# Chemical synthesis, surface characterization, and electrical properties of nanocomposite films for dielectric applications

H. A. Al-Yousef<sup>a</sup>, B. M. Alotaibi<sup>a</sup>, M. M. Abdelhamied<sup>b</sup>, A. Atta<sup>c</sup>, E. Abdeltwab<sup>c,\*</sup>, M. F. Alotiby<sup>d</sup>, S. F. Alarfj<sup>a</sup>

<sup>a</sup> Department of Physics, College of Science, Princess Nourah bint Abdulrahman University, P.O. Box 84428, Riyadh 11671, Saudi Arabia

<sup>b</sup> Radiation Physics Department, National Center for Radiation Research and Technology (NCRRT), Egyptian Atomic Energy Authority (EAEA), Cairo, Egypt <sup>c</sup> Physics Department, College of Science, Jouf University, P.O. Box: 2014, Sakaka, Saudi Arabia

<sup>d</sup> Nuclear Technologies Institute (NTI), King Abdulaziz City for Science & Technology (KACST), P. O. Box 6086, Riyadh, 11442, Saudi Arabia

This work used the solution preparation casting method to fabricate the composite (CA/PANI), which consisting of conducting polymer polyaniline (PANI) and cellulose acetate (CA). The effects of PANI on the surface, electrical and structural characteristics of CA are studied. The XRD confirmed the successful fabrication of the composite CA/PANI films. Additionally, the surface morphology of nanocomposite films shows that the PANI nanoparticles are spatially scattered and shown in a random arrangement. The films of CA and PANI/CA were also tested for surface free energy, adhesion work, and contact angles. After increasing the PANI concentration in CA films from 10% to 20%, the contact angle of water dropped from 74.75° to 67.25°, and the work of adhesion increased from 91.02 to 99.82 mJ/m<sup>2</sup>. Also, as the PANI content grows from 10% to 20%, the total surface energy increases from 28.42 to 40.22 mJ/m<sup>2</sup>, the polar free energy increases from 11.64 to 14.06 mJ/m<sup>2</sup>, and the dispersive free energy increases from 21.87 to 26.16 mJ/m<sup>2</sup>. Additionally, electrical conductivity was tested between 50 and 10<sup>6</sup> Hz. With the addition of PANI, the conductivity increased from  $4.5 \times 10^{-8}$  S/cm for CA to  $1.8 \times 10^{-7}$  S/cm for CA/20%PANI. The obtained data show the composite CA/PANI improved in the electrical and surface properties, which is significant for dielectric applications.

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#### 1. Introduction

Polymer composites have many advantages over more conventional ceramic dielectrics, such as being malleable and easy to shape [1, 2]. Polymer composites is important for electrical uses and electronic devices that demand materials with a high dielectric constant, such as transistors and capacitors [3]. Wearable and flexible electronics often make use of polymer composites because of their excellent dielectric characteristics and mechanical flexibility [4]. Electronic equipment can be equipped with EMI shielding using composites that contain conductive fillers. They find usage as sensors because of their dielectric responsiveness and pliability [5].

The electrical characteristics of polyaniline, a conductive polymer, can vary depending on its oxidation state [6]. PANI is well-suited for a range of uses due to its relative stability under ambient conditions [7]. Although PANI is insoluble in most solvents when unmodified, it can be made more processable through doping and other changes. Because of its strong polarization in an electric field, PANI has excellent dielectric characteristics and finds usage in dielectric applications. PANI's redox activity and conductivity make it useful in chemical and biosensors, make it an ideal dielectric material for capacitors [8]. Electronics and telecoms benefit from PANI composites' ability

<sup>\*</sup> Corresponding author: eaelsayed@ju.edu.sa https://doi.org/10.15251/DJNB.2025.201.159

to reduce electromagnetic radiation. PANI is beneficial for protective coatings due to its conductivity, which aids in dissipating static charges [9].

Cellulose acetate (CA) is eco-friendly and biodegradable since it is made from natural cellulose. The resulting films are both mechanically strong and flexible, which can be used in the storage devices [11]. When subjected to mild chemical and thermal environments, CA remains stable and performs admirably as a dielectric [12]. Because of its high dielectric strength and excellent dielectric characteristics, CA finds use in dielectric applications [13]. One advantage of processing CA is dissolves in organic solvents such as acetone and methylene chloride [14]. CA is utilized in filtration membranes because of its mechanical qualities and transparency, can be applied in optical devices and coating applications [16]. The improved of the adhesion properties of CA, can be useful in applications such as membrane filtration, biomedical devices, and sensors [17]. Increasing the electrical conductivity of cellulose acetate is a key motivation for adding PANI to the material. The insulating properties of pure cellulose acetate are enhanced by the presence of a conductive polymer PANI, allowing it to display higher electrical conductivity [18].

Fabricating nanocomposites with novel characteristics to be applied in a range of devices is the focus of this work. Polyaniline is an ideal material for sensors and capacitors due to its dielectric characteristics and adjustable conductivity. Many different kinds of electrical and energy storage devices can benefit from polymer composites because of their malleability in terms of dielectric characteristics. The CA/PANI composites used in this study were made via solution casting and then analyzed by XRD and SEM. The composite CA/PANI samples will be employed for a variety of coating, printing, and supercapacitors applications after the effects of PANI on their surface characteristics and conductivity are examined.

# 2. Experimental work

The materials used are Cellulose acetate (CA; MW 58.700 g/mol), acetone (98.7) and hydrochloric acid (35%) were purchased from the company of Sigma–Aldrich. Aniline, ammonium per-oxide-sulfate (99%) and N,N-dimethylformamide (DMF) were obtained from Merck Company (Germany). Firstly, we prepared the PANI by the chemical polymerization reactions of the aniline monomer [19]. The aniline monomer has been polymerized in presence of hydraulic acid (HCl) that used as a Catalyst material and ammonium persulfate (APS) as an oxidizing agent. After that, these PANI solutions have been mixed with CA using the magnetic stirring for 2 h, then were gush in petri dish and left for 3 day to dry. Finally, we obtained PANI/CA nanocomposite films with different ratio of PANI, which labeled as (PANI/CA)-1, (PANI/CA)-2, (PANI/CA)-3.

The XRD of the produced films, including their crystalline structure, was obtained using Cu Ka radiation at a wavelength of 0.15406 nm. Using a Hitachi S-4800 FESEM (Tokyo, Japan), the SEM analyses were carried out. Before being placed on a scanning electron microscope (SEM) stand, samples were manually sliced with a scalpel, covered with two-sided carbon tape, and inflated with pressured air. With the use of an RLC bridge (3532 LCR) operating in frequency between 50 Hz and 5 MHz, the conductivity was measured.

#### 3. Results and discussions

Figure 1 exhibits the XRD patterns of the pristine CA film, and PANI/CA nanocomposite films. The pattern of pristine CA film appeared two main broad peaks at 2 theta  $8.56^{\circ}$  and  $17.40^{\circ}$ , which agrees with the other pattern [20]. The broad peak at  $2\theta$  is  $8.56^{\circ}$  indicates the semi-crystalline acetylated of the CA film [21]. The pattern of PANI powder is exhibited three peaks are located at two theta approximately of  $13.86^{\circ}$ ,  $20.31^{\circ}$ ,  $25.15^{\circ}$ , which are related to the crystal planes of 121, 113, and 322, confirming the semi crystalline structure of PANI. The observed data concede with the Diffraction Standards of PANI Powder (no. 72-0634) [22]. The crystallite size (L) of PANI powder is deduced from the Scherer expression [23].

$$L = \frac{0.9\,\lambda}{\mathrm{K}\cos\theta} \tag{1}$$

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It is confirmed that crystalline size of the PANI is 5.93, 6.9, and 6.42 nm for the 121, 113, and 322 crystal planes. Additionally, it is seen that the patterns of (PANI/CA)-1 and (PANI/CA)-3 present some distinct in comparison with the model of the pristine CA. In which, the peak intensity of the pristine CA reduces after insertion the PANI as shows in the (PANI/CA)-1 pattern, which more decreases for the (PANI/CA)-3 pattern. Moreover, this result proves that the crystalline degree of the pristine CA film decreased after addition the PANI. To confirm that, the area under peaks was calculated. It is obvious that the area under peaks at 8.56° and 17.40° for the pristine CA was 1156 and 2628, respectively, which was increased to 1547, 2798 and 2657, 3237 of the (PANI/CA)-1, (PANI/CA)-3 nanocomposite films. The decrease in crystalline degree affirms the ionic crosslinking that raises between the PANI and CA [24]. Meanwhile, the peak position of the controlled CA also was shifted after introducing the PANI, which increased with the increase in ratio of the PANI material. These results supported that PANI was loaded onto the matrix of CA [25]. Furthermore, this concluded record the increase in charge carrier's concentrations of the nano composite films [26], which confirm improving the electrical properties of the PANI-loaded CA samples.



Fig. 1. XRD patterns of the pristine CA film, PANI, (PANI/CA)-1, and (PANI/CA)-3.

Furthermore, the variation in the surface morphologies of the pristine film after impeding the nano-filler was detected by studying the SEM images of samples. Figs. 2(a-d) depict the SEM images of the pristine CA and three nanocomposite films of PANI- loaded CA, respectively. The morphology of the pristine CA appears smooth and compact. This smooth and homogeneity manifest that CA surface is exempt from non-cellulosic hard components including the lignin and other contaminants. From Figs. 2(b-d), PANI nanoparticles display granule shapes with highly agglomerated, which reveals the amorphous structure of PANI and strong interaction between CA and PANI nanoparticles [27]. The surface morphology of nanocomposite exhibits that PANI nanoparticles are very dispersed and arranged randomly. In which, the high amount of PANI concentrations leads to cellulose acetate surface has been full coated by PANI. The homogeneity of PANI dispersion and its morphology within the cellulose acetate matrix also play critical roles. The surface morphology of nanocomposite films also exhibits that PANI nanoparticles are very dispersed and arranged randomly owing to the high amount of PANI content in the nanocomposite films, which leads to that cellulose acetate surface has been coated by PANI.





Fig. 2. SEM images of the (a) CA, (b) CA/10%PANI, and (c) CA/15%PANI, and (d) CA/20%PANI.

As shown in Figure 3, the contact angle drops with increasing PANI concentrations because functional groups coalesce at the hydrophobicity interface between CA and PANI. The contact angle of water dropped from 74.75° to 67.25° and for diiodomethane reduced from 71.82° to 64.21° as PANI increased from 10% to 20%, as demonstrated in Table 1. In comparison to pure CA films, CA/PANI compounds have a reduced contact angle by the addition of PANI. [28]. The conductive polymer polyaniline (PANI) drastically alter the surface characteristics of the CA polymer. The composite materials are high-tech uses because PANI when mixed with cellulose acetate, drastically lowers the contact angle and changes the surface wettability by boosting hydrophilicity [29].



Fig. 3. Contact angle for the composite CA/PANI films at different content of PANI.

The work of adhesion  $(W_a)$  is estimated by [30].

$$W_a = \gamma_l (1 + \cos\theta) \tag{2}$$

The relationship between the  $W_a$  and the growing PANI content is seen in Figure 4. According to Table 1, when the PANI in CA is enhanced from 10% to 20%, the  $W_a$  for water liquid enhanced from 91.02 to 99.82 mJ/m<sup>2</sup>, and for diiodomethane it increases from 66.05 to 72.90 mJ/m<sup>2</sup>. Variations in surface hydrophilicity induced changes in the adhesion work over a range of liquid polarities [31]. The interaction with liquids changes the surface adhesion work. The adhesion work can be fine-tuned to create certain surface qualities by adjusting the concentration and distribution of PANI. Because of their enhanced hydrophilicity, PANI/CA composites may find more use in drug delivery systems and other biomedical applications that rely on cell adhesion and proliferation. The development of a covalently linked network strengthens interfacial adhesion and increases the work of adhesion when PANI induce cross-linking in polymers.

Table 1. The contact angle for the composite CA/PANI films at different content of PANI.

The samples	Contact angle (degree)		Work of adhesion (mJ/m <sup>2</sup> )	
	Water	Diiodomethane	Water	Diiodomethane
CA	81.60	76.34	82.63	62.83
CA/10%PANI	74.75	71.82	91.02	66.05
CA/15%PANI	70.50	68.26	96.04	69.65
CA/20%PANI	67.25	64.21	99.82	72.90



Fig. 4. The work of adhesion for the composite CA/PANI films at different content of PANI.

The following equation is used to find the unknown value of the total  $(\gamma_s^t)$ , polar  $(\gamma_s^p)$  and dispersive  $(\gamma_s^d)$  surface energies of CA and CA/PANI by relating the surface tensions of solid-liquid  $(\gamma_{sl})$ , solid-vapor  $(\gamma_s)$ , and liquid-vapor  $(\gamma)$  interfaces [32].

$$\frac{\gamma_{l}(1+\cos\theta)}{2\sqrt{\gamma_{l}^{d}}} = \sqrt{\gamma_{s}^{d}} + \sqrt{\gamma_{s}^{P}} \cdot \sqrt{\frac{\gamma_{l}^{P}}{\gamma_{l}^{d}}}$$
(3)

Figure 5 displays the variations in dispersive  $\gamma_s^d$ , polar  $\gamma_s^p$ , and total  $\gamma_s^t$  for CA and CA/PANI with varying PANI contents. Table 2 shows when PANI increased from 10% to 20%, the  $\gamma_s^p$ ,  $\gamma_s^d$ , and  $\gamma_s^t$  all increased from 11.64 to 14.06 mJ/m<sup>2</sup>, and from 21.87 to 26.06 mJ/m<sup>2</sup>, and from 33.51 to 40.22 mJ/m<sup>2</sup>, respectively. There is a direct correlation between the concentration of PANI and the activation of the composite surface, as shown here [33]. Cellulose acetate's surface wettability can

be changed by adding PANI, which usually makes the material more hydrophilic [34]. This shift occurs because PANI contains polar functional groups like amines and imines, which can reduce the surface contact angle of water by forming hydrogen bonds with water molecules [35].



Fig. 5. Polar, dispersive and total surface free energies for CA/PANI at different content of PANI.

Table 2. The polar, dispersive, and total free energy for CA/PANI at different content of PANI.

The samples	Polar $\gamma_s{}^p$ (mJ/m <sup>2</sup> )	Disoersive $\gamma_s^d (mJ/m^2)$	Total $\gamma_s^t$ (mJ/m <sup>2</sup> )
CA	8.90	19.42	28.42
CA/10%PANI	11.64	21.87	33.51
CA/15%PANI	13.19	23.88	37.07
CA/20%PANI	14.06	26.16	40.22

The conductivity  $\sigma_{ac}$  is estimated by [36].

$$\sigma_{ac} = 2\pi f \varepsilon_o \varepsilon'' \tag{4}$$

The frequency relation with the  $\sigma_{ac}$  values for both the pure CA and composite CA/PANI are shown in Figure 6, in frequency range of 50Hz to 5MHz. By increasing the PANI content, the conductivity at frequency 50 Hz, is enhanced from  $4.5 \times 10^{-8}$  S/cm for CA to  $6.5 \times 10^{-8}$  S/cm for CA/10%PANI, and to  $1.1 \times 10^{-7}$  S/cm for CA/15%PANI and to  $1.8 \times 10^{-7}$  S/cm for CA/20%PANI. This because the increase of PANI in the CA, enhancing the speed of ionic transit via the polymer chain scissoring, which directly rise the conductivity for the composite [37].



Fig. 6.  $\sigma_{ac}$  with frequency for the polymer CA and the composite CA/PANI.

The shortened polymer chains may lead to better electron transfer between the polymer and filler, thereby enhancing conductivity.

The following equation is estimating the potential barrier height  $W_m$  [38].

$$W_m = \frac{-4k_BT}{m} \tag{5}$$

The k<sub>B</sub> is Boltzmann constant, and m is determined from the slope of Ln ( $\varepsilon''$ ) versus Ln ( $\omega$ ), as seen in Figure 7 using the next relation [39]

$$\varepsilon'' = A\omega^m \tag{6}$$

In the case of pure polymer CA, the calculated  $W_m$  changed from 0.193 eV for CA to 0.201 eV for 10% PANI, 0.209 eV for 15% PANI, and 0.223 eV for 20% PANI. Because of the synergy between CA and PANI, the electrical conductivity can be dramatically increased when the two substances are mixed. PANI is a conducting polymer [40]. Conductive battery separators made of composite CA/PANI are great because of the mechanical strength and improved ionic conductivity by the combination of CA flexibility and PANI's conductive properties. The charge carrier mobility is improved due to better alignment, which permits electron delocalization along the polymer backbone. The conductivity is improved because the energy barriers for charge transfer are reduced due to the greater delocalization.



Fig. 7. Ln ( $\varepsilon''$ ) with Ln ( $\omega$ ) for the polymer CA and the composite CA/PANI films.

## 4. Conclusions

The solution casting preparation process was used to generate nanocomposites of flexible CA and PANI polymers. The CA/PANI composite was successfully prepared as analyzed by the SEM and XRD methods. The surface morphology of nanocomposite exhibits that PANI nanoparticles are very dispersed and arranged randomly. By determining the contact angle, the surface energy and the adhesion force were estimated. Raising the PANI content from 10% to 20% raises the polar energy from 11.64 to 14.06 mJ/m<sup>2</sup> and the dispersion energy from 21.87 to 26.16 mJ/m<sup>2</sup>. Also, the enhancing of PANI ratio creates a cohesive bond on the CA/PANI chain, which improves the surface adhesion of the two compounds. By increasing the PANI content, the conductivity at frequency 50 Hz, is enhanced from 4.5x10<sup>-8</sup> S/cm for CA to 6.5x10<sup>-8</sup> S/cm for CA/10%PANI, and to 1.1x10<sup>-7</sup> S/cm for CA/15%PANI and to 1.8x10<sup>-7</sup> S/cm for CA/20%PANI. This because the increase of PANI in the CA, enhancing the speed of ionic transit via the polymer chain scissoring, which directly rise the conductivity for the composite The charge transfer performance is enhanced by including conductive PANI into the CA matrix. The fabricated CA/PANI nanocomposite sheets, as shown by the results, can be used in microelectronic devices and supercapacitors.

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