Investigation of Frequency and Temperature Dependent Electrical and Structural Characterization of PVC-PMMA Thin Films with Salicylic Acid Doping

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ABSTRACT

This research explores the electrical, molecular, and structural characteristics of PVC: PMMA Polyblend (1:2) doped with 8% salicylic acid. AC conductivities and dielectric constants were measured over a temperature range of 303 K to 353 K using a Precision LCR meter, revealing nuanced with dopant concentration, temperature, and frequency. FTIR spectroscopy highlighted distinct peak differences in PVC-PMMA films with and without the dopant, emphasizing the impact of salicylic acid on molecular interactions. XRD analysis demonstrated an amorphous nature in both doped and undoped thin films, while SEM and EDX revealed heterogeneous structures with varying dopant percentages. The study provides valuable insights into the intricate interplay between temperature, frequency, and dopant concentration in the electrical and structural properties of PVC: PMMA blends. These findings contribute to the understanding of material behavior for potential applications in diverse fields such as materials science and pharmaceuticals.

Keywords: *PVC-PMMA; AC conductivity; dielectric constant; frequency; salicylic acid; FTIR; XRD; SEM; EDX*

INTRODUCTION

Polymer composites have garnered significant attention for their diverse applications in the field of electronics, owing to their tunable electrical properties and flexibility. Among these materials, Polyvinyl Chloride (PVC) and Poly(methyl methacrylate) (PMMA) blends have gained prominence as potential candidates for various electronic and optoelectronic devices due to their intriguing characteristics, such as high dielectric strength, thermal stability, and ease of processing. To further enhance their electrical performance, the introduction of dopants has emerged as a promising strategy.

EXPERIMENTAL

Thin films of PVC-PMMA with different dopant concentrations were prepared using the isothermal evaporation technique. Preparation of a Polyblend thin film of PVC-PMMA in 1:2 weight proportional, the dopant and the polymer mixture were dissolved in a solvent (THF) were mixed in solution form .for a complete Homogeneous solution was kept for two or three days. after two or three days solution are in a homogeneous form then the solution mixture was poured onto a perfectly planed glass plate floating freely in a pool of mercury for perfect leveling. it was thereafter allowed to evaporate at room temperature further, and it was dried for two days to remove any traces of solvent. the

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dry film removes from the glass plate and cuts into pieces of desired size then measure the thickness of the thin film by DIGMATIC micrometer, which was then coated on two sides with silver paint then by using the multimeter check whether the two electrodes working or not .then investigate the conductivity .Two sets of films were fabricated: one without salicylic acid (0% dopant) and the other with 8% salicylic acid as the dopant. AC conductivity measurements were performed using 4284 A precision LCR meter (Agilent Make), covering a frequency range of 20 Hz to 1MHz. The measurements were carried out at two different temperatures: 303 K to 353 K. The ln f and ln AC conductivity and dielectric values were recorded for further analysis.

Graph related to Dielectric constant of PVC-PMMA 1:2 with 0% SA and 8% SA

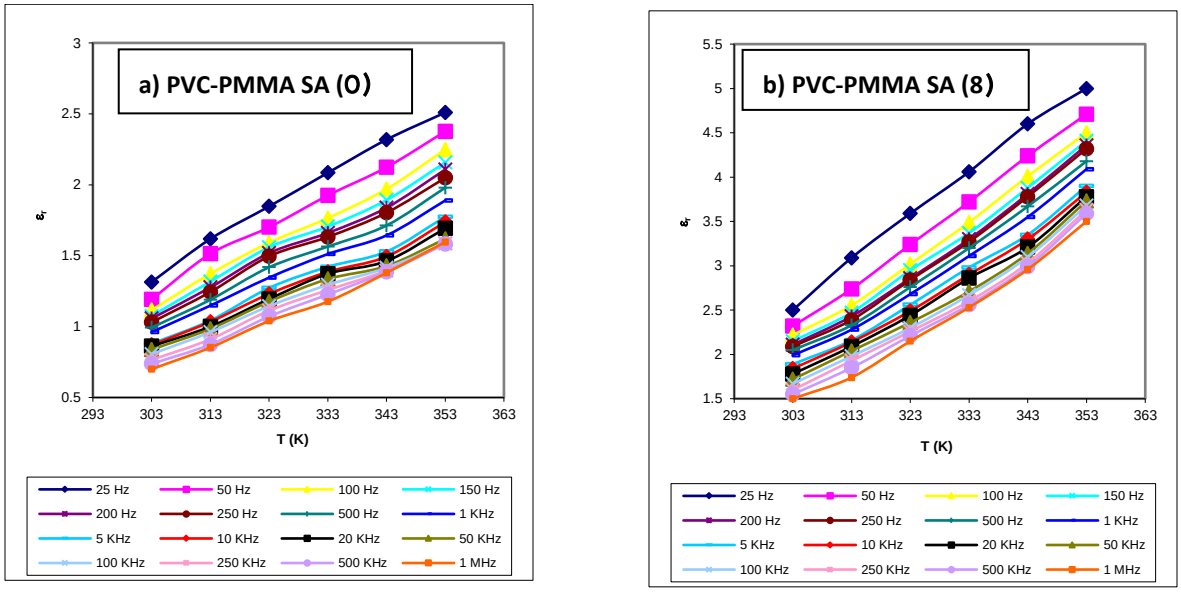


Fig 1.1. (a-b)Variation of ε_r with T(K) at different frequencies for 1:2 PVC-PMMA System

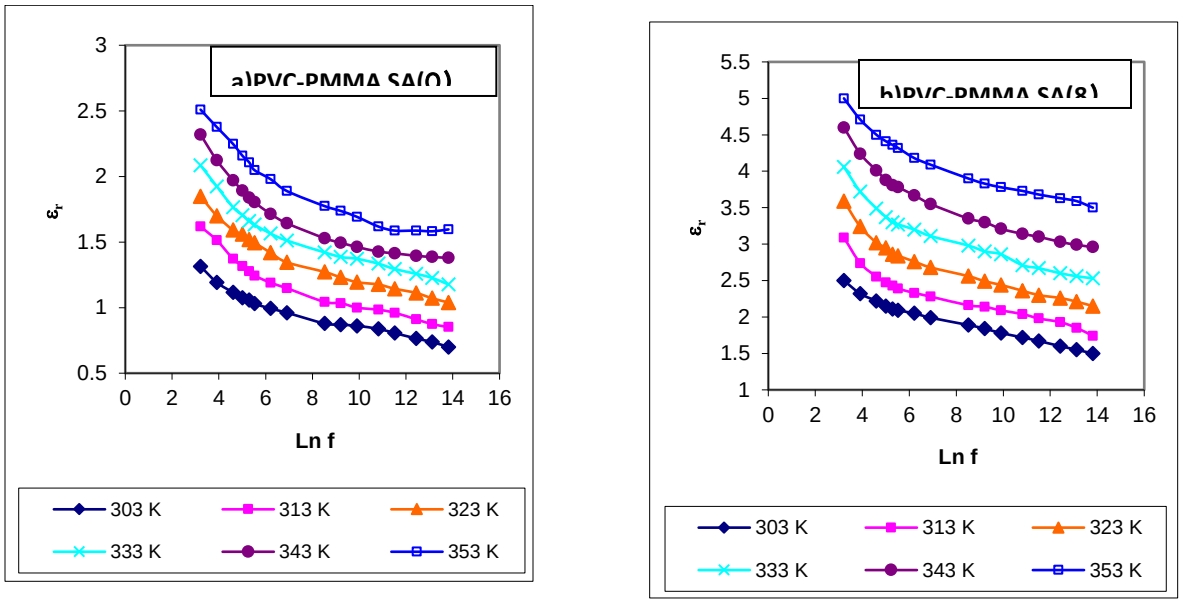


Fig 1.2.(a-b)Variation of ε_r with Ln f at different temperatures for 1:2 PVC-PMMA System

Graph related to AC Conductivity of PVC-PMMA 1:2 with 0% SA and 8% SA

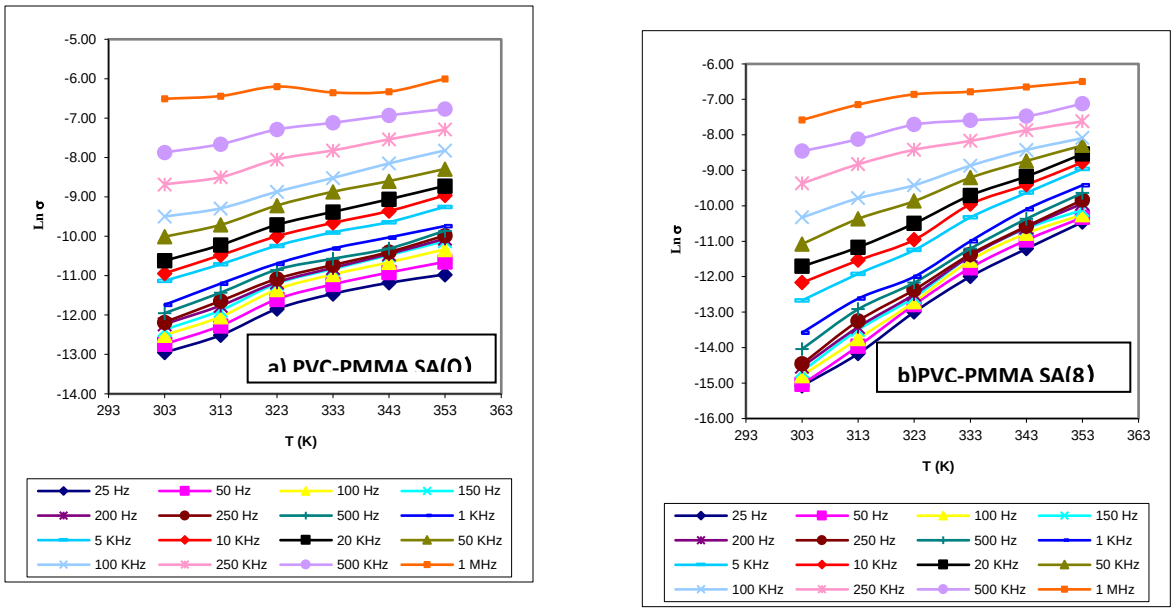


Fig 1.3. (a-b)Variation of Ln σ with T (K) at different frequencies for 1:2 PVC-PMMA System

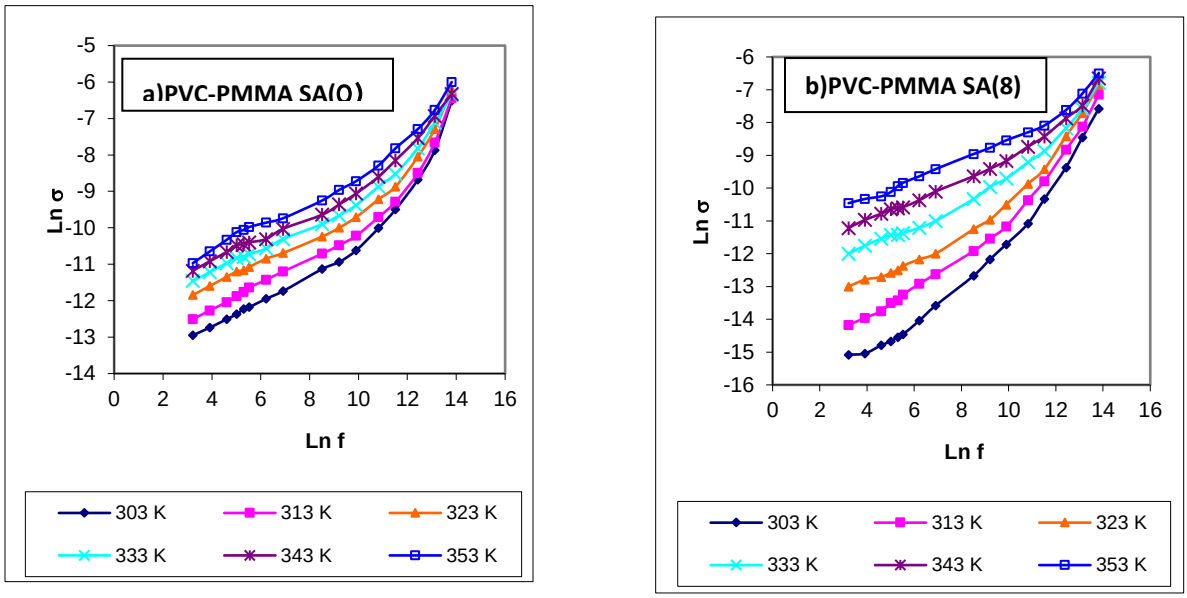


Fig 1.4.Variation of Ln σ with Ln f at different temperatures for 1:2 PVC-PMMA System

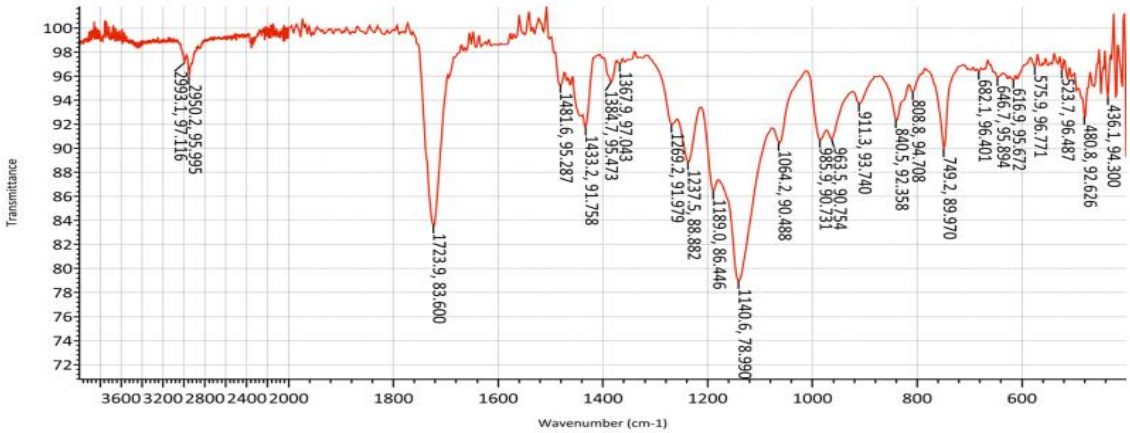


Fig 1.5 FTIR of 1:2 (PVC-PMMA) SA (0)

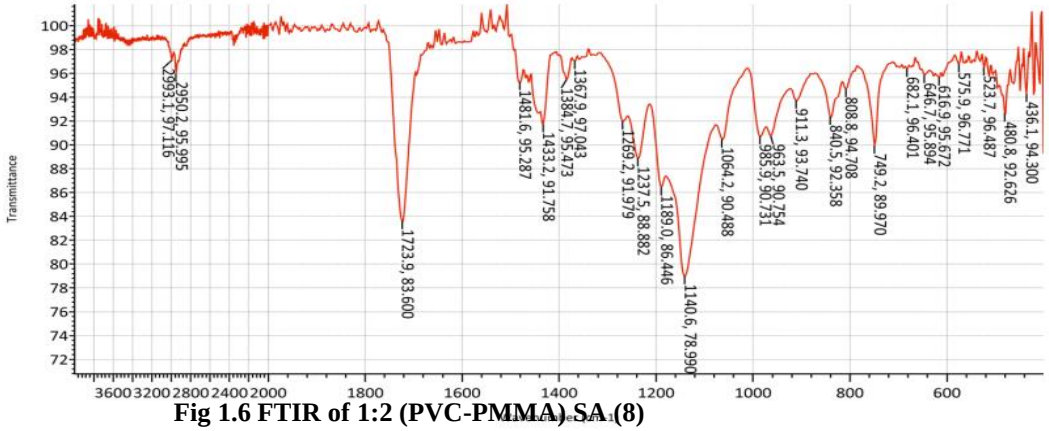


Fig 1.6 FTIR of 1:2 (PVC-PMMA) SA (8)

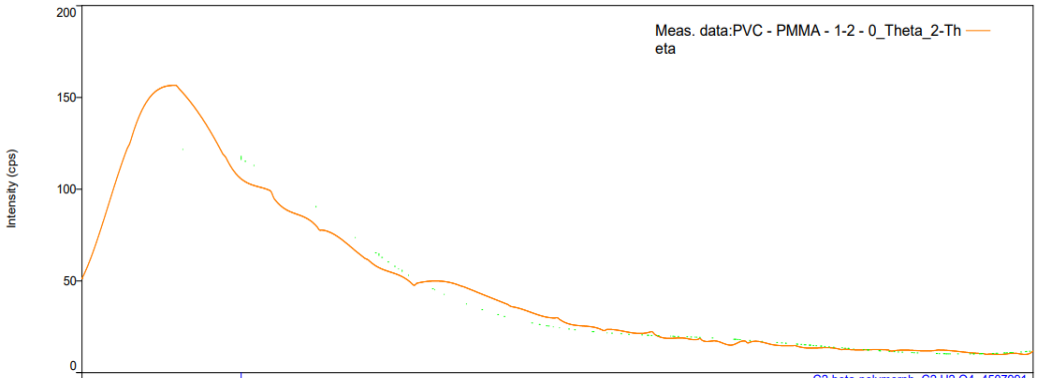


Fig 1.7 XRD Spectra of 1:2 PVC-PMMA 0% SA

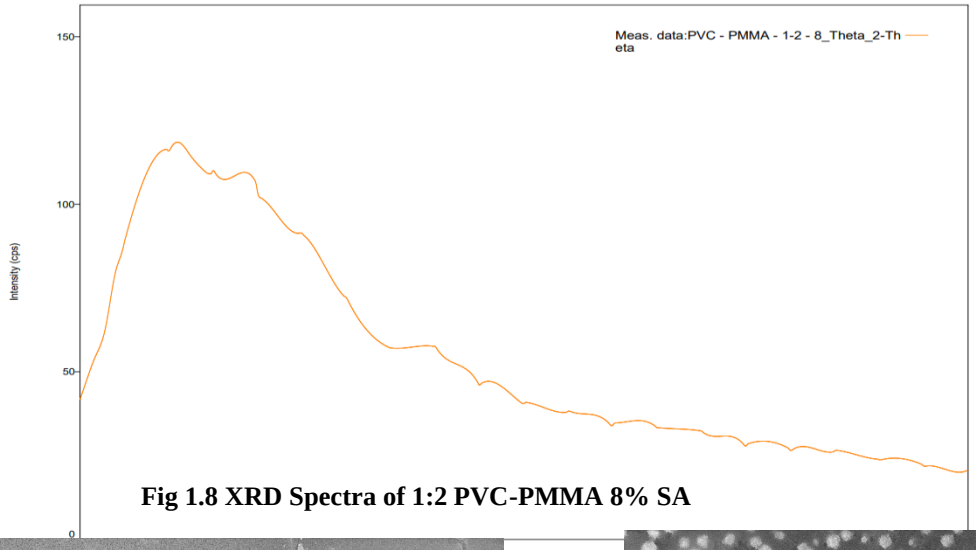
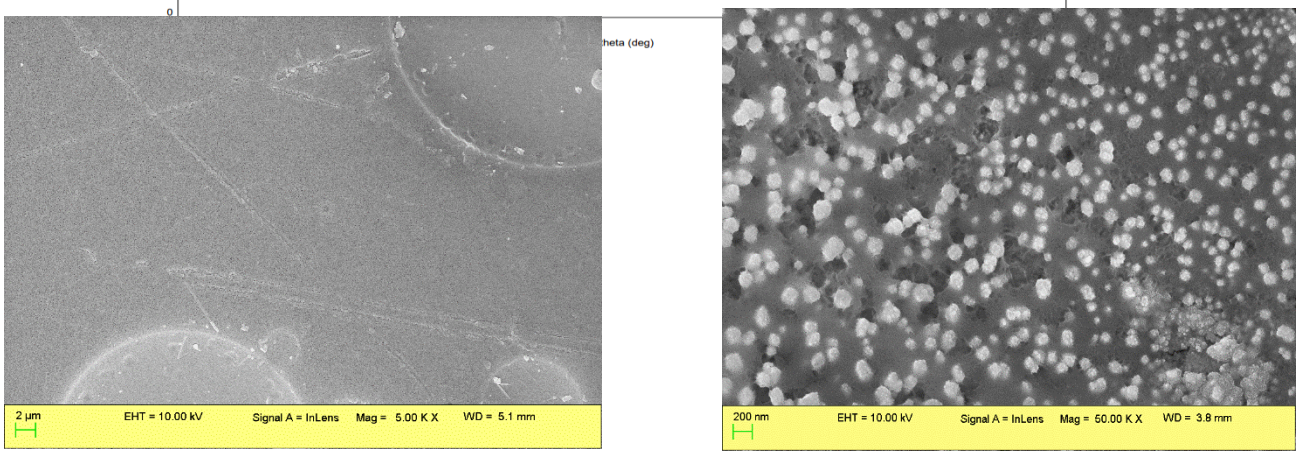


Fig 1.8 XRD Spectra of 1:2 PVC-PMMA 8% SA



SA **Fig 1.10 SEM of 1:2 PVC-PMMA 8% SA**

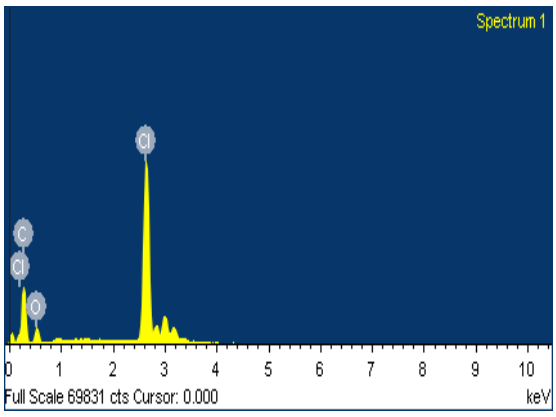


Fig 1.11 SEM of 1:2 PVC-PMMA 0% SA

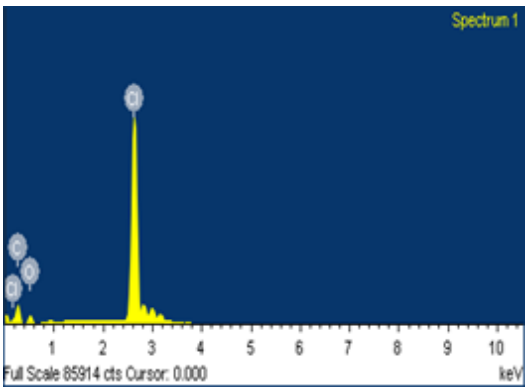


Fig 1.12 SEM of 1:2 PVC-PMMA 8% SA

RESULTS AND DISCUSSION

AC conductivities and dielectric constant:

The AC conductivities and dielectric constant, XRD, SEM, EDAX of PVC:PMMA blends (1:2)doped with 8% percentages of Salicylic acid were examined in this study. Measurements were taken using a 4284 A precision LCR meter (Agilent Make) with a frequency range of 20 Hz to 1 MHz. The tests were conducted over a temperature range of 303 K to 353 K.

Here's a summary of the key findings:

1. Variation of dielectric constant with temperature at different frequencies with and without dopant

2. Variation of dielectric constant with frequency at different temperatures with and without dopant

3. Variation of AC conductivity with frequency at different constant temperatures with and without dopant

4. Variation of AC conductivity with temperature at different constant frequencies with and without dopant

5. Variation of dielectric constant with concentration of dopant at various temperatures.

6. AC conductivity with concentration of dopant at various temperatures

The study explored PVC: PMMA blends (1:2) doped with 8% salicylic acid, examining various properties using a Precision LCR meter. Dielectric constant increased with temperature due to enhanced dipole flexibility, particularly notable in the polar PVC: PMMA blend. Conversely, dielectric constant decreased with increasing frequency, attributed to orientation polarization struggling to match field variations at higher frequencies. AC conductivity rose with frequency, indicating higher loss currents in response to changing electric fields. Notably, the dielectric constant increased with dopant percentage but decreased in AC conductivity, suggesting the salicylic acid's impact on the blend's functional sites. This sheds light on the nuanced interplay between temperature, frequency, and dopant concentration in the electrical and structural properties of the PVC: PMMA blends.

FTIR:

The FTIR spectroscopy is a very useful technique to elucidate the intermolecular interaction between PVC, PMMA and Salicylic Acid. FTIR spectra were collected using an Agilent Micro Lab instrument. The spectra were recorded in the range of 4000-400 cm^{-1} each sample was scanned 64 times, and background scans were taken 32 times to ensure accuracy and reliability. The FTIR spectra of PVC/PMMA/SA films were depicted in Fig. 1.5 demonstrates the FTIR spectrum of PVC-PMMA thin film without doping and Fig 1.6. Demonstrates the FTIR spectrum of PVC-PMMA thin film with doping.PVC-PMMA with SA. The FTIR spectra of PVC-PMMA thin films with and without an 8% SA dopant reveal notable differences in peak wavenumbers and intensities. The prominent peaks and their implications are discussed below In Fig 1.5.FTIR of PVC-PMMA 0% SA the peak 1062-1064.2 cm^{-1} rocking vibration of CH_2 group of PVC.The Peak 2950.2 In both film with and without SA which shows C-CH_3 ,C-H Stretching in PMMA (Rajendra and Uma (2000)) .The Peak 1481.6 C-H Stretching in PMMA. The Peak 1481.6 C-H Stretching in PMMA. The peaks at 2993.1 cm^{-1} and 2951-2950.2 cm^{-1} corresponds to the stretching of–CH_3 and –CH_2– groups of PMMA.The band 616.9-617 cm^{-1} demonstrate the C–Cl bond of the isotactic and syndiotactic structure of PVC.Broader and stronger bands in the region 1300-1000 cm^{-1} correspond to C–O stretching vibrations which usually consists of two asymmetric coupled vibrations. i.e. C—C (=O)—O and O—C—C.The Peak at 749.2 cm^{-1} corresponds to out of plane C—H bending in PVC. A sharp band located at 1723.9 cm^{-1} was ascribed to the carbonyl group (C=O) in the film, which is typically attributed to PMMA.. Similarity Peak at 840.5 cm^{-1} PVC-PMMA without SA: Intensity of 94.708 and PVC-PMMA with 8% SA: Intensity of 92.801This peak is associated with CH_2 rocking vibrations in PVC. In Both Film the band at 1433-1433.2 cm^{-1} is due to the wagging of methylene groups in PVC and/or owing to the asymmetric stretching of O–CH_3 group of PMMA. FTIR analysis highlights the presence of both PVC and PMMA components in the film. The intensity of various peaks provides information about the relative composition and structural aspects of the film. The reduction in intensity for specific PVC-related peaks suggests a decrease in the PVC content and potential structural changes. Fig 1.6 .FTIR of PVC-PMMA 8% SA which shows the stretching vibrations of C-H bonds 2849.5 cm^{-1} [21]. The bands at 1328.8 cm^{-1} represents the deformation of –CH_2 in PVC and attributed

the overlapping CH_2 wagging in PMMA, 1249 cm^{-1}, The peaks at 609 cm^{-1} demonstrate the C–Cl bond of an isotactic and syndiotactic structure of PVC.609.4 cm^{-1} signifies C-Cl bonding. The characteristic peak at 1722-1723.9 cm^{-1} can be attributed to the C=O stretching vibration of acrylate carboxyl group of PMMA and Both Film the asymmetric stretching of CH_3 group was observed at 1433.2-1435 cm^{-1}. The bands at1140.6- 1141 cm^{-1} and1237.5- 1238 cm^{-1} are because of the C–O–C absorption and the stretching vibration of $–OCH_3$ group of PMMA. The characteristic absorption bands of PMMA occur at 1189.6-1190 cm^{-1}, 1064-1064.2 cm^{-1}, 985-985.9 cm^{-1} and 840-840.5 cm^{-1} [26, 28]. In PVC-PMMA doped SA thin film The 1727.6 cm^{-1} characteristic peak is attributed to the C=O stretching vibration of the acrylate .The band located at 1148-1146.2 cm^{-1} was attributed to the C–O group carboxyl group of PMMA and.In Both Film The bands at 961-963.5 cm^{-1} are of CH_2 rocking vibration. The bands at 688-689.6 cm−1 and Peak at 436.1 cm^{-1} PVC-PMMA without SA: Intensity of 94.300 and PVC-PMMA with 8% SA: Intensity of 93.656 .This peak is attributed to C-Cl stretching in PVC. The decrease in intensity in the doped film suggests a reduction in the PVC component. The decrease in intensity indicates changes in the polymer structure due to the presence of SA.Peak at 1384.7 cm^{-1} PVC-PMMA without SA: Intensity of 95.473 .PVC-PMMA with 8% SA: Intensity of 95.505.This peak represents the CH_3symmetric deformation in PVC. The marginal difference in intensity suggests minor changes in the polymer structure. Peak at 1587.8 cm^{-1}.PVC-PMMA without SA: Absent .PVC-PMMA with 8% SA: Intensity of 98.436.This strong peak corresponds to the CO stretching in SA, confirming the successful incorporation of SA into the doped film. Peak at 2950.2 cm^{-1} .PVC-PMMA without SA: Intensity of 95.995.PVC-PMMA with 8% SA: Intensity of 95.158.This peak is linked to CH_3stretching in PMMA. The similar intensities suggest that SA has a limited effect on PMMA. Peak 1587.8 cm^{-1} corresponds to CO stretching in SA, confirming the successful incorporation of SA into the film. Peak 23 at 1727.6 cm^{-1} indicates the presence of C=O stretching vibration of the acrylate carboxyl group of PMMA and is attributed to the SA dopant. In the film doped with 8% SA, several new peaks emerge, suggesting successful incorporation of SA: The presence of SA is evident with a strong peak at 1587.8 cm^{-1}, representing CO stretching in SA. Other peaks related to PVC and PMMA, including C-Cl stretching and CH_2 rocking vibrations, are also present. The FTIR analysis indicates that SA has a limited effect on the characteristic peaks of PMMA, as the peak related to CH_3 stretching remains similar in intensity.

The introduction of Salicylic Acid into the PVC-PMMA thin film is confirmed by the appearance of SA-specific peaks. The decrease in the intensity of PVC-related peaks in the doped film suggests a reduction in the PVC component, indicating a change in the polymer structure due to SA doping. SA has a limited impact on PMMA, as indicated by the consistent intensity of PMMA-related peaks. This FTIR analysis underscores the importance of studying the molecular interactions and structural changes when incorporating SA into PVC-PMMA films, which has implications for various applications, including materials science and pharmaceuticals. The FTIR analysis confirms the presence of SA in the film, as evidenced by the SA-specific peaks. The peaks related to PVC and PMMA are still present in the film, indicating the coexistence of these components with SA. The similar intensity of PMMA-related peaks suggests that SA has a limited effect on PMMA. This analysis provides essential information about the molecular interactions and structural changes occurring when SA is introduced into the PVC-PMMA thin film, highlighting the impact of the dopant on the film's chemical composition and structure.

Result from XRD Spectra and SEM and EDX analysis:

XRD of undoped and doped thin film show almost amorphous Nature. There are no sharp peaks in all the X-RD patterns. It is well known that the absence of peaks in intensity versus 2θ curve indicates that the (thin films) samples are amorphous in nature. Energy dispersive X-ray (EDX) was engaged for elemental analysis of the PVC-PMMA Polyblend without and with SA thin film. The EDX spectrum presented in Fig.1.11-1.12. Confirmed sharp peaks due to the following elements: fig 1.11 shows C (67.84%), O (13.21%) and Cl(18.94 %) and fig 1.12 shows C (56.81%), O (11.07%) and Cl (32.12 %) in addition to hydrogen. The occurrence of these elements will generate charges on the surface of the polymer and create electrostatic forces of attraction between the samples

Explanation from SEM Photography Scanning electron microscope images of the 1:2 PVC-PMMA blend without SA and with SA. The images exhibit heterogeneous structure in both blends. Fig 1.9 shows the morphology of blends show a phase separated region. The PVC domains are visualized as the hole s from which the material was pulled out. Systems the phase separations were observed. This is in agreement with result reported by (Yongseok Kim et al., 2008), (Rajendran S. et al, 2008).fig 1.10. Shows as dopant percentage increases. That is due to the increase in amorphousity. This is in agreement with XRD results.

CONCLUSIONS

In conclusion, the study investigated (1:2) PVC:PMMA blends doped with 8% salicylic acid. AC conductivity and dielectric constant variations were studied at different temperatures and frequencies, revealing nuanced interactions. FTIR analysis confirmed the successful incorporation of salicylic acid, impacting PVC but having a limited effect on PMMA. XRD patterns indicated an amorphous nature in both doped and undoped thin films. SEM images showed a heterogeneous structure, with increasing dopant percentages leading to higher amorphousity. Overall, the findings highlight the complex interplay of temperature, frequency, and dopant concentration on the electrical and structural properties of PVC:PMMA blends.

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