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RESEARCH ARTICLE

Biodegradable Nanocomposites Thin Films for Recyclability Electronic Covering

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ABSTRACT

In this study, the nanocomposite thin films were prepared from polyvinyl alcohol (PVA) that reinforced with different proportions of carbon nanotubes (CNTs) via hand casting method. The current work aims to create the connection between improving surface characteristics for high efficiency electronic covering and biodegradability. The nature of bonding between the PVA and CNTs were studied by Fourier-transform infrared spectroscopy (FTIR). Wettability properties were studied via contact angle analyzer. The two dimensions' morphology of nanocomposites was studied using atomic force microscopy (AFM). Biodegradable tests of nanocomposites were performed via burial in soil for seven days. According to the contact angle data, adding PVA to CNTs causes the contact angle decreased from 95 degrees to 56 degrees at a concentration of 0.001 wt% of CNTs, and it then increases to 78 degrees at a concentration of 0.03 wt% of CNTs. The results of the infrared analysis showed that the physical interaction between the carbon nano tubes and the poly vinyl alcohol matrix, as well as, the IR intensity transmittance decreases with the addition of carbon nano tubes. Atomic force microscopy results prove that the roughness average decreases with increasing the CNTs ratios from 0.01 up to 0.03 wt% percent, bearing index increases with increasing the carbon nano tubes concentrations, and surface flatness increases with increasing the carbon nano tubes ratios. The best sample for this study was TH2 (0.002 wt of CNTs.

Keywords: Biodegradability, CNTs, Contact angle, Nanocomposites, PVA, Thin films

Introduction

Pollution stems from a variety of sources, however non-recyclable electronic waste is apart a major contributor due to the accumulation and poor decomposition. Due to their low quality and susceptibility to break or deteriorate fast, millions of inexpensive imported electronic devices of lower origin have recently expanded. Consequently, it was essential to get eliminate them.[1] The development of biodegradable nano composites polymers, which dissolve or break down in the environment without causing pollution, has great promise for efficiently reducing the load on the environment as poly vinyl alcohol reinforced with conductive ions[2] or conductive nanoparticles as CNTs.[3] Furthermore, biocompatible biodegradable polymers allow electronics to be employed in implantable biomedical applications. However, most biodegradable polymers fall short of some specific application requirements, like flexibility,[4] electric conductivity,[5] biodegradability,[6] as well as gas and water vapor barrier.[7] Although carbon nanotubes (CNTs) are commonly used in the fabrication of electronic components, their inability to decompose after usage presents a major problem for the disposal of electronic waste, particularly when embedded in thermosetting polymers, Particularly when embedded in thermosetting polymers

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The field of nanotechnology and science studies is very extensive. It is one effective strategy for improving performance and creating new jobs for lightweight material implementations and is the combination of active nanoparticles with polymers. Due to the benefits of the matrix phase and nano-reinforcement phase, the resulting polymer nanocomposites are of interest.[8]

Polymers nano composites are crucial parts of modern electronics. Polymers are essential to nanocomposites because they are lighter than other engineering materials like metal and ceramic and are easier to mold than other materials from previous generations.[9]

Fortunately, Carbon nanotubes (CNTs) have attracted a lot of research interest from a range of engineering and scientific domains because of their remarkable chemical stability and physical properties, such as high surface area and extremely low density The superior optical, electrical, and mechanical capabilities of CNTs are well known. They show excellent mechanical and thermal stability, and outstanding flexibility. They are excellent candidates for nanotechnological applications because they can conduct electricity.[10]

Degradable polymer nano composites reinforced with a CNTs or others carbon forms have a very wide applications as filtration[11,12] Air Purification,[13] solar cell,[14] anti-corrosion coating,[15] food packaging[16] CNTs and PVA are utilized in anti-pollution applications because they are recognized as key technology for sustainable pollution control engineering.[17,18]

Hydro-biodegradable polymers play an important role in anti-pollution applications due to its hydrophilicity nature and ability on degradation under nature conditions.[19] The materials classify into three types according to their reaction with the hydrogen bonds including hydrophobic nature (anti-reactive with water or H-bonds),[20] degraded hydrophilic nature (reactive with water or H- bonds)[21] and Oleophyllic materials, which materials with the capacity to separate oil from water through two different mechanisms : hydrophilic and hydrophobic[22] and PMMA[23] and polystyrene.[24]

There are many previous studies that are interested in biodegradable nanocomposites based polymer matrix that used in different fields, Khdejah et al.[25] released a report in 2018 describing the successful synthesis of polyvinyl alcohol/mixed graphene–carbon nanotubes using an effective solution mixing technique and ultrasonic radiation. A 50%:50% mixed graphene and carbon nanotube ratio. Using the substrate, polyvinyl alcohol polymer, several loadings of mixed graphene–carbon nanotubes (2, 5, 10, 15, and 20 wt%) were added. Thermal degradability, crystallinity, and antibacterial properties were studied. They concluded there is an enhancement of these properties by adding the carbon nano fillers.[25]

Xiong J et al.[26] published a study in 2021 that attempted to improve the PVA/CNT thermal interface characteristics. They created PVA/CNT sheets as a novel thermal interface material (TIM) that they may utilize in their research instead of more traditional TIMs. PVA:vapor-grown carbon fiber (VGCF) mass ratios of 1:0.100, 1:0.070, 1:0.050, 1:0.030, and 1:0.025 were employed to produce five distinct PVA-based CNF sheet types. They deduced that the PVA aggregates connected and arranged the VGCFs in a random sequence. The porosity of the CNF sheets reduced as the amount of VGCFs decreased, as a result of water evaporation creating many pores between the VGCFs. Furthermore, results proved that CNF sheets were produced with an appropriate thickness, low weight, and high elasticity for the practical use of TIM.[26]

Lina et al.[27] published a paper about improving mechanical and antibacterial characteristics of polyvinyl alcohol composite films using cellulose nanocrystals. PVA/ECNC-Qn nanocomposite films were created by blending ECNC-Qn with a PVA matrix using a solvent casting technique. A variety of characteristics were examined, such as the ability to transmit light, mechanical characteristics, and antibacterial activity against both Gram-positive and negative bacteria. The findings of their investigation demonstrated that the PVA/ECNC-Qn nanocomposite films exhibited greater antibacterial activity and tensile strength when compared to pure PVA films.[27]

Yandri et al.[28] published an paper about using the α-Amylase on the recyclability of Bentonite/Chitosan thin films by adoption methods. They concluded the α-Amylase/Bentonite/Chitosan can save it's activity for 6 periodic of recycling process, and the α-Amylase can be reused after freezing process. α-Amylase/Bentonite/Chitosan has more activity than free α-Amylase[28]

Saowanee W.[29] published a paper about effecting of pyrolysis temperature and time on the properties of bio char for reducing the migration changing. The following characteristics of biochar were examined: moisture content (MC), volatile matter (VM), fixed carbon (FC), ash, pH, and the content of carbon (C), hydrogen (H), nitrogen (N), and oxygen (O). These characteristics determine the biochar's efficiency and application potential. With increasing temperature and holding time, the polarity significantly decreased and the aromaticity significantly increased. The results of this study demonstrated that temperature and holding time have a substantial interaction that simultaneously affects the amounts of MC, ash, FC, C,

Table 1. Contents of nano composites thin films samples.

No. of samples	Contents
TH0	Pure PVA
TH1	PVA + 0.001CNTs wt.
TH2	PVA + 0.002 CNTs wt
TH3	PVA + 0.003 CNTs wt

H, N, and O (R2 of 0.997) and pH (R2 of 0.999). The DBC biochar that was produced by pyrolysis for 20 and 60 minutes at 450 and 500 degrees Celsius could be used to mitigate climate change.[29]

The purpose of this research is to develop covers for electronic devices that are biodegradable in the environment, allowing for the proper disposal of broken devices and preventing environmental pollution. To develop biodegradable electronic shells, we attempted to use polyvinyl alcohol as a matrix and carbon nanotubes as reinforcement.

Materials and methods

Poly vinyl alcohol (PVA), a white, soluble powder with a molecular weight of 4000 g/mol, from Sigma-Aldrich USA was purchased and employed for preparing thin films. As a reinforcing material, carbon nanotubes (CNTs) with an average diameter of 40–50 nm were also purchased from Sigma – Aldrich TM and utilized. Table 1 shows the percentage and composition of the synthesized samples used in this work.

Preparation of thin films nanocomposites

Hand casting method was used to prepare the thin film nanocomposites as the following procedure: 20 g of PVA powder was weighed and added to 200 ml of distilled water.to prepare 10% w/v conc. of PVA solution. PVA solution was heated to 80 °C (boiling point of water) with stirred continuously until the fully and homogenous dissolving solution. Then it was left to cool at room temperature gradually. This step was replayed four times to prepare the four samples as shown in Table 1. Additionally, the desired amount of CNT powder, specifically 0.001, 0.002, and 0.003 wt%, was dispersed separately through water using a probe ultrasonic device for 10 minutes at 50 °C for high dispersion process. PVA solutions were then added separately to CNTs/water suspension, and the water was heated and stirred continuously to evaporate the water and return the volume to about 200 ml. To obtain the free bubble solutions, all solutions were left for 24 hours. Then 50 ml of each solution was cast in a square glass dish with $20 * 20\ cm^2$ dimensions for obtaining the thin films nanocomposites. The glass dishes were left for 24 hr for drying, then it left in the oven at under 50 °C for 24 hr to obtain the fully drying thin films. After that, the produced thin film was removed from the dishes and left cool at room temperature. The prepared thin films were cut according to each test sample.

Test and measurements

Thin film nanocomposites were subjected to some tests including the FTIR test to study the bonding of the nanocomposite components using the type (IR Affinity-1), Kyoto, Japan tool. As well as, the contact angle of the nanocomposites thin films were tested via VL 200C - Optical Dynamic I Static Interfacial Tensiometer & Contact Angle Meter,KINO,USA industry. In addition, the biodegradable test was performed via the soil burial to evaluate the degradation rate of the thin films according to the Eq. (1).[30]

$$D.R\,\% = \frac{W_o - W_1}{W_o} * 100\,\% \tag{1}$$

Were D. R % : degradation rate %.
Wo : sample weight before the soil burial.
W1 : sample weight after the soil burial.

The two-dimensional morphology of the nano composite thin films was utilized to examine the surface roughness characteristics using AFM equipment labeled as the Nano Scope IIIA Multi Mode.

Results and discussions

FTIR spectrum analysis

Figs. 1 and 2 show the FTIR analysis of excremental chart for pure PVA and PVA reinforced with 0.002 wt% CNTs and standard chart of pure PVA corresponding to Tellamekala et al.,[31] respectively.

Figs. 1 and 2 demonstrate the excellent agreement between the standard reference scheme in Fig. 2 and the real scheme of polyvinyl alcohol under investigation. The FTIR spectrum of pure PVA, as depicted in Fig. 1, displays many bands that are indicative of the stretching and bending vibrations of the O-H, C-H, C=C, and C-O groups. The linkages that can be seen in Fig. 1. of polyvinyl alcohol diagram are displayed in Table 2.

However, we also see that PVA without CNTs has a higher intensity of transmittance than PVA with 0.002 wt% CNTs. This is because CNTs are opaque materials, and the carbon particles prevent light from passing through to the base material. The absorption values of the polymer rise with the addition of the

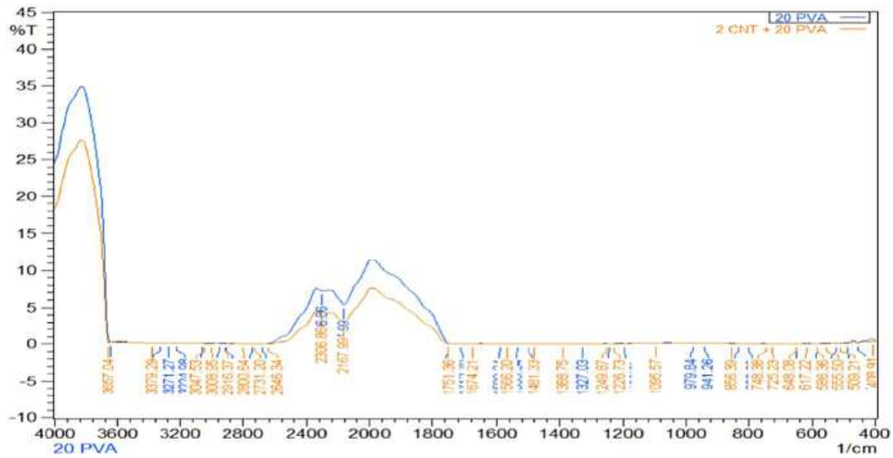

Fig. 1. FTIR analysis of pure PVA and PVA/CNTs.

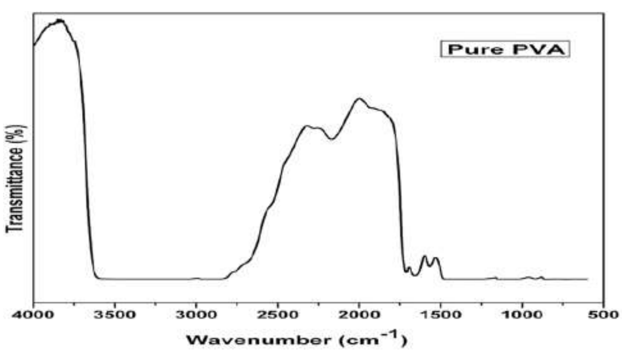

Fig. 2. Standard FTIR spectrum of pure PVA corresponding to Tellamekala et al.[31]

Table 2. FTIR analysis of PVA and PVA+CNTs thin films.

Wave no. cm^{-1} Fig. 1a	Wave no. cm^{-1} Fig. 1b	Bonds	Groups
2800–3600	2813–3601	O-H	Hydrocele
2916	2905	Asymmetry CH_2	
2200, 2400, 2600	2168	CH_2, CH symmetry stretching	
1717	1712	C=O & MWCNT-COOH	Ester
1600	1500	CO stretching	Ester

wavelengths that fall on it, this is matched with Malikov et al.[27] The other specimens exhibit the same general tendency of decreasing permeability intensity with increasing nanotube carbon content, and this is according to Zawawi et al.[32]

Contact angle

Fig. 3 shows the contact angles of the nanocomposites thin films. As shown in Fig. 3, the contact angle of CNTs is approximately 95°, and for PVA it is approximately 42°. The contact angle of CNTs decreases to 56° after being modified with PVA at a weight percentage of 0.001 wt, and it increases to 75 and 78° after being modified with PVA at a percentage of 0.002 and 0.003 respectively. This modification maintains the hydrophilic properties of CNTs/PVA thin films, meaning they can degrade after a limited amount of time in the soil.[33,34]

According to this research, the modification of carbon nanotubes (CNTs) with polyvinyl alcohol (PVA) enhances the degradation of this electronic device following damage. CNTs are a common component that is widely used in electronic devices, but they cannot degrade after damage, especially when modified with hydrophobic substrate or thermosetting materials.[35]

Biodegradability results

Fig. 4 illustrates the nanocomposites samples' biodegradability based on a weight loss test conducted by burying them in soil for seven days (after this period the thin films are cut, broken, and removed of some it's parts in this study). The weight loss values were obtained using Eq. (1).

Fig. 4 shows that in nanocomposite thin films, weight loss percentages increase with longer soil burial times but decrease with greater CNT weight percentages. Following seven days, the TH2 sample lost the most weight—roughly 0.5%. It is comparable to the pure sample TH0, which lost weight at 0.52 percent. After the same periodic, TH3 sample has the lowest weight loss about 0.223 percent. Contact angle for TH2 was about 75 degrees and it was less than 90 degrees (it has hydrophilic properties) and about 78 degrees for TH3 (also it is less than

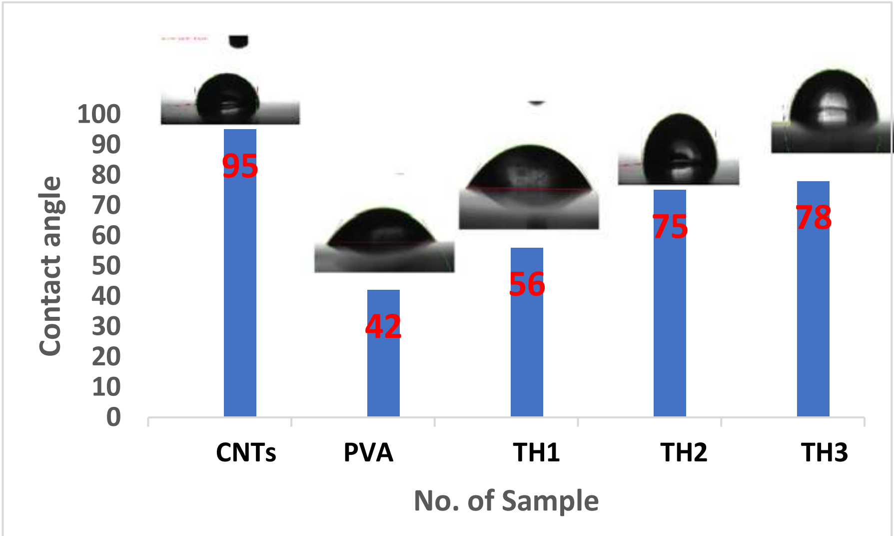

Fig. 3. Contact angles of thin films nanocomposites.

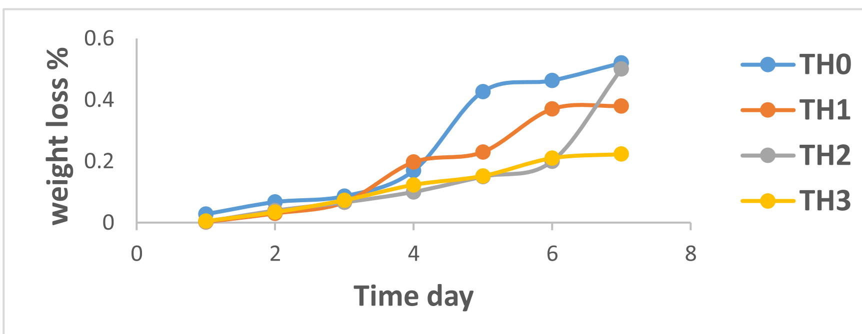

Fig. 4. Biodegradability via weight loss percent of thin films.

90 degrees and it has a hydrophilic characterize). Many factors can affect the contact angle value, including heterogeneity, surface roughness, mechanical properties, crystallinity, porosity, molecule size and shape, It can also differ depending on whether the surface is hydrophilic or hydrophobic. The contact angle typically increases for hydrophilic surfaces (with a contact angle less than 90°). The AFM results of the samples, are displayed in Figs. 5 to 8 and Table 3, support this explanation. We see that the surface roughness decreases as the CNTs content increases, as well as, the crystallinity of samples increases with increasing the CNTs contents as a result to decrease in the grain boundary in the thin film samples and the growth of the molecules.[36,37] Additionally, sample TH3 has the highest bearing index, indicating that it has the best mechanical characteristics and the lowest roughness, which permits a raised contact angle to 78 degrees and a low weight loss percentage. TH2 was the best sample for this test since it had the highest percentage of weight loss. On the other hand, there are many factors according to the type of soil affect on the biodegradable ratio of plastics through as: acidity, humidity, and temperature.[38,39]

2D – analysis by atomic force microscopy (AFM):

Figs. 5 to 8 and Table 3. show the 2D morphology of PVA thin films and it's composites reinforced with (0.001, 0.002, and 0.003) wt% of CNTs respectively.

In the current study, the AFM technique was used to measure the roughness parameters of PVA thin films and their nanocomposites with CNTs in various reinforcement ratios. The above-mentioned roughness parameters are listed fully in Table 3. It can be noted, the comparison of Rave and Rrms makes it abundantly evident that as CNTs are included in the PVA film, the roughness of the surfaces reduces (There is a very small difference in the Rave value for the TH1 and TH2 samples, and it continues to decrease for the TH3 sample), leading to the formation of smaller domains and an accompanying drop in peak height.

Table 3. Roughness parameter analysis via AFM test of all samples.

Sample	Roughness average Rave nm	Sdr (Surface area ratio)	Bearing index	Root mean square Rrms nm	Surface Skewness	Surface Kurtosis
TH0	12	2.85	0.7	15.5	0.336	3.47
TH1	4.32	0.2	2.9	5.68	–0.081	2.9
TH2	4.34	0.127	6.06	4.92	–0.01	1.58
TH3	1.21	0.04	0.8	1.72	0.175	4.21

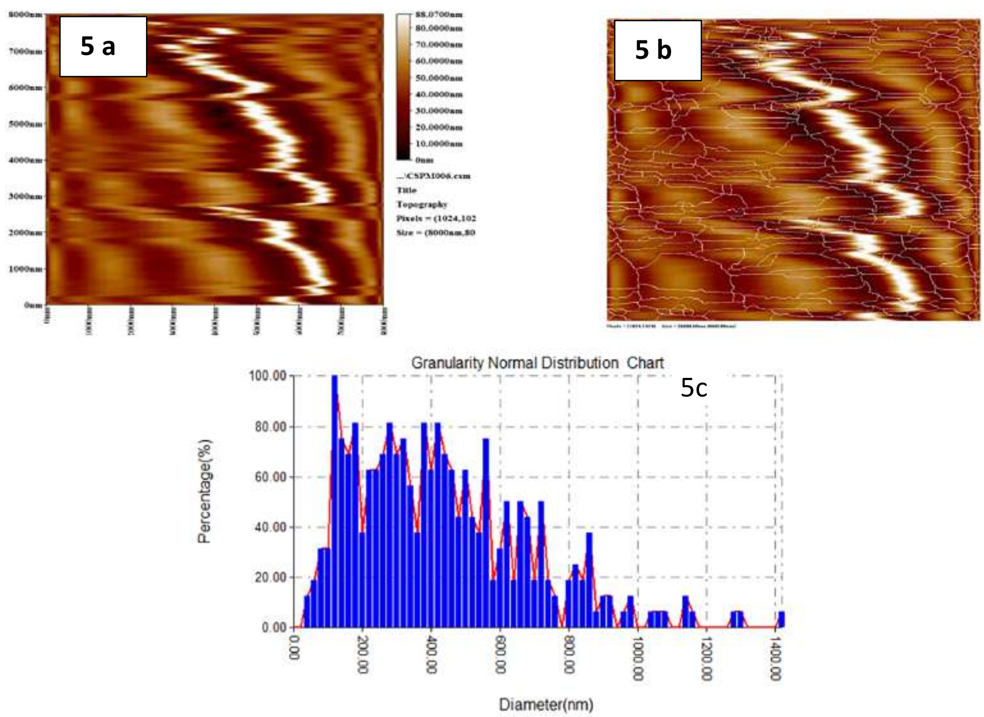

Fig. 5. 2-dimention images of PVA thin film a. morphology b. grain boundaries c. distribution histogram.

Table 3 makes it evident that the surface skewness for PVA film is positive, indicating that the surface is disorganized and has more peaks than valleys (which means it has high roughness, low contact angle and high biodegradability). However, with a high dispersion process of CNTs through PVA, the value of the surface skewness turns negative, indicating that the valleys are prevalent in the surface morphology and that the surface becomes more planar. Comparing Kurtosis moments for various experimental films produced similar types of findings in the current study. For PVA film, the value is significantly higher than 3, and the 2D picture diagram shown in Fig. 5 has more peaks than valleys. Kurtosis nanocomposites have values that are less than 3 for other nanocomposite films, indicating that the surfaces are flat, Figs. 6 to 8. The difference in the coefficient of Kurtosis for PVA and doped PVA films in various CNT ratios indicates that changes to the functional groups will modify the surface chemistry and surface architecture, which will in turn have an impact on the surface energy of both films. The bearing index parameter was shown to increase with the addition of CNTs, and it continued to increase with increasing CNT concentration; this is an indication of the high mechanical loads that can be achieved using CNT-based doping (which means that, increasing the CNTs concentration with high dispersion process leads to high mechanical properties of thin films[40–42] and low biodegradability under other constant conditions of the soil and the time of the burial). The PVA thin film had large grain boundaries; however, as Figs. 5 to 8 illustrates, the addition of CNTs caused the nanocomposite films' grain boundaries to diminish and the molecule sizes to grow. Figs. 5 to 8 display the histogram of the distribution of doped PVA thin films and PVA thin film domains. Reducing the grain boundaries increases prepared thin-film work efficiency, surface energy, electron free mobility in CNTs, and photovoltaic efficiency, this is matched with Abdullah et al.[43]

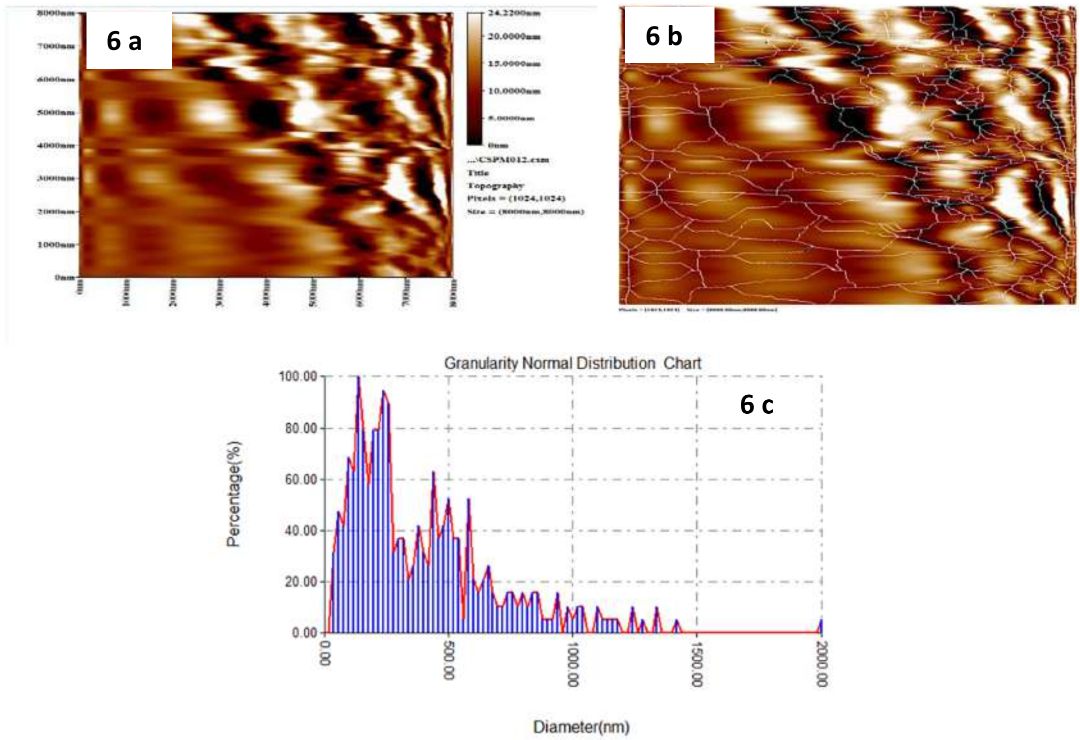

Fig. 6. 2-dimention images of PVA + 0.001 CNTs wt% thin film a. morphology b. grain boundaries c. distribution histogram.

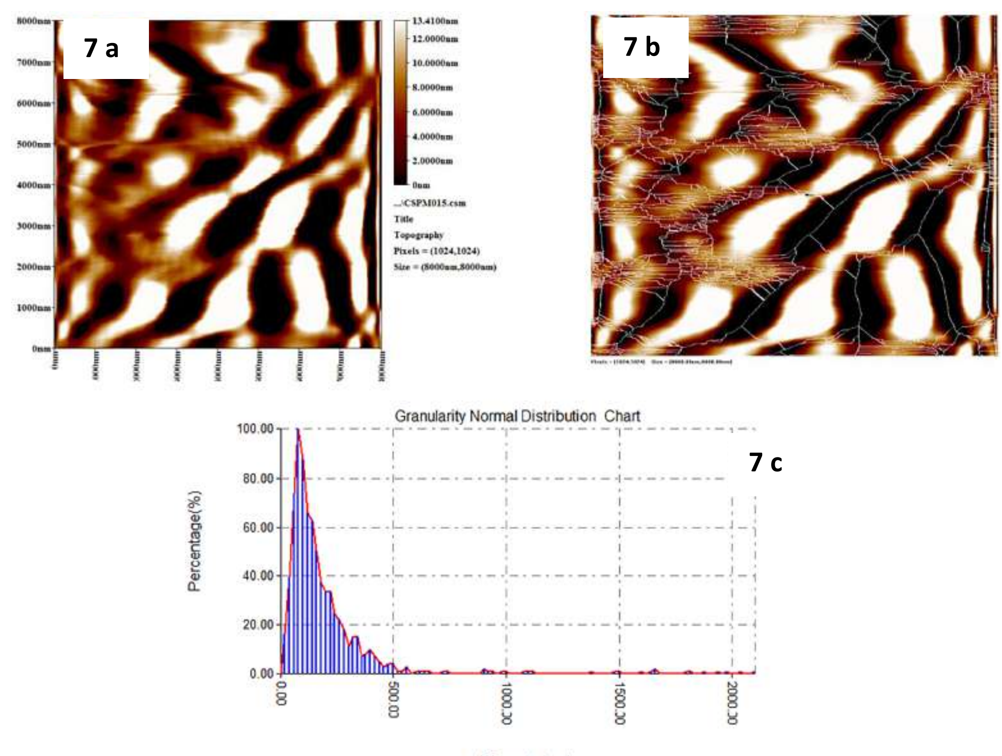

Fig. 7. 2-dimention images of PVA + 0.002 CNTs wt% thin film a. morphology b. grain boundaries c. distribution histogram.

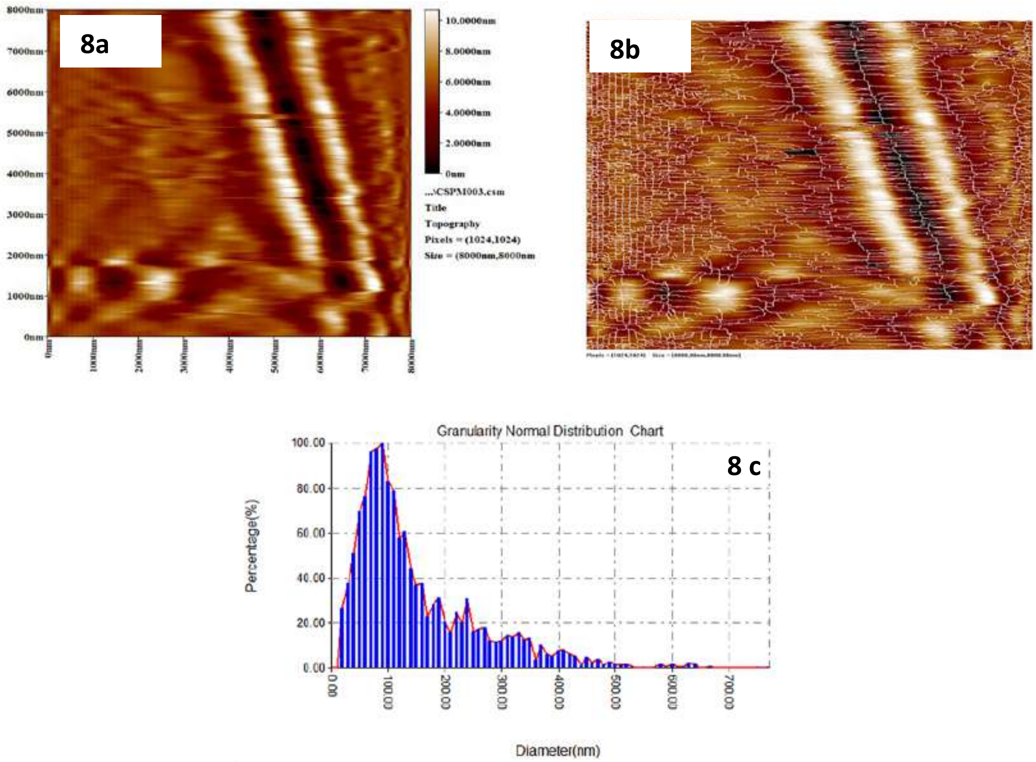

Fig. 8. 2-dimention images of PVA + 0.003 CNTs wt% thin film a. morphology b. grain boundaries c. distribution histogram.

Conclusions

This work concludes that the addition of PVA to carbon nanotubes (CNTs) leads to a decrease in the wetting angle of pure CNTs and a change in their behavior from hydrophobic to hydrophilic. There is no new chemical bonding between PVA and carbon nanotubes, which allows the process of thin film biodegradability under the influence of other factors, such as temperature, age of burial in the soil, random morphology, porosity, and humidity. The wettability angle of nano thin films increases with increasing nanocarbon content and depends largely on the surface properties. The TH2 sample showed the greatest results from this study since it decomposes the fastest through outthe seven-day burial period.

The expensive and non-biodegradable nature of carbon nanotubes is one of the limitations of this study. When other parameters that affect biodegradability, including temperature and decomposition time, are constant, using CNTs in high ratios causes higher product costs and an increased wetting angle, which reduces the capacity of the product to degrade. In future, work, the results could be enhanced by adding some naturally occurring enzymes, like those identified in the O-Glycosyl hydrolases group, that help in the hydrolysis of the glycosidic bond between two or more carbohydrates or between a carbohydrate and a non-carbohydrate component.

The contribution of each researcher

B. J, A. E, H, N. H., and B. M contributed to the design and implementation of the research, to the analysis of the results and to the writing of the manuscript.

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Authors' declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been

included with the necessary permission for re-publication, which is attached to the manuscript.
- No animal studies are present in the manuscript.
- No human studies are present in the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at University of Babylon's.

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أغشية رقيقة من المترااكبات النانوية القابلة للتحلل الحيوي للاغلفة الكترونية القابلة لإعادة التدوير

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المستخلص

في هذه الدراسة تم تحضير الأغشية الرقيقة من المركبات النانوية من كحول البولي فينيل المقوى بنسب مختلفة من أنابيب الكربون النانوية بطريقة الصب اليدوي. يهدف البحث الحالي الى إيجاد ترابط بين تحسين خواص السطح وقابلية التحلل البيولوجي لاغلفة الكترونية عالية الكفاءة . تمت دراسة طبيعة الترابط بين البولي فينيل الكحول وأنابيب الكربون النانوية بواسطة مطيافية الأشعة تحت الحمراء. تم دراسة خواص التبلل بواسطة محلل زاوية الترطيب . تمت دراسة خواص السطح للمتراكبات النانوية باستخدام مجهر القوة الذرية. تم إجراء اختبار قابلية التحلل الحيوي للمركبات النانوية عن طريق دفنها في التربة لمدة سبعة أيام. أظهرت نتائج زاوية الترطيب أن إضافة كحول البولي فينيل إلى الأنابيب النانوية الكربونية يؤدي إلى انخفاض زاوية الترطيب من 95 درجة إلى 56 درجة بتركيز 0.001 بالمائة وزناً للأنابيب النانوية الكربونية واستمرت زاوية الترطيب في الزيادة إلى 78 درجة مع 0.003 بالمائة وزناً للأنابيب النانوية الكربونية. أظهرت نتائج تحليل الأشعة تحت الحمراء أن التفاعل فيزيائي بين أنابيب الكربون النانوية والبولي فينيل الكحول، وكذلك فإن شدة نفاذية الأشعة تحت الحمراء تقل مع إضافة أنابيب الكربون النانوية. أثبتت نتائج الفحص المجهري للقوة الذرية أن متوسط الخشونة يتناقص مع زيادة نسب الأنابيب الكربونية النانوية من 0.001 إلى 0.003 نسبة وزنية، كما أن مؤشر التحمل يزداد بزيادة تراكيز أنابيب الكربون النانوية، كما أن تسطيح السطح يزداد بزيادة نسب أنابيب الكربون النانوية.. أفضل عينة لهذه الدراسة كانت للنموذج المسلح بنسبة 0.002 من انابيب الكاربون النانوية.

الكلمات المفتاحية: اغشية رقيقة ، انابيب الكاربون النانوية ، بولي فينيل الكحول، تحلل احيائي، زاوية التبلل ، متراكبات نانوية.