The Republic of Iraq
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"Studying of the physical and chemical properties of CNT/ PVA Nano composite thin films"

Graduation project submitted to the Department of Polymers and Petrochemical Industries -Faculty of Materials Engineering - University of Babylon as part of the requirements for obtaining a bachelor's degree

By :

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2022-2023

﴿ وَلَمَّا بَلَغَ أَشُدَّهُ وَاسْتَوَىٰ آتَيْنَاهُ حُكْمًا وَعِلْمًا وَكَذَٰلِكَ نَجْزِي الْمُحْسِنِينَ ﴾

سورة القصص- الآية [14]

Supervisor Acknowledgment

I acknowledge that this tagged project: (The effect ofcarbon nanotubes on the physical and chemical properties of polystyrene)

Prepared by the student:
(Ibrahim salman Akmoush , Zahra Hussain Zwaid)

It was done under my supervision in the Department of Polymers and Petrochemical Industries - College of Materials Engineering

Signature- : Supervisor Name-:Academic Rank-: Date- :

Dedication

To whom I fed me and my water and overwhelmed me with the flood of his blessings and my Lord ... Almighty.

To the cities of knowledge and its door and the salvation ships ... the Messenger of God and the pure God ... (peace be upon them) .. my masters.

To the argument of God, adult, and blessed with a genius ... the existing imam and the expected justice ... (God hurry, his joy is honorable)

To my first role model and my brings, who illuminates my path to those who gave me and still gave me without limits to those who raised my head loudly with him ... My dear father, God blessed him with an asset to me

To the purest hearts of my life ... my dear parents ..

I dedicate you a summary of my scientific effort

Ibrahim salman Akmoush

Zahra Hussain Zwaid

Thanks

At the outset, we thank God Almighty for having enabled us to complete the research project. Then I would like to thank Dr. (Dr. HanaJawad) who supervised my graduation project, whose experience was invaluable in formulating the most important topics and methodology of the project. Then I would like to express my appreciation to my colleagues for their wonderful cooperation and support for me.

I would also like to thank all the teaching staff in the Department of Physics for their valuable guidance throughout theperiod of my studies. Their feedback provided me with the right

experience that enabled me to choose the right direction and
.successfully complete my project

In addition, I would like to thank my parents for their wise
advice and great support. You have always been the first support for me.
Finally, I could not have completed this letter without the supportof my
friends who gave me motivating advice, moral support and everyone who had
a contribution to making the project

Ibrahim salman Akmoush

Zahra Hussain Zwaid

Abstract

In this study, nanocomposite samples were prepared from polyvinyl alcohol polymer as a matrix material, and carbon nanotubes as a reinforcing material.

The hand casting method was used to produce thin films of nanocomposite solutions.

Different proportions of the reinforcement particles were used, including 0.01, 0.02, and 0.03. wt%

The electrical, thermal and wettability properties were studied.

Infrared spectroscopy was used to analyze the bonds of the prepared samples.

The results showed that the addition of carbon nanoparticles in small proportions of 0.01 percent by weight leads to an improvement in the thermal properties represented by an increase in the value of the glass transition degree, which increases from 85.17 degrees Celsius for the pure material to 92.13 degrees Celsius for the strengthening percentage of 0.01 percent by weight of carbon. nanoparticles

The degree of glass transition continues to increase with the increase in the percentage of carbon nanoparticles to reach 107 degrees Celsius with a strengthening percentage of 0.03 percentage by weight of carbon.

The value of electrical conductivity improves with the addition of a small percentage of carbon nanoparticles, as it increases from 14.04 milliohms / cm to 15 .57 milliohms / cm, but it decreases at a slight rate with the increase in carbon nanoparticles.

The value of the wetting angle improves with the addition of a small percentage of carbon nanoparticles, as it increases from 78.78 degrees to 87.15 degrees, but it decreases by a slight percentage with the increase of carbon nanoparticles.

The results of the infrared analysis showed that the physical interaction between the carbon particles and the base material, and the permeability decreases with the addition of carbon nanoparticles.

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Chapter one

The Introduction

1. Introduction

Using new materials with enhanced qualities is always fundamentally necessary in industry. Due to their exceptional chemical and physical characteristics, such as high hardness and high strength but extremely low density, carbon nanotubes (CNTs) have drawn significant study attention from a variety of engineering and scientific fields. These spherical, hollow nanostructures were made possible by Iijima's discovery of hexagonal unit cells in 1991. The characteristics of carbon nanotubes have been estimated and developed through a number of investigations. Among the most significant goals of these research publications were the determination of the tensile strength (up to 63 GPa), Yunck's modulus (approximately 1 Tb), and the vibrational stability of carbon nanotubes. Before the advent of the composites industry, studies into short fiber- reinforced

1.1 / Introduction to carbon nanotubes:

They have cylindrical nanostructures and are carbon allotropes. It should be mentioned that the length to diameter ratio of carbon nanotubes is 132,000,000:1, which seems to be far longer than that of any other substance. These carbon molecules have unique qualities that make them valuable in a variety of applications in nanotechnology, electronics, and optics, as well as in a variety of other departments linked to materials science, as well as a variety of additional prospective uses in the domains of architecture. It could also be employed in the creation of body armor in rare cases. They have great strength, distinctive electrical characteristics, and are effective heat conductors.

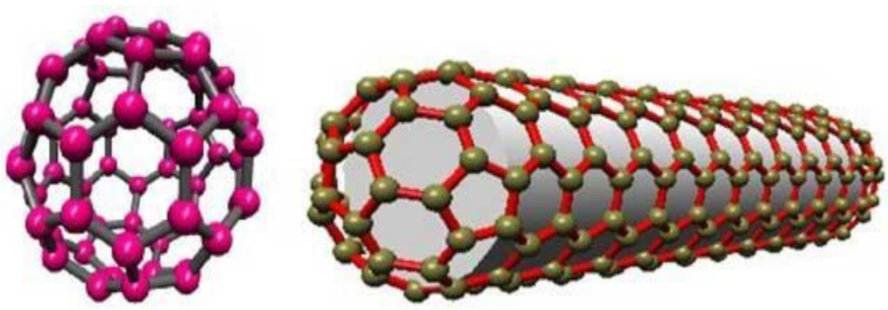
a quantum chemistry, in particular orbital hybridization. Similar to graphite, the chemical bonds that make up nanotubes have sp2 orbital hybridization. The strength and toughness of the nanotubes come from these bonds, which are stronger than the sp3 bonds present in diamond. In addition, the nanotubes line themselves up as "ropes" held together by van der Waals forces..

1.2 Historical overview:-

Nanotechnology is a scientific precedent for the twenty-first century that will lead the world to a new industrial revolution that provides many benefits to humanity, especially in the fields of computer technology, medicine and materials science (material science) in Nano dimensions, so we will talk about carbon nanotubes, or what is known as nanotubes. Which takes a large space in the fieldof nanotechnology.

In 1991 the nanotube was discovered by the Japanese scientist Ijima Sumiowhile he was studying carbon products in the process of electric discharge between two carbon electrodes, while using the transmission electron microscope(TEM Transmission Electron Microscope). But before we start studying the nanotube, we must get acquainted with the single-walled carbon sphere called Fullerene.

In 1985, Kroto and Smalley were able to discover the monolayer ball consisting of 60 carbon atoms, and it was the most stable among the carbon balls with a number of more or less than 60 atoms, thus realizing Euler's theorem. To the architect Buckminster Fuller, after which it was named Fullerene (see Figure1-1).[2]



(a) (b)

Figure (1-1): (a) The monolayer sphere, that is, fullerene, made of 60 carbon atoms.

(b) Single Walled Carbon NanoTube (SWCNT: Single Walled Carbon Nanotube)

3 Previous work

1- Fabrication and Material Characteristics of Polystyrene/CarbonNanotubes :-

Polystyrene/carbon nanotube (PS/CNT) composites have attracted considerable attention in research and industry fields due to high strength and electrical conductivity of CNT. Recently, several investigations showed that the PS/CNT composites exhibited the significant enhancement in thermal, mechanical, and electrical properties at room and/or high temperatures as well as possessed excellent process-ability which was quite similar to those of pure polymers. In this study, the various processing methods used to fabricate the composites are outlined with a special focus on solution processing, melt mixing, in-situ polymerization, etc. This paper discusses the non-covalent and covalent modifications of CNTs with PS, which are commonly applied to improve the dispersion and compatibility of the PS/CNT composites. The thermal, rheological, and mechanical properties of the present composites are also reviewed. The influences of different variables, such as type and content of CNT processing method and temperature, on the electrical responses are also highlighted. The discussion of the different properties in this study concluded that the addition of CNT would be beneficial for improving the materials performanceof PS composites for industrial applications.[3]

2- Making carbon nanotubes with polystyrene:-

Single-walled and multiple-walled carbon nanotubes were functionalized with a polystyrene copolymer, poly(styrene-co-p-(4-(4‘-vinylphenyl)-3- oxabutanol)). The functionalization reaction conditions were designed for the esterification of the nanotube-bound carboxylic acids. The polymer-attached carbon nanotubes are soluble in common organic solvents, making it possible to characterize the samples using not only solid-state but also solution-based techniques. The solubility has also allowed an intimate mixing of the functionalized carbon nanotubes with polystyrene. Results from the characterization of the functionalized carbon nanotubes, including the chemical and thermal defunctionalizations of the soluble samples, and the fabrication of polystyrene−carbon nanotube composite thin films using a wet-casting method are presented and discussed.[4]

The theoretical part

1 / What do we mean by carbon nanotubes?

Carbon (C) atoms are the only component of carbon nanotubes, which are incredibly tiny, incredibly thin tubes or cylinders. The only way to see its tubular structure is using an electron microscope. It is a dark solid made up of a complex network of extremely tiny bundles or bundles made up of several dozen nanotubes.

"Nano" stands for "extremely tiny" in a prefix. The measurement is one billionth of a measurement, as indicated by the term "Nano" used in the name. One nanometer, or nanometer, is one billionth of a meter, or 1 nanometer = 10-9 m, for instance.

Each tiny carbon nanotube is made up of one or more graphite sheets that have been wrapped around one another. Single-walled nanotubes, which consist of a single coiled sheet, and multi-walled nanotubes are two different types (two or more cylinders, one inside the other).

Carbon nanotubes are extremely resilient to breaking, strong, and flexible. It has excellent thermal and electrical conductivity. They create a very light material as well.

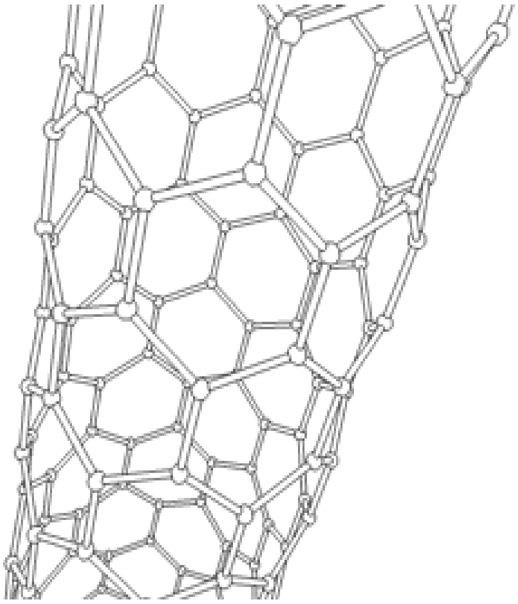
Because to these characteristics, it may be used to many different industries, including those in the automotive, aerospace, electronics, and other sectors. They have also been utilized in the medical field, such as when delivering proteins, vaccinations, and anti-cancer medications.

*Nonetheless, it needs to be handled carefully since it might harm the lungs if breathed.[5]

2-2 / Construction of carbon nanotubes

2-2-1 / Physical Structure

Carbon nanotubes are extremely fine and small tubes or cylinders whose structure can only be seen with an electron microscope. It consists of a sheet of graphite (graphene) wrapped in a tube.



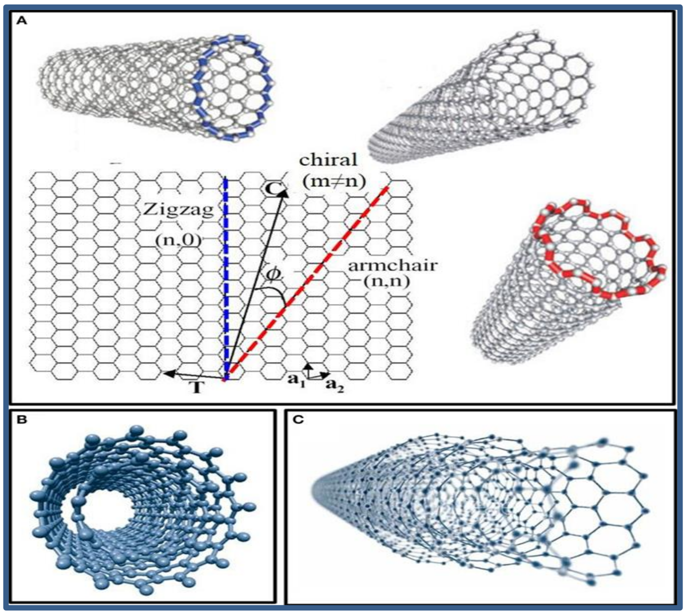


Figure (2-1): Nano-tube

They are hollow, cylindrical particles consisting only of carbon atoms. The carbon atoms are arranged in the form of small hexagons (six-sided polygons) similar to benzene and connected to each other (benzene condensed rings).

Tubes may or may not be clogged at their openings and can be quite long when compared to their diameters. It is equivalent to graphite sheets wrapped in seamless tubes]6[.

2.2/ Chemical composition

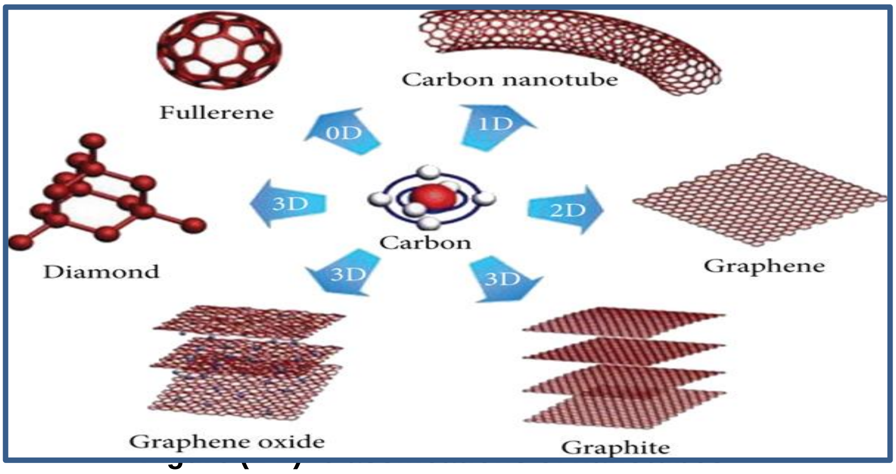
CNTs are polycyclic structures. The bonds between carbon atoms are covalent (ie they are not ionic). These links are at the same level and are very strong.

The strength of the C=C bonds makes carbon nanotubes extremely tough and strong. In other words, the walls of these tubes are very strong.

The out-of-plane joints are very weak, which means that there are no strong joints between one tube and the other. However, they are attractive forces that allow the formation of bundles or bundles of nanotubes]7[.

3 / Classification by number of tubes

Carbon nanotubes are divided into two groups: single-walled nanotubes, or SWCNTs. Single-walled carbon tube, multi-walled nanotubes, or MWCNTs. (Multiwall Carbon Nanotube)



Single-walled carbon nanotubes (SWCNTs) consist of a single graphenesheet wrapped in a cylinder, in which the vertices of the hexagons fit together perfectly to form a seamless tube.

Multi-walled carbon nanotubes (MWCNTs) consist of concentric cylinders placed around a common hollow center, that is, two or more hollowcylinders placed within each other]8[.

4 / physical properties

Solid carbon nanotubes exist. A extremely thick and intricate network is created when they combine to form bundles, bundles, bundles, or "chains" of several dozen nanotubes.

They are stronger in tensile than steel. This indicates that they are very resilient to fracture under stress. Theoretically, it may be a thousand times stronger than steel.

It is incredibly flexible and may be folded, twisted, and bent without suffering any harm before returning to its original shape. They weigh relatively little. They are effective heat and electricity conductors. They are believed to have highly conductive electronic activity or an extremely varied electronic behavior. The behavior of CNT tubes with hexagons organized in an armchair-like pattern is metallic or metal-like.

Both metallic and semiconductor materials may be organized in spiral and zigzag patterns. [9]

5 / Chemical properties

Due to the strength of the bonds between carbon atoms, carbon nanotubescan withstand very high temperatures (750 °C at atmospheric pressure and 2800 °C in vacuum).

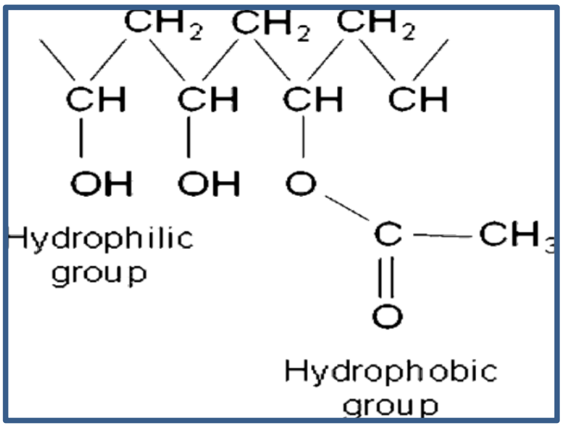
The ends of the nanotubes are more chemically reactive than the cylindrical part. If they are subjected to oxidation, the ends are oxidized first. Ifthe tubes are closed, the ends open. When treated with nitric acid HNO3 or sulfuric acid H2Sw4 under certain conditions, CNTs can form carboxylate- COOH or quinone-type groups O=C-C44-C=0

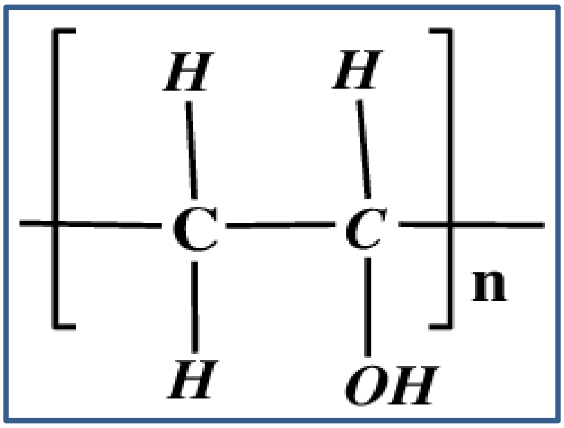
Carbon nanotubes with smaller diameters are more reactive. Carbonnanotubes can contain atoms or molecules of other types in their internal channels]10[.

2-6 / polyphenol alcohol?

Polyvinyl alcohol (PVA), which is essentially made from polyvinyl acetate through hydrolysis, is easily degradable by biological organisms and in water is a solubilized crystalline structure polymer . PVA is an artificial polymer that has been used during the first half of the 20th century worldwide. It has been applied in the industrial, commercial, medical, and food sectors and has been used to produce many end products, such as lacquers, resins, surgical threads, and food packaging materials that are often in contact with food. PVA is a biodegradable imitation of natural polymers used in paper coating and textile sizing . This polymer is widely used by blending with other polymer compounds, such as biopolymers and other polymers with hydrophilic properties; it is utilized for various industrial applications to enhance the mechanical properties of films because of its compatible structure and hydrophilic properties .
PVA is a biodegradable polymer, and its degradability is enhanced through hydrolysis because of the presence of hydroxyl groups on the carbon atoms.
Chemical Structure of PVA
The properties of polyvinyl acetate depend on the extent or degree of its hydrolysis, specifically whether it is full or partial (Figure 2.3), which in turn dictates its

categorization into two groups, namely, (a) partially hydrolyzed and (b) fullyhydrolyzed)





(a) (b)

Figure (2,3)The chemical structure of PVA :
(a) partially hydrolyzed (b) fullyhydrolyzed

The aim of this study :

This study aims to prepare carbon nanocomposites based poly vinyl alcohol matrix, for improving thermal, wettability , and electrical conductivity of prepared samples.

Chapter TWO

Expermental part

1-2 Introduction

This part contains the material, method of preparation samples and tests that performed on prepared samples

2-2 Materials:

- Polyvinyl alcohol (PVA) as a white soluble powder by water with molecular weight 4000 g/mol.
- Carbon nanotubes (CNT) powder with average diameter 40-50 nm
- Distilled water
- Plastic dishes
- Ultrasonic device

3.2 Method of preparation of nanocomposites

Hand casting method was used to prepare the nanocomposites thin film as following procedure

1. Weigh out 20 g of PVA powder and add it to a beaker containing 200 ml of distilled water.
2. Heat the beaker on a hot plate and stir continuously until the PVA powder is completely dissolved in the water.
3. Add the desired amount of CNT powder to the PVA solution (0.001 g, 0.002 g, or 0.003 g) and stir well.
4. Transfer the PVA/CNT solution to a plastic dish.
5. Use an ultrasonic device to disperse the CNTs in the solution for about 10 minutes.
6. Allow the solution to cool to room temperature.
7. Leave the solution to stand for 24 hours to ensure the complete dispersion of CNTs.
8. Place the plastic dishes containing the PVA/CNT solutions in an oven set at 50°C and leave them to dry for 24 hours.
9. Remove the plastic dishes from the oven and allow them to cool to room temperature.
10. Cut the dried PVA/CNT samples into small pieces for testing.

4.2 Tests and measurements

4.2.1 Infrared Fourier transform spectrometer (FTIR)

Fourier transforms infrared technique used to characterize the prepared samples using instrument type (IR Affinity-1) made in (Kyoto Japan) located in Polymer and Petrochemicals Department. To measure a sample, calibrate the device using the KBr, and then prepare a powder of the sample to be examined, and mixed with KBr (mixing ratio 99% KBr). FTIR spectrum provides a diagram between the permeability or absorption and the number of waves that show the chemical composition of the material. This test was carried out according to ASTM E1252

4.2.2 Differential scanning calorimetry (DSC)

This device is used to measure thermal transitions, entropy, specific heat, and enthalpy. This test was done according to ASTM D3418-03 using the SH1MADZ-4 DSC-60 device. Samples of nanofibers were in the form of powder, then compress in a pan and placed in the device to conduct the examination. The specimen was tested under nitrogen gas and heating rate of 10 ˚C/min with a heating range from RT to 350 ˚C."

4.2.3 Wettability

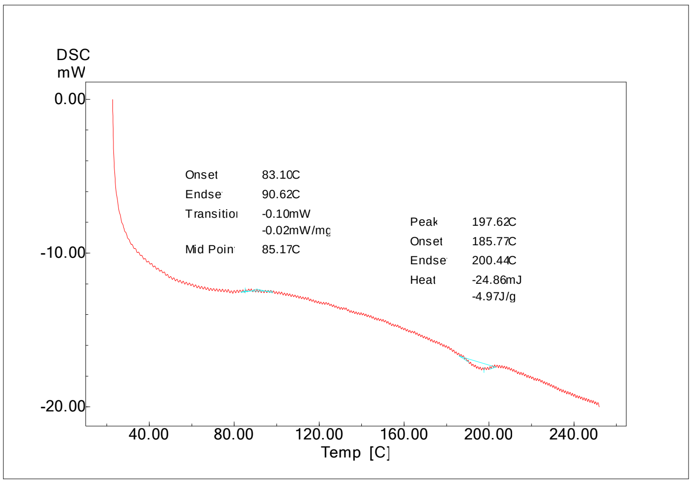
The used device is SL 200C - Optical Dynamic I Static Interfacial Tensiometer & Contact Angle Meter which is manufactured in KINO Industry Co., Ltd., the USA with a contact angle range from 0_o to 180_o This device makes calculation and comparison of left and right contact angle as well as calculate their average value giving a Real-time data graph monitoring changes in contact angle with video recording.

Chapter Three

Results and discussion

1-DSC analysis :-

Figure1 and table 1. Showed the DSC analysis of the CNTs/PVA nanocomposites samples



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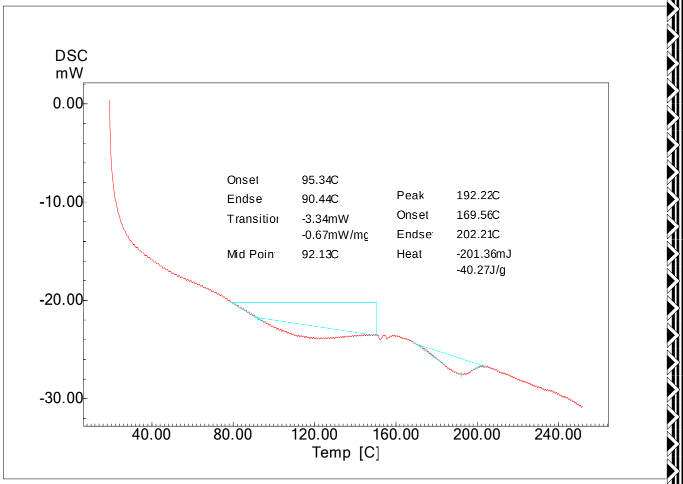

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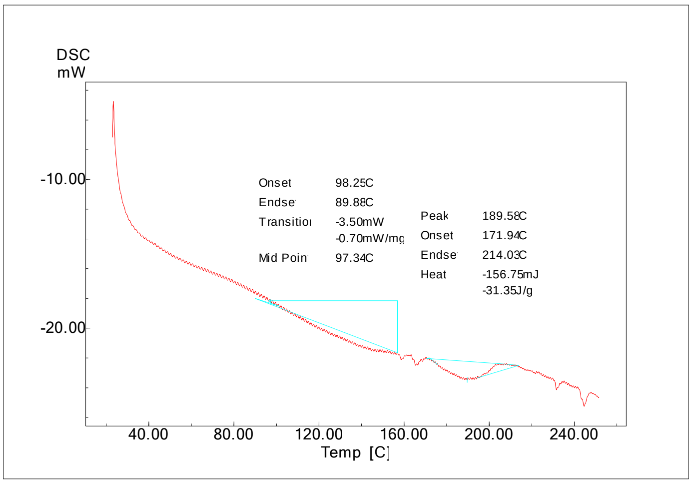

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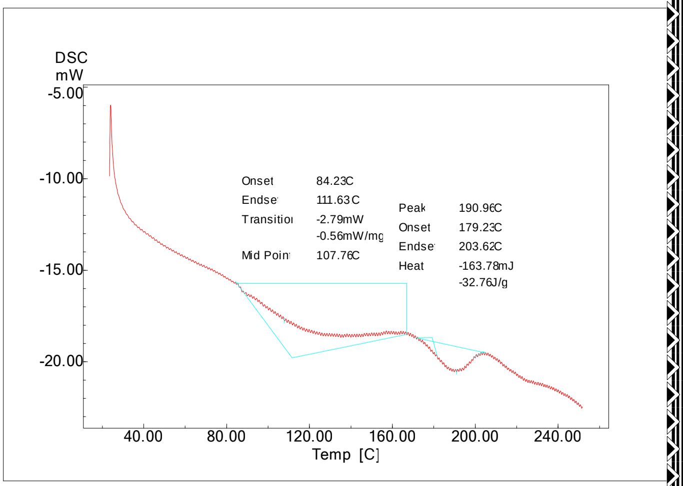
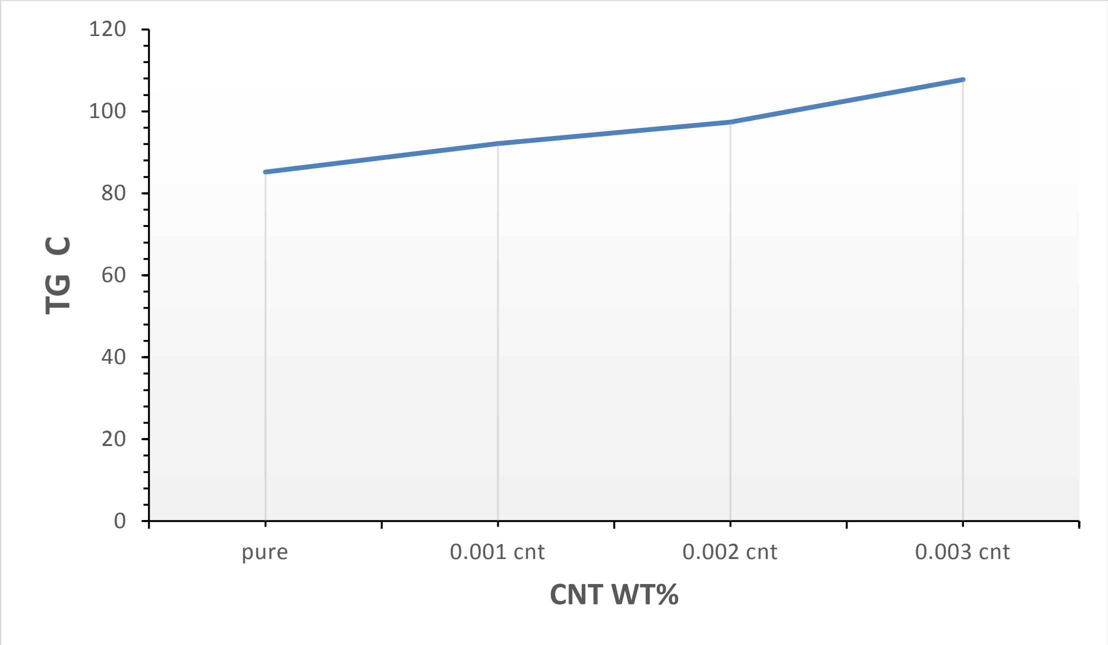


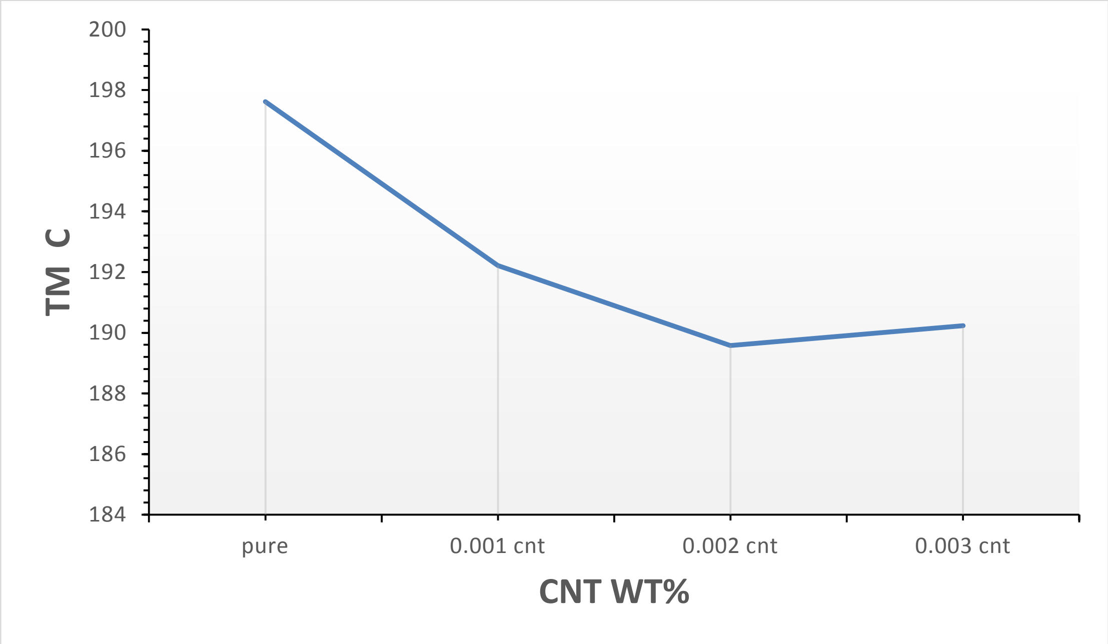
d

Figer. 1. DSC analysis of the Nanocomposites

Table 1. Thermal properties of CNTs/PVA nanocomposites

No	Sample Components (Add CNT to PVA	Tg ºC	Crystallization Under TgmW/mg	TM ºC	crystallization under Tm j/g
1	Pure	85.17	(0.02)	197.62	(-4.97)
2	0.001	92.13	-0.67	192.22	-40.27
3	0.002	º97.34	-0.70	189.58	-31.35
4	0.003	107.76	-0.56	190.23	-32.76





Figer. 1. (a) show the relationship between CNTs ratio and Tg of samples

(b) show the relationship between CNTs ratio and Tm of samples

When CNTs are added to PVA, the thermal properties of the resulting composite material can change due to the interactions between the PVA and CNT. The glass transition temperature (Tg) and melting temperature (Tm) are two important thermal properties that can be affected by the addition of CNTs.

In general, the addition of CNTs to PVA can increase the Tg due to it make on decreasing the moving of polymer chain and increasing the stiffness of these chains . Tm was decreased due to the CNTs have high thermal conductivity and high surface area and more effecting with applied temperature.

Crystallinity results of samples showed that the adding the small amount of CNTs leads to increase the crystallinity due to the homogenous desperation process of CNTs through PVA and CNTs have high surface area with small volume and high crystallinity . Adding more amount of CNTs leads to decreasing the crystallinity value and referees to exothermic reaction behavior. [14]

2-FTIR ANALYSIS:-

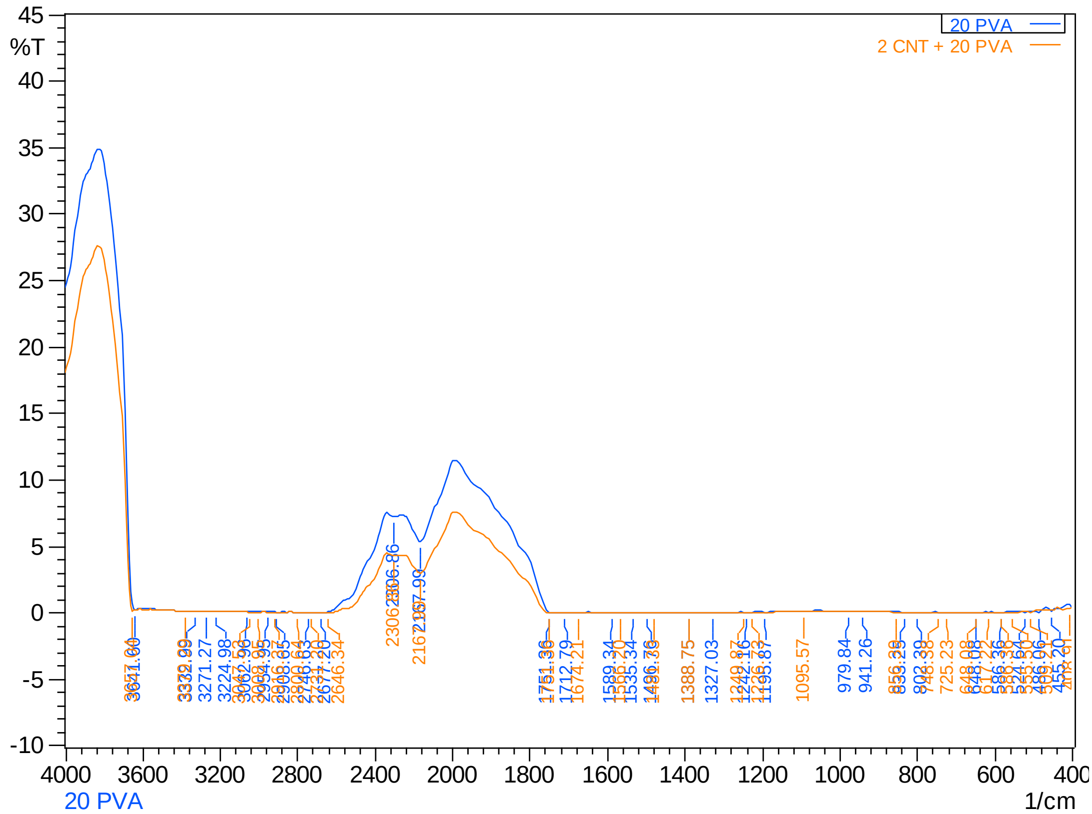
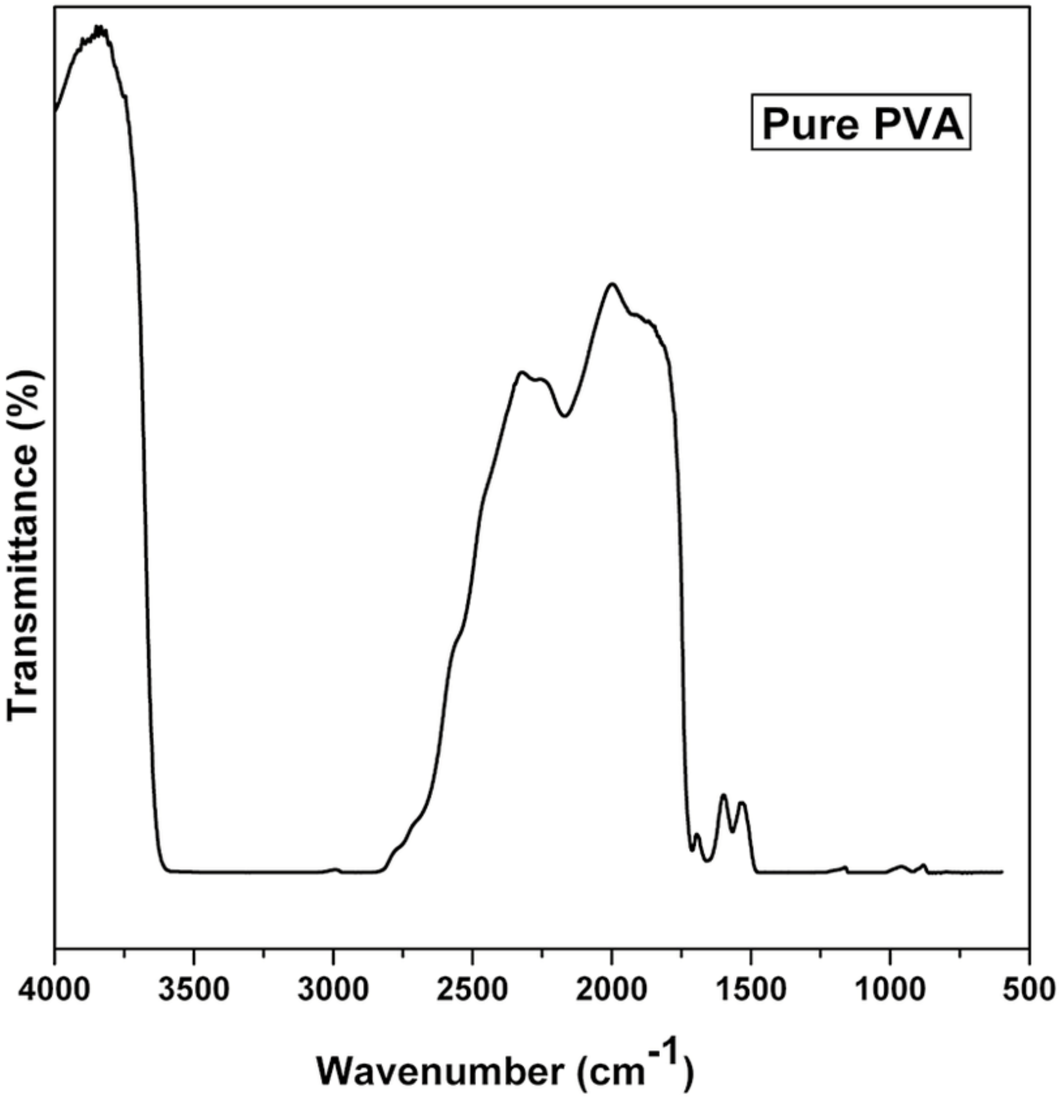


Figure 2. a FTIR analysis of pure PVA and PVA/CNTs



(b) standard FTIR of PVA

Figer. 2. FTIR analysis of samples

According to the results of the examination with the comparison, we note that the intensity of transmittance of PVA without adding CNTs is higher than that of PVA containing CNTs, and that is because CNTs is an opaque materials and the carbon particles block the rays from penetrating into the base material . When added to the polymer, the polymer's absorption values increase for the wavelengths falling on the polymer .[15]

3-Electrical conductivity :-

The electrical conductivity test results for the four samples can be interpreted as follows:

- The first sample, which is a pure PVA polymer material, has a low electrical conductivity due to the absence of any conductive fillers.
- The second, third, and fourth samples, which contain increasing amounts of CNTs, show a significant increase in electrical conductivity. This is because CNTs are highly conductive materials, and their addition to the PVA matrix creates a conductive pathway within the composite.

The increase in electrical conductivity with an increase in the percentage of CNTs is not linear and can be affected by various factors such as the dispersion of CNTs within the matrix, the alignment of CNTs, and the interfacial interaction between CNTs and the polymer matrix. In general, the electrical conductivity tends to increase with increasing CNT content up to a certain point, beyond which it may plateau or even decrease due to the formation of agglomerates or clusters of CNTs that can disrupt the conductive pathway.

Therefore, optimizing the CNT content and the dispersion and alignment of CNTs within the PVA matrix is crucial to achieving the desired electrical conductivity in PVA-CNT composites.[16]

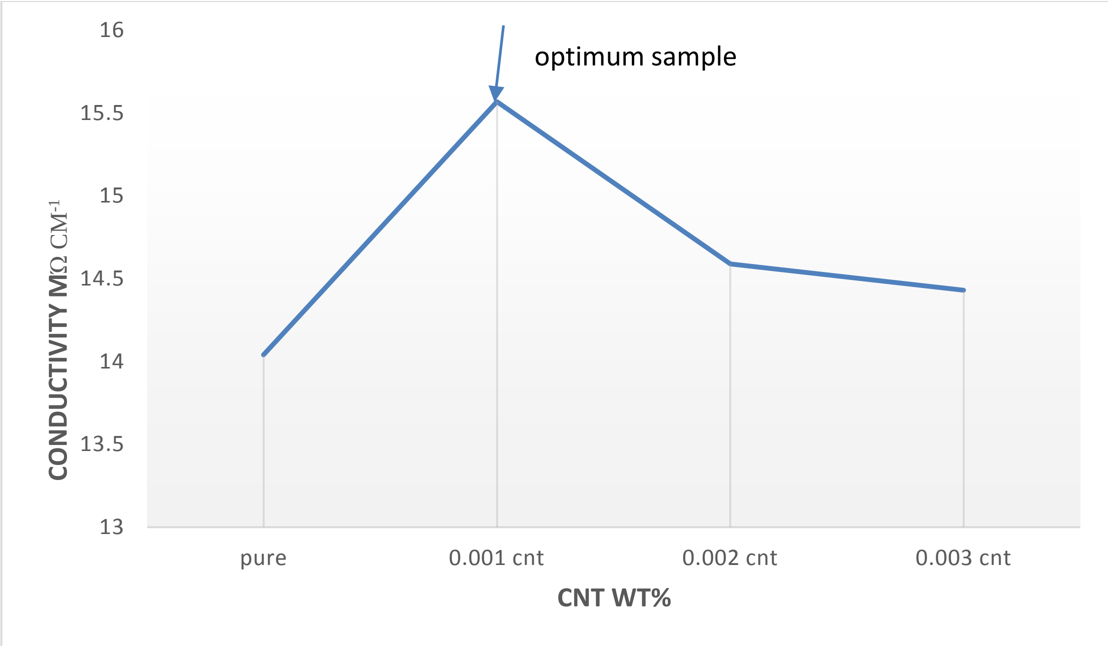
Also we notice there are small ratio of decreasing with high CNTs contents due to agglomeration of CNTs and uninform distribution the CNTs particles through the PVA matrix.

The optimum sample of electrical conductivity was the sample number (2) which contain [PVA+ CNTs (0.001

wt%)] which refers to high dispersion of nanoparticles was performed.

Table 2. Electrical properties of samples

No.	Sample Components (Add CNT to PVA)	Resistivity / mΩ	Conductivity mΩ cm^{-1}
1.	Pure	0.712	14.04
2.	0.001	0.642	15.57
3.	0.002	0.685	14.59
4.	0.003	0.693	14.43



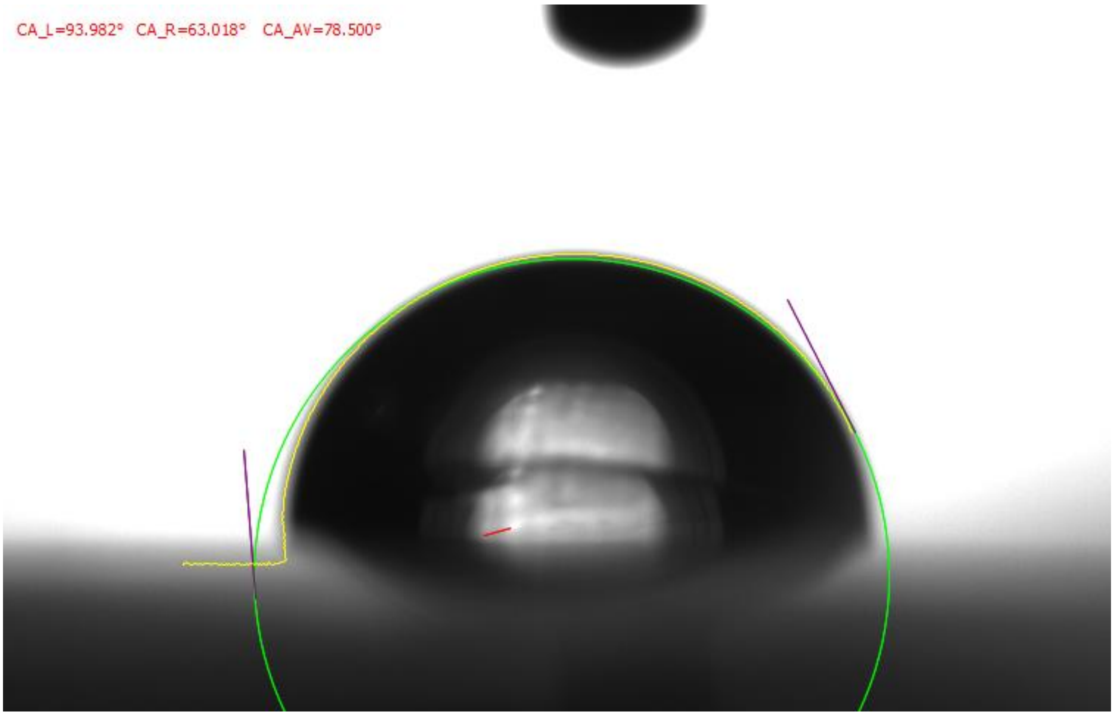
Figer.3 Electrical conductivity of CNTs/PVA

4- Contact angle:-

Figure 4 and table 4. show the wetting angle of the prepared samples

We notice an increase in the wetting angle when adding the first percentage of carbon nanotubes 0.01 percent by weight, and this is due to the hydrophobic nature of carbon nanotubes . The homogeneous dispersion of carbon nanoparticles leads to the high distribution of CNTs and more homogeneous surface of the sample

When the carbon nanotube content is increased, this leads to a decrease in the wetting angle due to the agglomeration occurring in the carbon particles and the lack of dispersion in a good manner, which leads to a non-homogeneous surface.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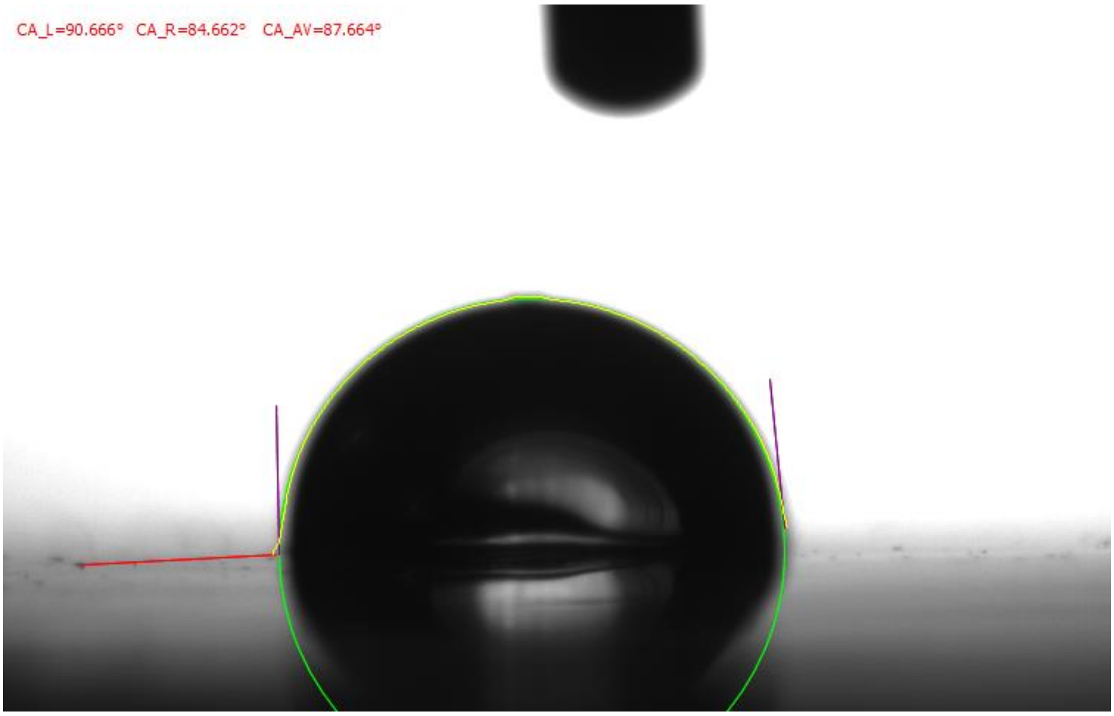
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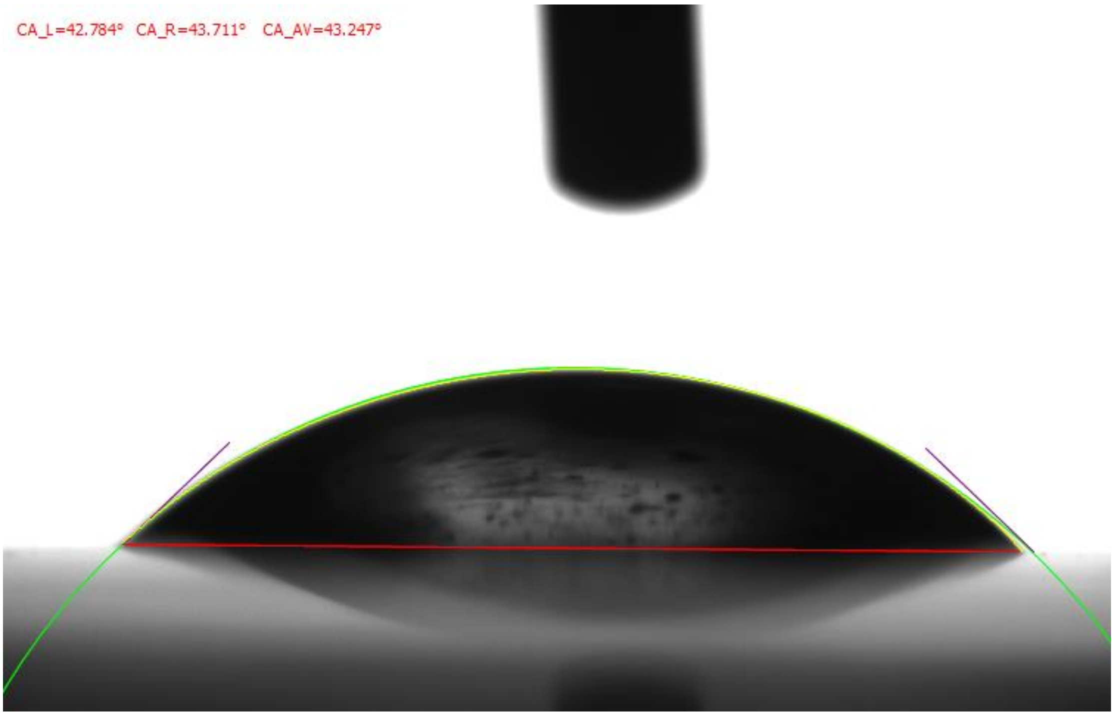
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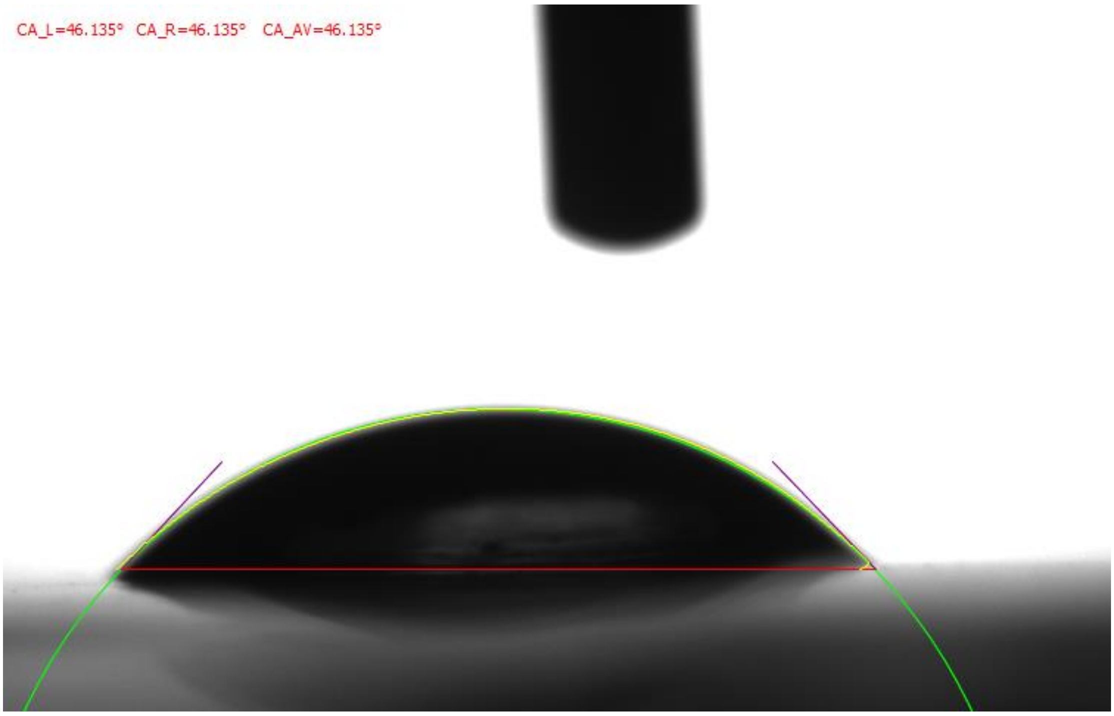

Table 4. contact angle of nanocomposites samples

No.	Sample Components (Add CNT to PVA)	Contact angle
1.	Pure	78.5
2.	0.001	87.66
3.	0.002	43.2
4.	0.003	46.1

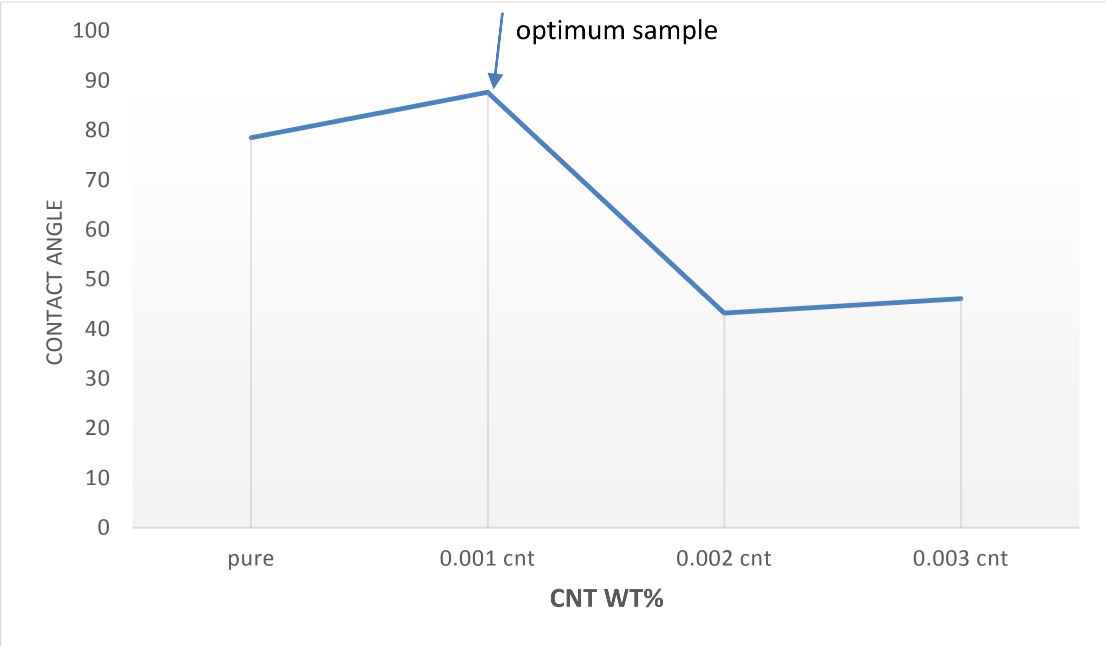

Figure 4. contact angles of all samples with CNTs ratio

Chapter Four

Conclusions and recommendations

4.1 Conclusions

We conclude from the current research that the addition of carbon nanotubes leads to an improvement in the electrical properties of the polymer, and that the homogeneous distribution using a few percentages of carbon particles leads to better properties such as wettability, electrical and thermal conductivity.

The interaction between the carbon particles and the polymer is a physical interaction that reduces the intensity of the rays penetrating into the model.

4.2 Recommendations

1- Using other polymers as a base material with carbon nanotube particles as reinforcing materials, such as polyethylene glycol and polyamide.

2- Using other types of nano-reinforcement particles such as silver and gold with comparing them with the current study

3- Studying other properties such as mechanical, optical and magnetic properties.

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الخلاصة

تم في هذه الدراسة تحضير عينات متراكبة نانوية من بوليمير البولي فاينيل الكحول كمادة اساس ، والكاربون نانوتيوب كمادة تقوية .

تم استخدام طريقة الصب اليدوي لانتاج افلام رقيقة من المحاليل المتراكبة النانوية.

تم استخدام نسب مختلفة من دقائق التقوية تتضمن 0.01 و 0.02 و 0.03 نسبة وزن. كما تم دراسة الخواص الكهربائية ، الحرارية وقابلية التبلل .

تم استخدام مطيافية الاشعة تحت الحمراء لتحليل الاواصر للنماذج المحضرة.

أثبتت النتائج ان اضافة الكاربون النانوي بنسب قليلة ٠,٠١ نسبة مئوية وزنية تؤدي الى تحسين الخواص الحرارية متمثلة بزيادة قيمة درجة الانتقال الزجاجي التي تزداد من 85.17 درجة مئوية للمادة النقية الى 92.13 درجة مئوية لنسبة التقوية ٠,٠١ نسبة وزنية من الكاربون النانوي .

تستمر درجة الانتقال الزجاجي بالزيادة مع زيادة نسبة الكاربون النانوي لتصل الى ١٠٧ درجة مئوية مع نسبة تقوية ٠,٠٣ نسبة مئوية وزنية من الكاربون.

تتحسن قيمة التوصيلية الكهربائية مع اضافة نسبة قليلة من الكاربون النانوي حيث تزداد من 14.04ملي اوم / سم الى 15.57 ملي اوم / سم ، لكنها تقل بنسبة قلبلة مع زيادة الكاربون النانوي .

تتحسن قيمة زاوية التبلل مع اضافة نسبة قليلة من الكاربون النانوي جيث تزداد من 87 .78 درجة الى 87.15 درجة ، لكنها تقل بنسبة قلبلة مع زيادة محتوى الكاربون النانوي .

اثبتت نتائج تحليل الاشعة تحت الحمراء ان التفاعل فيزيائي بين دقائق الكاربون والمادة الاساس ، وتقل النفاذية مع اضافة الكاربون النانوي .