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Investigation of Wettability and AC-Conductivity of EVA/PANI Composite

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Abstract: In the current work, EVA/PANI composite is successfully prepared by in-situ technique. Both prepared materials are characterized by XRD, FTIR, UV-Visible, and SEM analyses. Wettability and dielectric properties of both EVA film and EVA/PANI composite are studied. High intense peak is found in EVA/PANI composite and is indicated more crystalline nature compared to pristine EVA polymer film. π - π^* and polaron transitions are appeared in the UV-Visible spectrum of EVA/PANI composite. Different absorption band of EVA film and EVA/PANI composite are viewed in their spectrum. A study has been made of wettability of smooth surfaces of EVA and HCl doped EVA/PANI materials by observing the equilibrium contact angle formed by a liquid drop resting on the solid surface. Wettability results of EVA/PANI composite shows hydrophilic nature. Frequency dependent AC electrical conductivity and Dielectric are analyzed for both materials.

Keywords: Ethylene Vinylene Acetate, Solution Processing, Polyaniline Salt, Morphology, Structure, Wettability, Dielectric

1. Introduction

Most effective way to develop new polymeric material is blending methods. It is economical viable. Developed materials have unique properties from those individual constituent [1, 2]. Still, most polymer blends are showing poor properties due to their immiscible nature. For enhancing the performance, different methods are adapted i.e., block or grafting method [3, 4]. Particularly, functional polymers show better adhesive properties due to occurrence of functional groups in the backbone. It is termed as polar materials. In technological applications, blending of functionalized polyolefin and maleic anhydride (MA) is showed better adhesive property in melt processing. Melt processing is carried out in extruder or mixing chamber at elevated temperature [5].

EVA is established due to easy fabrication by injection molding, low density, high chemical resistance, cost-effective, etc. Also, it has poor in shrinking ability during compression. [6]. Characteristics of EVA depends on content of VA. Low density EVA is similar to low density polyethylene (LDPE), while high density EVA is like rubber.

Some reports are available on blends of EVA copolymer. EVA/metallocene-catalyzed ethylene- α -

olefin copolymers blends are mainly studied on different properties such as rheology, thermal, mechanical, and adhesive [7]. Blends of EVA copolymer and metallocene-catalysed low density polyethylene are reported on processability, phase, morphology, and melt viscoelastic properties [7-9]. Extruded foam profiles of LDPE/EVA blends are studied on static and dynamic mechanical properties, creep recovery behavior, thermal expansion, and thermal conductivity [10]. EVA/VA content (28, 22, and 15%) with ethylene- α -olefin copolymers blends (α belongs to 1-butene and 1-octene) is reported. The blends show enhancement of thermal, viscoelastic, rheological, morphological and mechanical properties.

Conducting polymers, particular, polyaniline cannot be used as dielectric materials. Reason may be due to their rigidity and brittleness. Furthermore, dielectric properties of these polymers changes low to high value from low to high frequency regimes. Dielectric value is not computable because high conductivity value at lower frequency regions. So, conducting polymer composite comprising insulating polymer matrix and can potentially use. One of conducting polymer is polyaniline, which has low cost monomer, easy synthesis method, and used potentially in different applications [11]. Dielectric properties of

those composite materials depend on polymerization process, temperature, concentration, distribution and dispersion, interfacial attraction between filler and polymer matrix [12]. Previously, some authors are reported polymer matrices/PANI composite, which is prepared by in situ emulsion polymerization process [13, 14]. Jiongxin et al. (2007) have prepared in situ synthesized PANI/ epoxy matrix (insulating) composite and are studied dielectric and electrical properties of the prepared materials [15].

Now a day, polymer/polymer-based composites is used potentially in different electrical and electronic applications if compared with other ceramics/metals/polymeric materials. Reasons behind are lightweight, flexible in nature, and easy to prototype [16]. Dielectric properties i.e., dielectric constant of insulating polymers are quite low. Therefore, insulating polymers will not use dielectric pressure sensors.

Demand of heterostructures i.e., composite materials specially, dielectric application, is needed insulated polymer. Conductive additives are significantly increasing demand during composite material preparation. It is used in aerospace, electrical, and electronic fields such as shielding enclosures, capacitors, sensors, microwave absorber, Electromagnetic Interference Shielding, Dielectric Sensor, Supercapacitors, etc. [17-23]. Dielectric material can be successfully used for sensing purpose. Some literatures are available on dielectric sensors materials [24-29].

Objective of the work is to prepare an environmentally stable PANI-ES composite that would show better electrical properties and, therefore, can be potential for gas sensor and other electronic applications. We selected EVA rubber in the composite preparation because of its high mechanical properties and low cost compared to other types of polymer and strong interaction with ionic electroactive polymer such as PANI. We are studied ATR-FTIR, UV-Visible and morphological analysis, wettability, and dielectric behaviour measurement at room temperature of HCl doped EVA/PANI-ES composites.

2. Experimental Details

2.1 Chemicals and Materials

Reagent grade chemicals such as aniline liquid, ammonium persulfate, hydrochloric acid, diethyl ether, chloroforms are procured from Merck, India. Ethylene-vinylene acetate (EVA) is used as base materials, which is purchased from Merck India. This is used during the preparation of EVA/PANI composite.

2.2 Preparation of Composites

HCl-doped EVA/PANI-ES composite is prepared by in situ technique and is discussed below;

2.2.1. Preparation of Eva Films

EVA films are prepared by solution processing followed by casting technique [30]. In the process, both EVA and CHCl_3 as polymer and solvent are used. EVA (2 g) is added to 20 mL of CHCl_3 in a 100 mL beaker and stirred for 3 h at room temperature. Transparent soluble product is poured into a petridish (diameter 10 cm) and left it for the solvent evaporation. EVA film formed after solvent evaporation. Such films removed from petridish and cut into small pieces for characterizations.

2.2.2. Preparation Hcl-Doped Eva/Pani-Es Composite

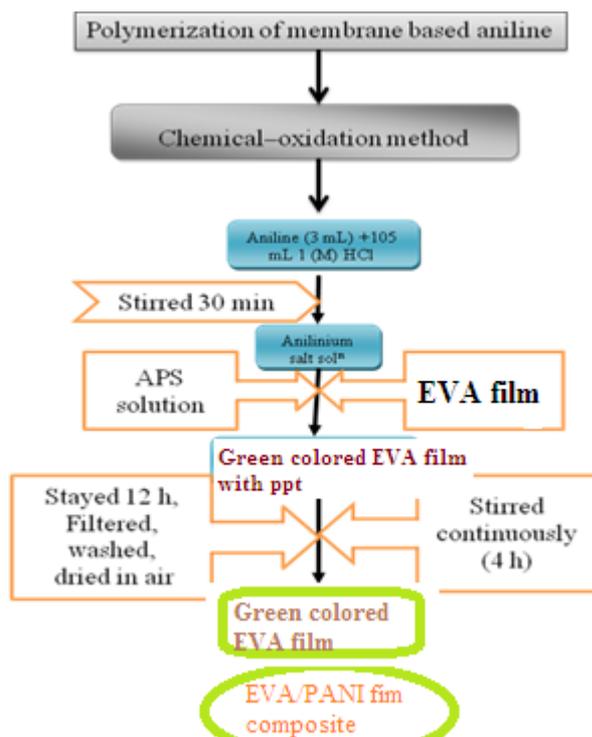
EVA/PANI-ES composites are prepared by *in situ* technique via chemical-oxidation polymerization method [12]. The polymerization reaction is carried out at room temperature. During the composite preparation both aniline and EVA film (1 cm^2) are taken as monomer and base material, respectively. In the polymerization process, two different types' solutions are required and are also prepared. 3 mL of aniline is added to 105 mL of aqueous 1 M HCl. It is stirred continuously for 0.5 h and slightly yellowish solution is formed. Such solution is known as monomer solution or solution 1. After that, EVA film (1 cm^2) is dipped in solution 1 and stirred for 12 h. Solution 2 is called oxidant solution. It is prepared by adding 7.47 g of oxidant (i.e., ammonium persulphate, APS) in 60 mL of 1 M HCl solution. Then, oxidant solution is added drop wise to the EVA mixed monomer solution (i.e., solution 1) for 1 h. The polymerization is occurred. It is observed that EVA film Color is changed from white to light green and then, finally to dark green. This is happened during the continuation of polymerization reaction. The reaction mixture is continued the stirring (for 10 h) to complete the polymerization reaction. Resulted HCl doped EVA/PANI-ES composite is finally picked out from the reaction mixture. It is washed with deionized water several times and dried in the ambient atmosphere. The schematic representation of whole preparation process of HCl doped DL-PLA/PANI-ES composites is presented in Scheme 1.

2.3 Characterization Techniques

X-ray diffraction partten

EVA and HCl doped EVA/PANI Composite are tested by X-ray diffraction (Phillips PW-1710 advance wide-angle X-ray diffractometer, Phillips PW-1729 X-ray generator, $\text{CuK}\alpha$ radiation, $\lambda = 0.154 \text{ nm}$). Film samples are taken to do the test.

Attenuated total reflectance Fourier transformation infrared (ATR-FTIR) spectra are recorded on a Thermo Nicolt Nexus 870 spectrophotometer in the range of $400\text{-}4000 \text{ cm}^{-1}$.



Scheme 1. Flow chart of whole preparation process of HCl doped EVA/PANI-ES composite

Both (EVA and HCl doped EVA/PANI Composite) materials are films.

Ultra-violet visible (UV-Vis) spectrum of HCl-doped EVA/PANI-ES composite film is recorded (Micropack UV-VIS-NIR, DH 2000 in the wave length region 200-1000 cm⁻¹).

Surface morphology of EVA, PANI-ES, and HCl-doped EVA/PANI-ES composite are tested by scanning electron microscopy (SEM, Carl Zeiss Supra 40 scanning electron microscope). Gold coating is required and is done before the SEM experiment.

Contact angle is determined on the substrate a layer and is positioned in the skinny layer. A distilled water drop is put on the substrate. Silhouette of drop is viewed in a digital camera, which is attached to personal computer (PC). Contact angle is estimated through Image analyze software. Attitude is determined 1 second after the drop, which is positioned on the substrate surface.

Electrical properties are measured using LCR meter at room temperature. Frequency dependent AC conductivity (σ_{ac}) is estimated by following relation [30];

$$\sigma_{ac} = \frac{t \times V_r \cos\left(\frac{\phi}{\pi} \times 180\right)}{RA} \dots\dots\dots(1)$$

Dielectric constant (k) is determined using the expression [30] as

$$\epsilon = \epsilon_0 \left(\frac{C \times d}{A} \right) \dots\dots\dots(2)$$

Here; C = capacitance of the pellet, d = thickness of the pellet (2.5 mm), A = cross sectional area of the flat surface of the pellet (100 mm²) and ϵ_0 the constant of permittivity of free space.

3. Results and Discussion

Occurrence of polyaniline slat (PANI-ES) on EVA film in the can further be confirmed by XRD studies (Figure 2). Superimposed XRD pattern of EVA and EVA/PANI-ES are shown Figure. 2. Major XRD peak of EVA shows at 21° and three more weak reflection peaks, indicating the amorphous nature of the EVA polymer [31, 32], whereas XRD peak of EVA/PANI-ES reveals at 21°. It shows the additional crystalline order or the formation of new crystalline phase. This is due to the presence of PANI-ES on EVA film. XRD peak is measure of crystallinity of a polymer. In a polymer, greater is the degree of regularity in arrangement, higher is the crystallinity [33].

Figure 3 shows FTIR spectra of EVA [31], EVA/PANI-ES composite. Important characteristic stretching frequency of EVA is found to be 3441 and 2919 cm⁻² and is designated as methyl group stretching vibration and symmetric/anti-symmetric CH₂ stretching band, respectively. The Stretching band at 2848 cm⁻² is assigned to the Bands at 1464 cm⁻². Band at 1644 cm⁻² is associated with trans-vinylene double bond. Stretching band is observed at 1743 and 1238 cm⁻².

Appearance of such band is due to carbonyl stretching and carboxyl stretching [34]. Band of EVA/PANI-ES composite are appeared at 579 cm^{-2} and is the stretching band of EVA/PANI-ES bond stretching. However, the spectrum of EVA with PANI-ES shows almost all the characteristic peaks of both EVA and PANI-ES. Pragmatic changes of stretching band in the spectrum of composites are due to interaction of polyaniline EVA polymer. Carbonyl peak is found at 1743–1723 cm^{-2} , which is indicated interaction between PANI-ES and vinyl acetate segment of EVA.

Mainly, PANI-ES has shown π - π^* transition (for benzene ring) and polaron to π^* (for benzenoid to quinoid ring and polaron transition), respectively [35]. There is no π - π^* transition in pristine EVA film [36]. Different transitions are found in HCl doped EVA/PANI-ES composites. Figure 3 show two types of bands i.e., π to localised polaron band and π - π^* band of benzenoid ring for HCl-doped EVA/PANI-ES composite. Both bands are proposed to the occurrence of aniline unit and oxidation unit [35, 36]. Figure 4 shows the absorption spectrum.

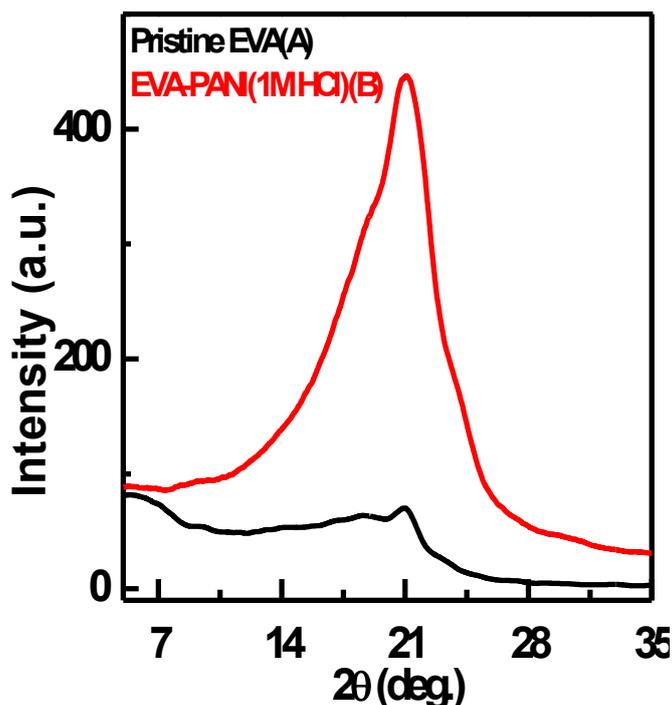


Figure 2. XRD pattern of EVA film and EVA/PANI (1M HCl) film

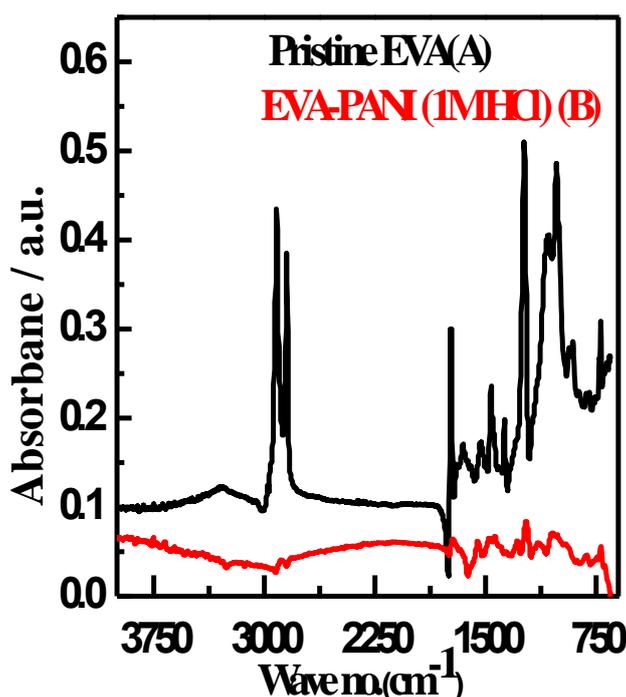


Figure 3. FTIR of Pristine EVA film (A) and EVA/PANI (1M HCl) film (B)

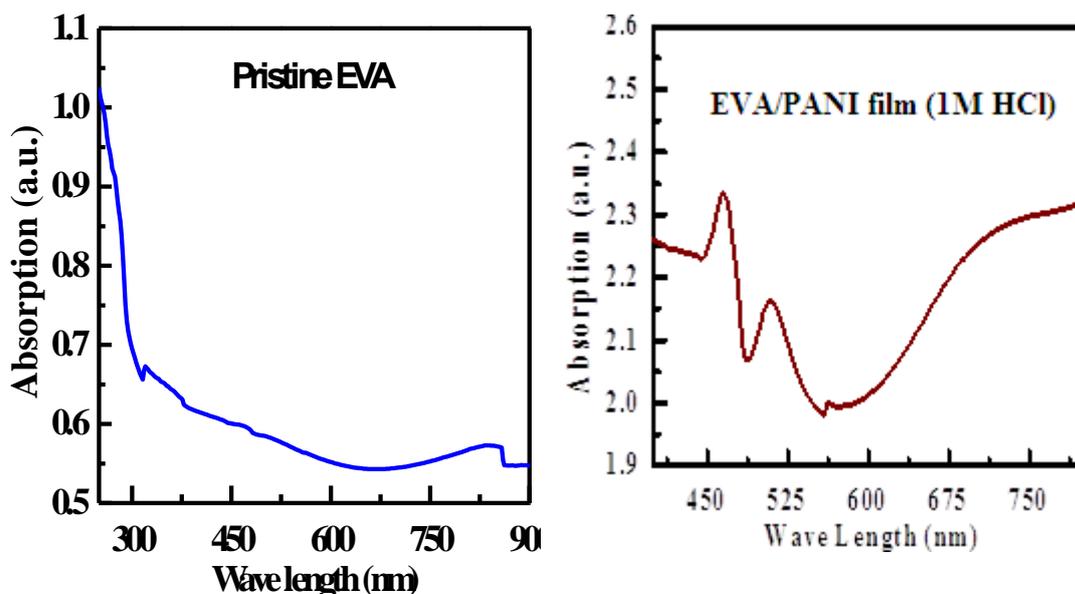


Figure 4. UV Visible spectrum of Pristine EVA (Indicated) and EVA-PANI 1M HCl (Indicated)

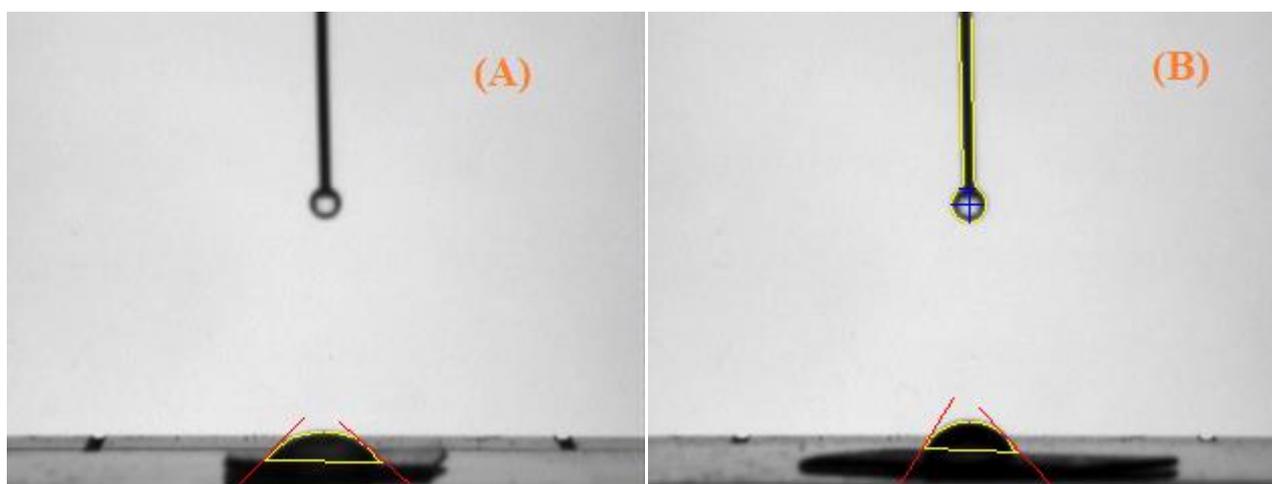


Figure 5. contact angle image of Pristine EVA (A) and EVA-PANI 1M HCl (B)

Table 1 Contact angle measurement results

S. No.	Sample Name	Contact angle (Degree)	Height from top of base (mm)
1	Pristine EVA	94.217	0.309
2	EVA-PANI 1M HCl	60.50892	0.2781

Contact angle experiments (Figure 5) show either hydrophilic or hydrophobic behaviour of tested materials. Analysis of the record is shown in the Table 1. It is found that EVA film indicated hydrophobic in nature (contact angle =94.127), whereas, EVA/PANI-ES composite is hydrophilic nature (contact angle =60.50). This may happened due to the occurrence of polyaniline salt en the EVA film

Figure 6 show SEM images of pristine EVA, HCl-doped polyaniline, and EVA/PANI composites During the SEM test, gold coated EVA and EVA/Pani composite film is carried out. But, Gold coated PANI-ES pallet sample is used to perform the SEM test. To

check surface morphology, the SEM study after application of pressure on EVA/Pani composite has also been performed. It is seen from that indicate neat EVA film and is having smooth surface which is ductile in nature. Figure 6B shows different-sized granular like morphology (neat PANI-ES). Grain-shaped morphology of conductive polyaniline is more suitable for potential application, particularly dielectric pressure sensor.

Dielectric behaviour of EVA and EVA/PANI Composite are analyzed at different frequencies and is shown in Figure 7. Dielectric property depends on shape of inclusion in host medium i.e presence of

materials between probes. With the contact of probes, there is a chance to develop inter particle electrical interaction through polarization. This is happened due to dielectric response. However, with the application of

electricity on prepared materials may undergo breakdown leading to the reduction in their aspect size/ratio.

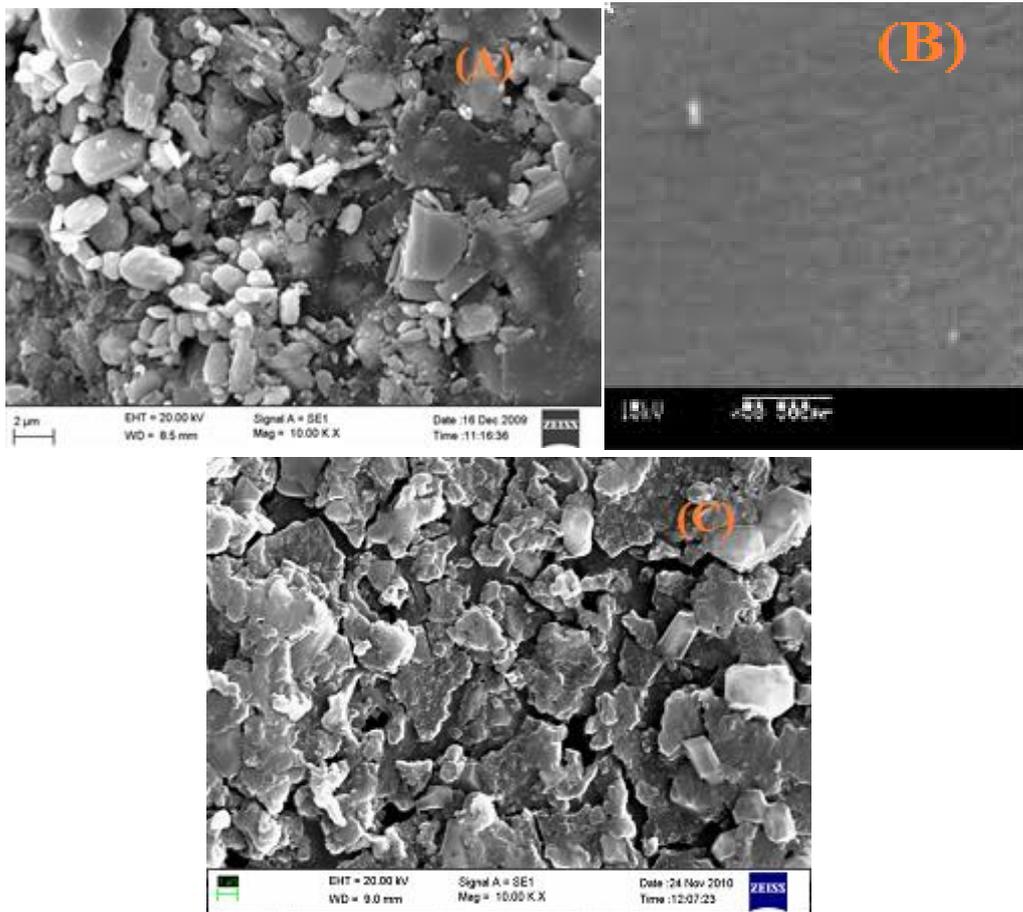


Figure 6. SEM image of PANI (A), EVA (B), EVA-PANI (C)

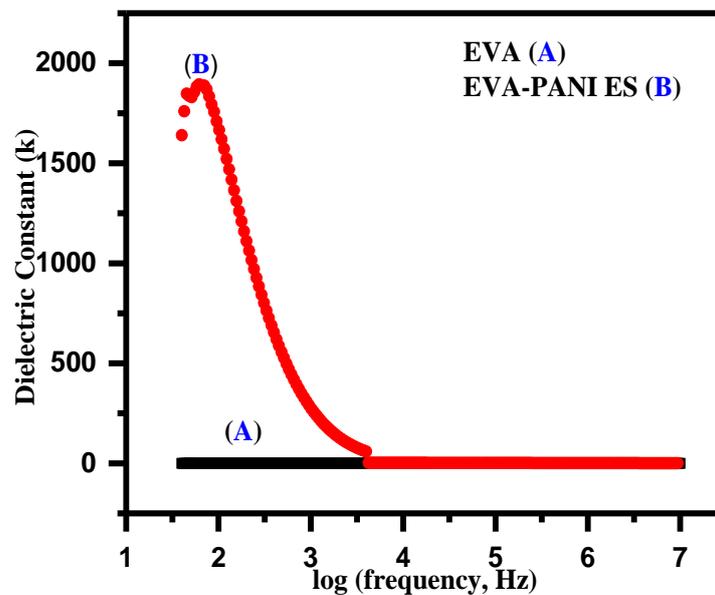


Figure 7. Dielectric constant vs log (f, Hz)

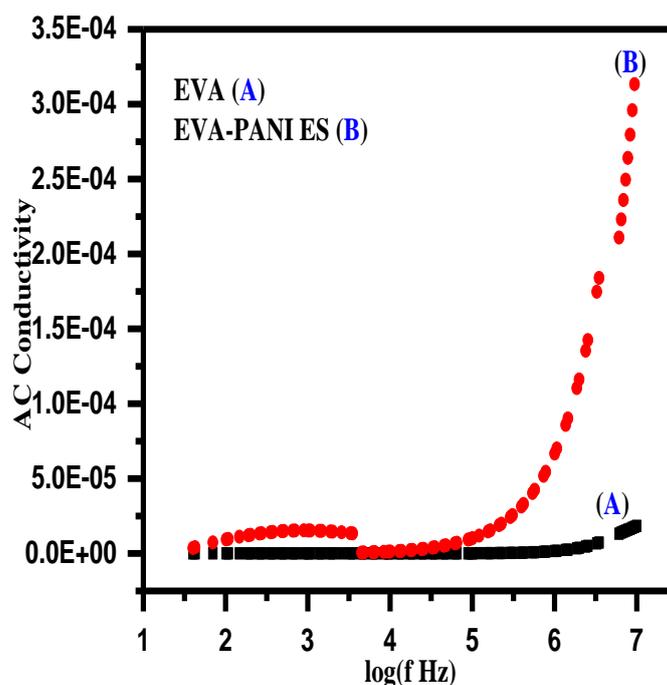


Figure 8. AC conductivity vs log (frequency Hz) of

This result in more irregular change and also decrease in dielectric response. Result is observed from Figure. 7 that the PANI particles are aggregated in the EVA matrix mainly because in this composite. PANI is continuous phase. There is also some change in surface morphology for pressed composite as shown in Figure. 7.

A.C. conductivity of EVA and EVA/PANI Composite are studied at different frequencies and is shown in Figure 8. This study is offers a useful information on electrical polarization process. AC conductivity of EVA film sample (Figure 8) shows constant value up to 10^6 Hz and then increases steeply with frequency. This nature is obeying universal power law and is associated with polarization function [37].

Low frequency behaviour is due to the hopping mechanism. A.C. conductivity is increased at higher frequencies and is attributed to polarons contribution. In this contribution, polarons are moving along shorter distance in PANI polymeric chains [38].

4. Conclusions

X-ray diffraction, Fourier transformation infra-red spectroscopy, UV-Visible, scanning electron microscope and dielectrical behaviors of EVA and EVA/PANI composite are tested for investigating different characteristics of prepared materials. In X-ray diffraction analysis, EVA/PANI composite has increased crystallinity compared to base polymer i.e., EVA polymer. In Fourier transformation infra-red spectroscopy analysis, presence of desired chemical groups and shifting peak position are observed EVA/PANI composite. Shifting is may be due to interaction between EVA polymer and PANI. In UV-

Visible, π - π^* -and polaron- transitions are observed. In scanning electron microscope analysis, distinction morphology is identified. In wettability investigation, hydrophilic nature is observed in case of EVA/PANI composite. It is due to occurrence of PANI on EVA polymer film. Dielectric performance of EVA/PANI composite is increased compared to EVA polymer.

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Conflict of interest

The Author has no conflicts of interest to declare that they are relevant to the content of this article.

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