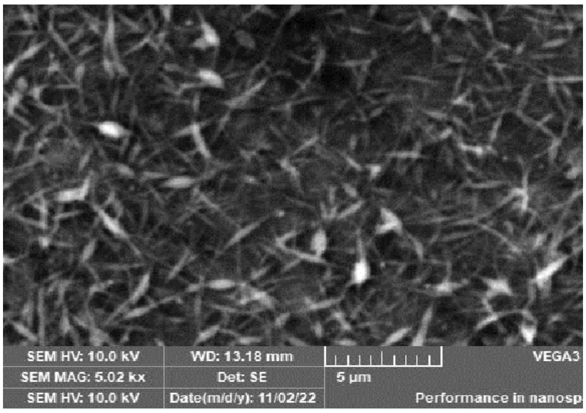

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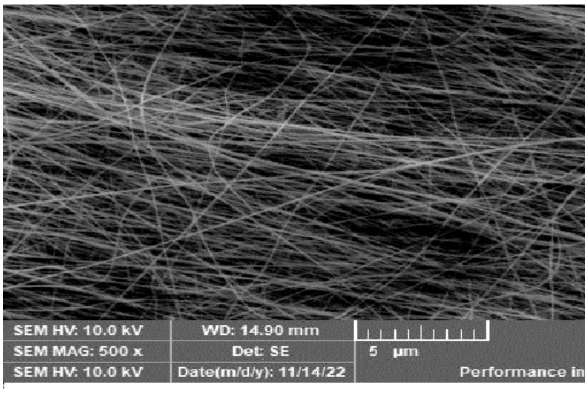


d

Figure 4 a-d. SEM images of a. PVA b. PVA+2% CNTs c. PVA+4% CNTs d.PVA+6% CNTs

The nanofibers diameter of PVA was between 235 - 400 nm, but it decreased to range between 211 - 350 nm with adding 2% wt. of CNTs , as well as, it continued to decrease at range about 150-200 nm with adding 4% wt. of CNTs. The best diameter of PVA + CNTs was around 150-200 nm, because CNTs enhance electrical conductivity and reduce viscosity, the diameter of nanofibers decreasing as their CNT ratios increase [18-19]

Electrical Conductivity of the Nanofibers

Fig. 5. and table 4, show the electrical conductivity of nanofibers samples . Note that the pure PVA sample, which is missing any conductive fillers, has a poor electrical conductivity according to fig.5 and table 4. There is a noticeable improvement in electrical conductivity in the NC1, NC2 and NC3 samples, which have higher CNT content. This is so because highly conductive CNTs are able can added to the PVA matrix to form a conductive medium within the composites. Electrical conductivity is

slightly decreasing in the NC4 sample as the amount of CNTs increases; this may be a result of the CNTs aggregating together in high concentrations. This is matched with Mora et. al. 2020 [20] . The NC3 sample, which had a 6% wt carbon nanotube concentration, had the ideal test results with an electrical conductivity of about 20 ohm^{-1} .cm^{-1}.

Table 4. Electrical properties of samples

No.	Sample contents PVA con%+ CNTs WT%	Resistivity Ω. Cm	Conductivity Ω^{-1}. cm^{-1}
NC0	PVA+00	0.07	14.28
NC1	PVA+2%	0.06	16.66
NC2	PVA+4%	0.055	18.8
NC3	PVA+6%	0.05	20

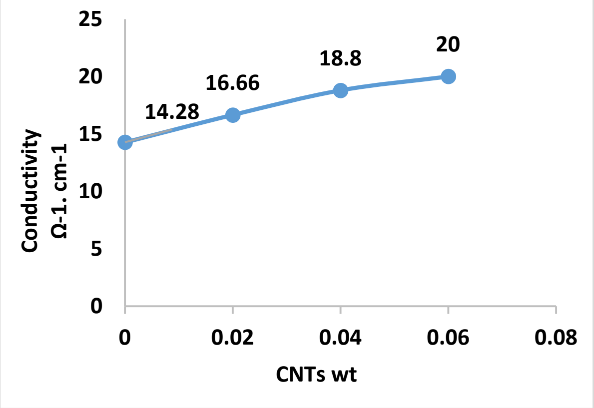


Figure 5. the relationship between the conductivity of nanofibers and CNTs wt.

FTIR Analysis

Table 5. and Fig 6. Show the FTIR spectra of PVA nanofibers and PVA – reinforced with MWCNTs.

Table 5. FTIR analysis of nanocomposites bonds

Wave no. cm^{-1}	Bonds	Groups
2800-3200	O-H	Hydrocele
200,2400-2600	CH_2 , CH stretching	Ester
1717	C=O & MWCNT-COOH	Ester
1020,1100,1250	CO stretching	

Fig. 6 illustrates that the characteristic peak of the PVA's hydrogen-bonded hydroxyl group can be seen in all spectra in the 2800-3200 cm^{-1} area as a broad, prominent peak. There are peaks at 1020, 1100, and 2000 cm^{-1} that is related to the C-O stretching modes of the ester groups were created, this is matched with Ruwaidah et.al [21] , and the strong sharp peak at 1620 cm^{-1} is the result of this stretching . The FTIR spectra of CNT-PVA display these peaks, which indicate that an esterification reaction occurred between the hydroxyl groups of the PVA macromolecule and the carboxyl groups of oxidized CNTs. Peaks at 2600, 2400, and 2000 cm^{-1} attributed to C-H stretching modes of the PVA backbone define CH2 groups. These peaks can be found in both spectra.

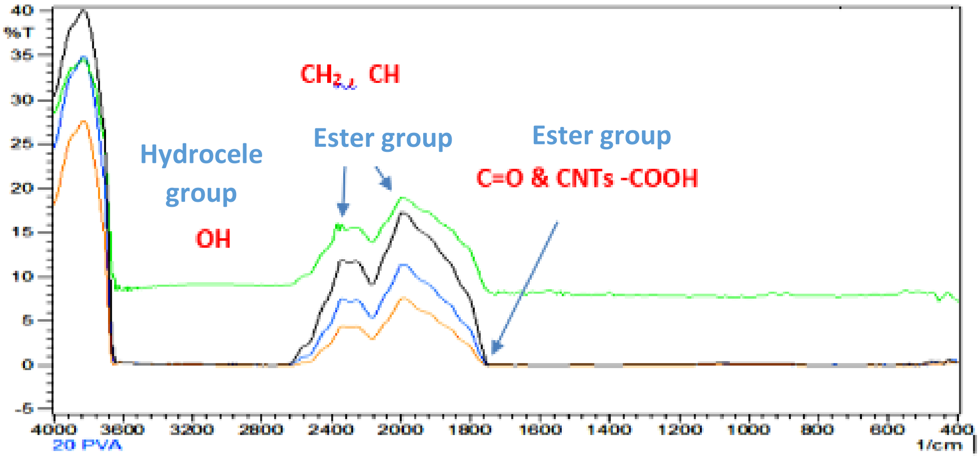


Figure 6. FTIR analysis of PVA and it's nanocomposites

In the spectra of the PVA-grafted CNTs, a signal that is less strong at 1717 cm^{-1} represents the carboxyl C-O group of the additional CNT-COOH traces. The meta-disubstitution process has affected the majority of benzene rings in carbon nanotubes, as indicated by double peaks in the 840-900 cm^{-1} and 750-840 cm^{-1} ranges. This corresponds to Malikov et al.[22].

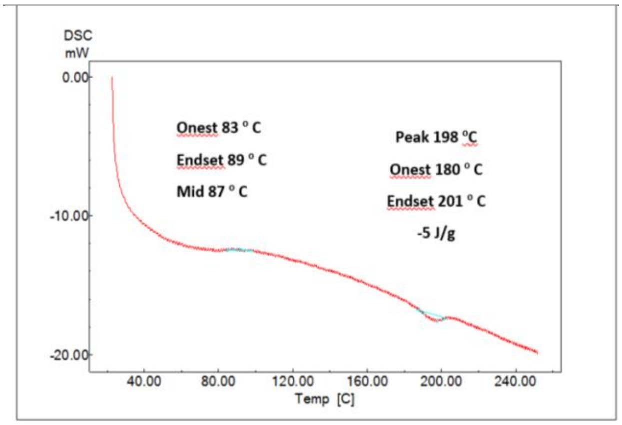
Thermal Properties by DSC

Fig. 7 a-d and table 6. Show the thermal analysis of

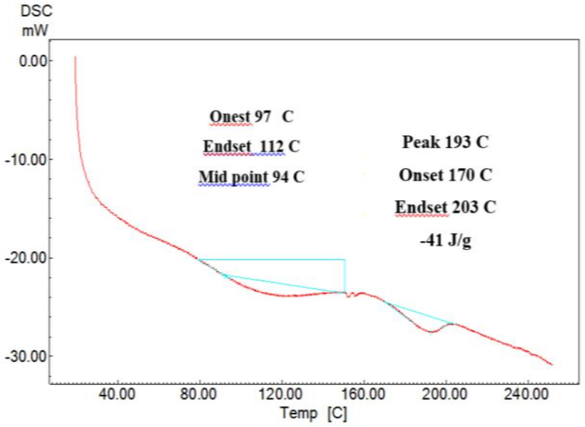
PVA and it's nanocomposites As seen in Fig. 7 a–d , the addition of CNTs to PVA can increase the Tg by reducing the movement of the polymer chains and increasing their stiffness. Because CNTs have a wide surface area, a high thermal conductivity, and are more effective at high temperatures, Tm decreased as CNT concentration rose. [23-24]

Table 6. thermal analysis of nanocomposites thin films

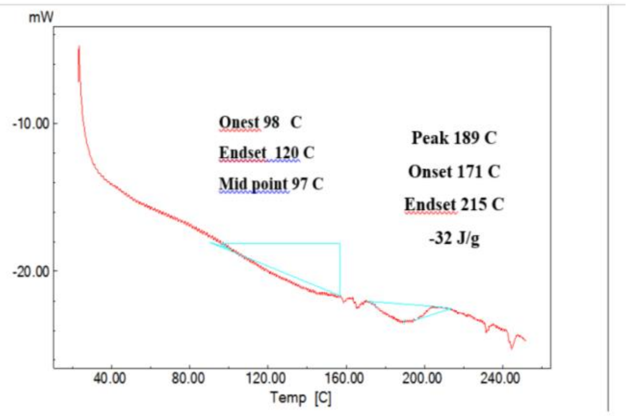
No.	Sample	Tg ºC	Tm ºC	Crystallinity -j/g
NC0	PVA NFs	87	198	**4**
NC1	PVA+2% wt CNTs	94	193	**41**
NC2	PVA+4% wt CNTs	97	189	**32**
NC3	PVA+6% wt CNTs	107	187	**33**



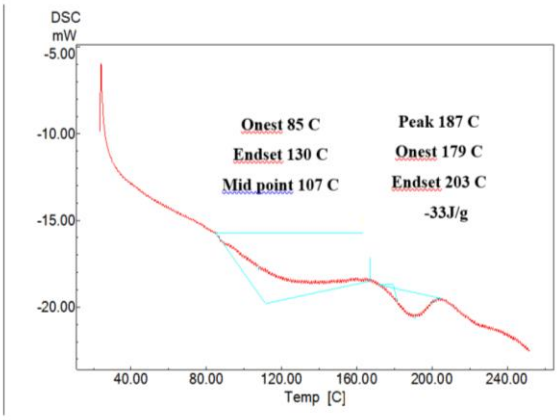
a. PVA



b. PVA+2% CNT



c. PVA+4% CNT



d. PVA+6% CNT

Figure 7.a-d thermal analysis of PVA Nano fibers and PVA +CNTs

The area under Tm with a J/g unit can be used to measure the crystallinity of the nanocomposite sample, however, as shown in Table 6 and Fig. e 7. The addition of CNTs increased the crystallinity ratio by 0.02 weight, from 5 J/g to 41 j/g. This is because low concentrations of CNTs can produce a high dispersion effect, which also results in a highly homogenous morphology of the sample. In addition, when the weight % of CNTs was increased to 0.04 and 0.06 wt, respectively, the crystallinity ratios reduced slightly from 41 J/g to 32 and 33 J/g. This could be because a small amount of agglomeration could occur with a greater quantity of CNTs. Fig. 7e shows the relationship between the CNTs ratios and the crystallinity of the nanocomposite samples. High optical properties can be obtained with low crystallinity ratios, as well as the high quantity of CNTs leads to more scattering light , low transmittance and high absorption intensity which leads to high efficiency in electro voltaic applications [25-26].

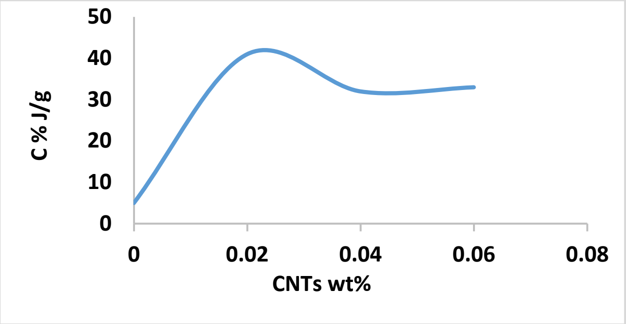


Figure 7e. the relationship between the CNTs ratios and the crystallinity of the nanocomposites sample

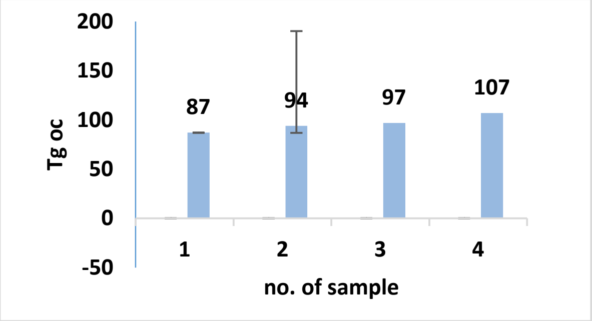


Figure 7f. Standard deviation and error part of Tg of the nanocomposites samples

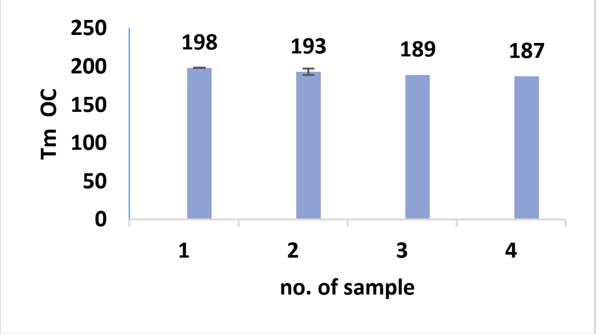


Figure 7g. Standard deviation and error part of Tm of the nanocomposites samples

As can be seen in Fig. 7f, there is a substantial error component with the sample NC1, which contains 0.02 weight percent of CNTs, while Fig. 7g, which displays the Tm standard deviation, does not exhibit any major error. The glass transition temperature and melting point of the nanocomposite samples leads to enhance the thermal stability of these sample and leads to increase the lifetime of the prepared device [27].

Optical Properties

Absorbance

The ultraviolet (UV) absorbance of PVA and samples of its nanocomposites is displayed in Fig. 8: It should be noted that PVA exhibits an absorption peak in the ultraviolet region at 250 nm. Because CNTs contain electrons that can absorb UV radiation because these wavelengths have higher energy than is necessary to stir up electrons, increasing the concentration of CNTs causes both a shift in the wavelength of UV radiation that is absorbed and an increase in the intensity of radiation absorption. We find that the resulting thin film is insufficient after 300 nm, and no absorbance wavelength peaks are discernible [28].

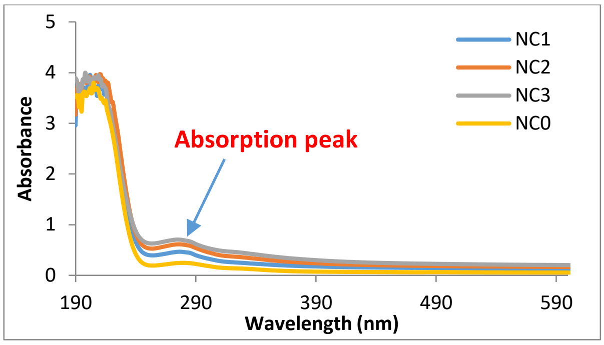


Figure 8. Absorbance of PVA+CNTs Nanofibers

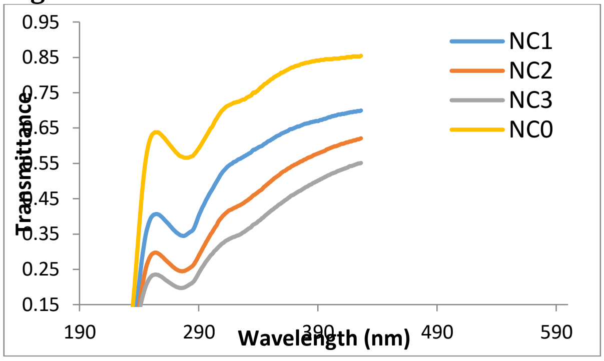


Figure 9. Transmittance of PVA+CNTs Nanofibers

Absorption coefficient

Using a UV spectrum that spans from UV radiation to visible light at 200–500 nm, Fig 10 shows the absorption coefficient of PVA nanofibers together with samples of their nanocomposites

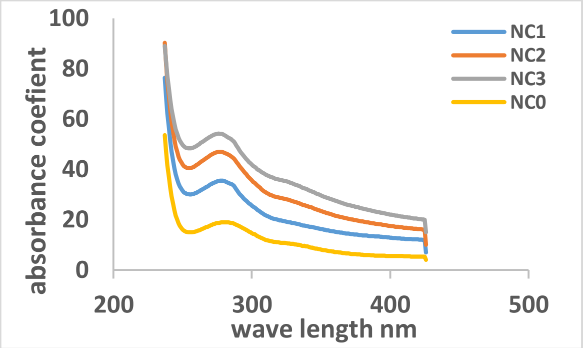


Figure 10. Absorption coefficient of PVA and PVA+CNTs Nanofibers

Fig. 10 refers to a significant absorption edge that can be seen in the UV region of the pure PVA's absorption spectrum, whereas the visible range shows a sudden decline in absorption. The maximum absorption coefficient is PVA + 6% wt of CNTs. This is because CNTs have n electrons, which allow them to absorb UV or visible light because the energy of these rays is more than what is needed to stir up electrons. This is in line with the results [29-31] . The absorption coefficient establishes the depth to which light of a given wavelength can pass through a material before being absorbed. Light is only weakly absorbed by materials with low absorption coefficients; if the material is sufficiently thin, it will seem transparent at that wavelength. Both the substance and the wavelength of light being absorbed affect the absorption coefficient. Because light with energy below the band gap cannot drive an electron from the valence band into the conduction band, semiconductor materials exhibit a sharp edge to their absorption coefficient. This light is not absorbed as a result. As photon energy increases, samples exhibit an increase in absorption coefficient.. PVA/MWCNT films have low transmittance and high absorption, which makes them useful for a variety of uses, including UV shielding and solar collector, this is matched with [32-34].

Direct Energy Gap

Fig. 11. and Table 7. show the direct energy band gap of PVA + CNTs nanofibers. Note that the direct energy gap reduces as the concentration of CNTs increases. well. As well as, this is because the CNTs with the lowest energy gaps help the electron move quickly and efficiently from the valence band to the conduction band .This is similar to that Majeed concluded [29]. The decrease in Eg as the weight percentage of MWCNTs increases may be attributed to the formation of localized states in the forbidden band gap as a result of Van Hove singularities and an increase in polaron production, this is matched with Farag and abdel-fattah [34] Electronic applications become more efficient when the energy gap in semiconductor materials narrows, thereby lowering the energy needed for additional electrons to participate in the electrical conduction process [35]. The stated Eg values of the samples under examination match those of earlier research quite [36].

Table 5. The Direct Eg of samples

No. of samples	Contents	Energy gap (eV)
NC0	PVA GEL	3
NC1	PVA GEL+50ppm	2.5
NC2	PVA GEL+4% wt	2.25
NC3	PVA GEL + 6% wt	1.75

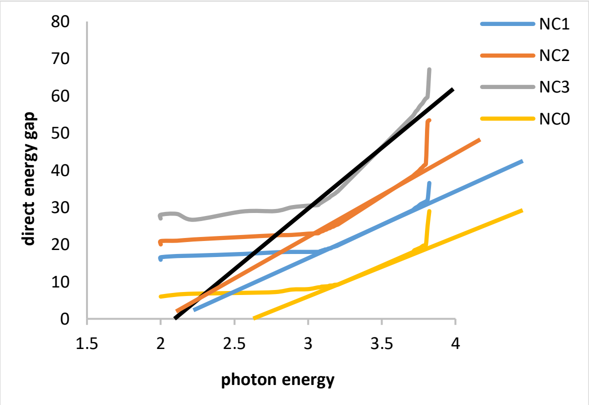


Figure 11. Direct Energy Band Gap of PVA+CNTs Nanofibers

Conclusions

This investigation leads us to the conclusion that adding CNTs to PVA membranes improves their optical, electrical, and thermal properties. The addition of CNTs results in a decrease in the energy gap with a growth in their concentration. Electrical conductivity increases along with the concentration of CNTs. Tg increases and Tm decreases as the concentration of CNT increases. With 6% weight of CNTs, the optimal sample exhibited the best absorption coefficient, lowest energy band gap of 1.75 eV, and maximum electrical conductivity of 20 $(ohm^{-1}.cm^{-1})$. As well as the glass transition temperature of the optimum sample about 107 ºC , and Tm was about 187 º C. On the other hands , can be concluded the addition of 0.06 wt of CNTs to the PVA solution leads to produce an homogenous and free beads nanofibers. According to our finding , we suggest the using of the prepared Nano composites fibers with electro voltaic devices as solar cell , sensors, and anti UV-devices. The prepared sample with electrospinning technique has many advantages as low cost, easy for fabrication with high controlling morphology and high electrical, thermal and optical properties

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Authors' declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Besides, the Figures and Images, which are not ours, have been given permission for re-publication attached with the manuscript.
- No animal studies are present in the manuscript.
- No human studies are present in the manuscript.
- No potentially identified images or data are present in the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at University of Babylon.

Authors’ Contribution Statement

In order to finish this research in its final form, each researcher's input complemented that of the others. R. M. prepared the samples. N. A. completed the FTIR spectroscopy test and viscosity of solutions. B. M. talked about the samples' optical qualities, R. M. discussed about their thermal characteristics, and H. J. finished up the nanoscale properties.

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تحسين الخواص الفيزيائية للالياف النانوية لبولي فاينيل الكحول باضافة انابيب الكاربون النانوية

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الخلاصة

في هذه الدراسة تم تحضير عينات من ألياف نانوية من كحول البولي فينيل وأنابيب الكربون النانوية بطريقة الغزل الكهربائي. تم استخدام نسب مختلفة من الانابيب النانوية الكربونية ، وهي 2 و4 و6% نسبة وزنية . تمت دراسة الخصائص الضوئية والكهربائية والحرارية. تم استخدام المجهر الالكتروني الماسح لدراسة مورفولوجية الالياف النانوية . تم استخدام التحليل الطيفي لفوريير لدراسة اواصر العينات المحضرة .تم استخدام المسعر الحراري التفاضلي لدراسة الخواص الحرارية المتمثلة بدرجة الانتقال الزجاجي ونقطة الأنصهار. أظهرت النتائج أن إضافة انابيب الكربون النانوية بنسب صغيرة 6% بالوزن يؤدي إلى تحسن في الخواص الحرارية حيث تزداد درجة الانتقال الزجاجي من 85.17 درجة مئوية إلى 107 درجة مئوية مع. نسبة تقوية 6% بالوزن . ترتفع الموصلية الكهربائية بإضافة 6% بالوزن من الأنابيب النانوية الكربونية من (14.28 الى 20 اوم$^{-1}$ سم$^{-1}$ تشير نتائج طيف الأشعة فوق البنفسجية إلى إمكانية التحكم في فجوة نطاق الطاقة بإضافة الأنابيب النانوية الكربونية وتقليل قيمتها من 3.25 الكترون .فولت الى 0.75 الكترون .فولت بإضافة 6% نسبة وزنية من المادة النانوية . كما ان خواص الامتصاصية ومعامل الامتصاص تزداد بإضافة انابيب الكاربون النانوية ، حيث اثبتت النتائج ان افضل نتيجة في تحسين الخواص كانت بالضافة 6% من انابيب الكاربون النانوية.

الكلمات المفتاحية: انابيب الكاربون النانوية ، بولي فينيل الكحول ، اغشية رقيقة ، متراكبات نانوية ، مطيافية الاشعة فوق البنفسجية.