Preparation and characterization of low dielectric constant soft polymer blends for engineering applications

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The preparation of polyvinyl chloride (PVC)/cellulose acetate (CA) blends has been performed by solution casting. The structural, morphology, thermal, dielectric properties and softness have been tested as a function of the weight fraction of the CA. The decrease in inter-planar distance has been confirmed by X-ray diffraction (XRD) spectroscopy. The decrease in direct band gap has been influenced by CA composition demonstrated by ultraviolet-visible (UV) spectroscopy technique. Scanning electron microscopy (SEM) demonstrates the micro porous nature of blends. Thermo gravimetric analysis (TGA) confirms the increase in thermal stability of the miscible blends. The real dielectric polarization (ε_r) has been measured as a function of frequency. The low dielectric loss tangent (tan δ) in PVC/CA blends has been observed based on the principle of restricted polarizibility. The shore 'A' durometer test enables to confirm the softness of miscible blends. Optimized low dielectric constant and softness properties would be suggested for various electrical and electronic engineering applications.

Keywords: Polyvinyl chloride (PVC), Cellulose acetate (CA), Dielectric properties, Softness

1 Introduction

Polyvinyl chloride (PVC) belongs to the class of commodity thermoplastic. Upon ethylene and styrene it is the third largest material in demand around the globe. The flexibility of PVC achieved with the combination of plasticizer. Under external stimuli of heat and pressure, PVC exhibits the chemical change¹. The presence of chlorine in the PVC moiety is highly suitable to withstand the fire resistance and better durability². It has low thermal conductivity³, highflame retardancy⁴, high chemical resistance⁵ and good mechanical stiffness⁶. It is flexible and long lasting polymer with suitable and biological resistance⁷. It is the most important material in medical devices and applications including blood storage bags. PVC is chosen because of low cost, chemical resistance and high compatibility with additives⁸. Some of the additives used in PVC formulations are plasticizers, stabilizers, lubricants and fillers⁹. PVC is used for the consumer products like cables, pipes, window frames, packaging, bottles, credit cards and audio recording. PVC is widely used in production of electric insulation materials.

Cellulose is a natural polymer used in numerous applications such as paper products, consumables, energy crops and bio fuels¹⁰. Cellulose acetate (CA) is

the acetate ester of cellulose and one of the most common biopolymers on earth. CA has many advantages such as biocompatibility, biodegradability and regenerative properties¹¹. Because of many advantages and recasting, CA is widely used in packaging, textiles and biomedical fields¹². CA is brittle in nature and has outstanding properties in reducing membrane fouling. It has poor mechanical strength, lacks flexibility and toughness¹³. It is hydrophilic in nature having high resistance and can be prepared into membrane. CA is widely used in diverse applications such as microfiltration¹⁴, ultrafiltration, nanofiltration¹⁵, reverse osmosis¹⁶ (RO) and gas separation¹⁷.

PVC/CA membrane micro porous blends demonstrated the increase in protein flux as function of PVC loading¹⁸. Scanning electron microscopy (SEM) shows the average diameter of pores in the range of 1 um spherical in nature. Reduction in pore size is observed in PVC-cellulose triacetate (CTA) film due to matrix formation of blends¹⁹. PVC/CA membrane is used as electrodialysis based on principle of cationic exchange of micro porous membrane²⁰. It presented the fabrication of PVC/CA heterogeneous cation exchange membrane confirmed that ionic permeability and flux were increased with CA ratio to 20 wt% in casting solution and then decreased slowly by adding CA content from 20 to 50 wt% and increased again from

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50 to 100 wt% in the prepared membranes. PVC/CA blends modified with N-phenyl amino malaeimides exhibit decrease in softening point and increase in conductivity and elasticity property as a function of modifier²¹. miscibility The of poly(methyl methacrylate)/cellulose acetate pthlate (PMMA/CAP) polymer blends was confirmed by a single glass peak²². temperature transition (T_{σ}) Ethylenepropylene-diene terpolymer grafted vinyloxyaminosilane/linear low density polyethylene (EPDM-g-VOS/LLDPE) blends with different compositions were prepared by two roll mixing mill. It increases the insulating behaviour with reduction of mechanical properties due to percentage composition²³. Polyvinylidene chloride-co-acrylonitrile/poly(methyl methacrylate) (PVDC-AN/PMMA) blends increase the ionic conductivity and polymer flexibility and show the conduction²⁴. ionic Polyethylene better oxide/poly(methyl methacrylate) (PEO/PMMA) blend supports the ionic conduction and shows the high crystalline portion and low ionic conductivity²⁵. Poly carbonate/poly(methyl methacrylate) PC/PMMA blend is highly miscible at elevated temperatures and has better mechanical strength²⁶. The reports on polymer blends modified with various fillers and improved properties feasible for the various applications are given in Table 1.

With this motivation we have attempted to prepare the PVC/CA blends and testing of electrical properties as a function of temperature which have been disclosed with structure and morphology that may be suitable for microelectronics domain.

2 Experimental Details

2.1 Materials

The polymer PVC with the molecular weight 233,000 g/mol, molar density 1.4 g/mol at 25 °C, tensile strength 22.8, specific gravity 1.38 (\pm 0.3), volume resistivity 8×10¹⁴ Ωm is supplied by Sigma Aldrich. Cellulose acetate (CA) CAS No: 9004-38-0 supplied by C Jivanlal and Company, Navi Mumbai, India in powder form. N-N-dimethylformamide (DMF) Batch No: 3787687 having the (molecular weight) 73.10 of AR grade is purchased from Sisco Research Laboratories, PVT, Ltd, Mumbai, India used as a solvent.

2.2 Synthesis of PVC/CA blends

A polymer PVC/CA blend was prepared by solution casting method. Initially, PVC was dissolved in DMF (stirred at 950 rpm at 32 °C) to get complete dissolution. Similarly CA dissolved in DMF (stirred at 690 rpm at 32 °C). Then the required solution of PVC/CA (100/0, 0/100, 90/10, 80/20, 70/30 and 60/40 (wt%)) was mixed together and stirred at 950 rpm at 50 °C. The prepared solution was poured in petri dish and kept in oven (for 8 h at 40 °C). The polymer film was peeled off from the petri dish and used for further characterization. The detailed protocol to obtain the blend is shown in Fig. 1 and the proposed scheme for PVC/CA moieties is shown in Fig. 2.

2.3 Characterization techniques

The X-ray diffraction (XRD) of the PVC/CA blends was performed by using CuK_{α} radiation having the wavelength of λ = 1.54 Å produced by Bruker AXS D8

Table 1 — Survey of polymer blends			
Polymer systems	Modifier	Properties	Applications
PVC/CdO	-	Physical properties dependent on internal structure	Optical and electronic devices ³
PVC/SBR	Iron oxide/ silver	Good electrochemical properties	Electro dialysis process ⁷
PVC/CA	PEG	Increase in porosity and macro void formation.	Separation of proteins ¹⁸
PVC/CTA	_	High thermal stability	Ultra filtration and dialysis membranes ¹⁹
PVC/CA	Resin powder	Ionic permeability and flux	Water recovery and waste water treatment ²⁰
PVC/CA	<i>n</i> -phenyl amino maleimide	Glass rubber transition temperature	Biodegradability of PVC waste products ²¹
PMMA/CAP	$\varepsilon_r = 70k$	Intermolecular hydrogen bonding	Automotive and home appliances ²²
EPDM-g-VOS/LLDPE	$\varepsilon_r = 3$	Better mechanical and little loss of electrical properties	High voltage cable insulation ²³ up to 35 kV
PVDC-AN/PMMA	$\varepsilon_r = 2.2$	Polymer flexibility and better ionic conduction	Lithium batteries ²⁴
PEO/PMMA	$\varepsilon_r = 1 \times 10^6$	Improves ionic conductivity	Lithium metal electrodes ²⁵
PC/PMMA	$\varepsilon_r = 1.49$	Highly miscible	Power industry ²⁶



Fig. 2 — Proposed scheme for PVC/CA blends

focus advance XRD meter (Rigaku, Japan, Tokyo). The power of XRD is 2.2 kW Cu anode, ceramic X-ray tube. The 2θ ranges from 10° to 80° with a scanning speed of 1° /mm and step size of 0.01°, respectively. XRD measurements were performed by step mode= 10° .

The UV-Vis spectrum of PVC/CA blends is performed by Shimazdu UV-2401 PC, UV-Vis

spectrophotometer in the range of 200-300 nm in the absorbance mode.

Fourier transform infrared spectroscopy (FTIR) of PVC/CA blends was performed by Paragon 500 FTIR spectrophotometer across the wave number range of 4000 cm^{-1} -500 cm⁻¹, respectively, in transmittance mode.

Scanning electron microscope (SEM) of PVC/CA blends was done by Zeiss scanning electron microscope to know the surface morphology for various CA loadings. The samples were stained with gold metal. The micrographs were done by 2 and 3 μ m resolution.

Thermo gravimetric analysis (TGA) was done by a ramp method and performed by SDT Q600 V kept at a room temperature at 20 °C/min under nitrogen atmosphere N₂ Purge = 100 mL/min with the size of 4.216, 4.080, 4.920 and 4.680 mg.

The electrical characterization of PVC/CA blends was performed by using N4L, PSM 1735 impedance analyzer. The test fixture was silver pasted on both sides (sample size of diameter of 10 mm and average thickness of 50 μ m). The temperature was controlled by ALAB BTC 9100 temperature controller. We operated the sample across the broadband frequency range of 50 Hz-10 MHz with the temperature range of 30 °C-150 °C.

The shore 'A' test was performed by Excel Shore softness tester-hardness durometer. The soft plastic test was done by using Shore 'A' tester.

2.4 Interfacial issue on crystalline and amorphous phase of polymer blends

Engineering property of selected polymers belongs to crystalline structure which demonstrates the influence on properties when blended with amorphous nature of polymers. Interfacial restricted polarization based on mixed phase of polymers has been demonstrated. This investigation deals with disclosing the interfacial performance, properties and utility of engineering polymer blends. Normally polymer blends preferred due to high performance properties desired for domestic and industrial applications.

3 Results and Discussion

3.1 XRD analysis of PVC/CA blends

X-ray diffraction (XRD) is an analytical technique used for characterization of crystalline material and provides information correlated to the unit cell dimensions. Figure 3 shows the XRD spectra of (a,b) virgin PVC, CA, (c) 90/10, (d) 80/20, (e) 70/30 and (f) 60/40 (wt %) of PVC/ CA blends.

The Miller indices planes were confirmed for the virgin PVC based on reports²⁷. The XRD of virgin PVC is shown in Fig. 3(a). It reveals that the Braggs peak is obtained at 2θ = 29.34° which is in good agreement with earlier report²⁸. The presence of CA in PVC is illustrated by Bragg's peak obtained at 2θ = 29.36°, 29.54°, 29.74° and 29.94° as shown in Fig. 3(c-f). Bragg's peak is obtained because of

increase in CA wt% which is supported by the previous report²⁹. The structural properties of blends were evaluated by XRD peaks co-related to the interplanar spacing (*d*) and crystallite size (*D*). By using Debye-Scherrer's formula the average crystallite size D (Å) was evaluated:

$$D = \frac{0.9 \,\text{\AA}}{0.000}$$

0 0 0



Fig. 3 — XRD spectra of (a) virgin PVC, (b) virgin CA, (c) 90/10, (d) 80/20, (e) 70/30 and (f) 60/40 PVC/CA blends



Fig. 4 — UV-visible spectra of (a) pure PVC, (b) pure CA, (c) 90/10, (d) 80/20, (e) 70/30, (f) 60/40 PVC/CA blend and (g) band gap energy vs loading of cellulose acetate (wt%)

where $\lambda = 1.54$ Å is the incident X-ray wavelength, β is the full width half maximum (FWHM) intensity of the diffraction peaks and θ is the Bragg's incident angle.

The evaluated inter-planar distance (d) and crystallite size (D) are given in Table 2. The interplanar spacing for 10 wt% of CA blends is further decreased whereas the crystallite size of polymer blends is increased. The sharp peak denotes the crystalline nature and broad peak exhibits the amorphous nature of the polymer blends³⁰.

3.2 UV -Visible of PVC/CA blends

UV-Vis absorption spectra are used to investigate the band structure of the material. Figure 4(a,b) represents the optical absorbance of virgin PVC and CA at 260-300 nm wavelength regions. The absorption band at 277 nm is assigned to π - π^* transition formed by conjugated double bonds by dehydrochlorination in PVC/CA³¹⁻³³. Decreased absorption peak due to CA weight fraction has been observed. It is influenced over the optical band gap.

The UV-Vis absorbance spectrum of PVC/CA is observed in the wavelength range of 260-277 nm as shown in Fig. 4(c-f). The absorbance intensity

	PVC /CA blen	ds	
Sample details (wt%)	2θ (°)	d (Å)	D (Å)
Virgin PVC	29.34	3.040	5.337
	22.97	3.869	1.853
Virgin CA	17.39	5.096	1.255
	13.21	6.699	4.531
PVC/CA	29.36	3.038	4.013
(90/10)	24.77	3.591	3.434
PVC/CA	29.54	3.020	1.116
(80/20)	24.97	3.563	7.855
PVC/CA	29.74	3.000	4.581
(70/30)	26.74	3.329	2.902
PVC/CA	29.94	2.980	5.255
(60/40)	27.14	3.281	5.016

Table 2 — Structural parameters evaluated from XRD spectra of

increased as the function of CA loading and peak width decreased. This significant increase in the absorbance is associated with the chlorine bond³⁴.

The shift in absorption edge in the blend reflects the variation in the optical band gap. The absorption peak is slightly shifted towards the longer wavelength side and this may indicate the miscibility between PVC and CA. Table 3 shows the calculated value for direct band gap (E_g) decreased from 4.25 to 4.20 eV.

Table 3 — Direct band gap values evaluated from UV-Visible			
	spe	ctrum	
Sample code	Sample deta	ils (wt%)	$E_{\rm g}({\rm eV})$
а	Pure PVC		4.32
b	Pure CA		4.38
с	PVC/CA (9	0/10)	4.25
d	PVC/CA(80)/20)	4.24
e	PVC/CA (7	0/30)	4.21
f	PVC/CA(60)/40)	4.20
Table 4 — FTIR peak assignments for virgin PVC, CA and			
	PVC/CA (60/40) blends	
Wavenumb	ers (cm ⁻¹)	Bonds assignments	
286	50	C-H stretching	
165	54	C-H aliphatic bendi	ng
1438		C-H ₂ wagging	
86	4	C-Cl stretching	
657		C-H wagging	
657		C-Cl stretching	

Figure 4(g) shows the decrease in E_g as increasing the loading of CA. This might reveal that variation in the number of final states within the band gap and also increasing the density of localized states in the band structure^{35, 36}.

3.3 Fourier transform infrared spectroscopy analysis of PVC/CA blends

FTIR technique is an easy way to identify the presence of certain functional groups in polymer composites and blends. The chemical structure of virgin PVC, CA and PVC/CA blends is done by using transmission mode³⁷. The characteristics peaks of all the blends are shown in Fig. 5(a-c). The FTIR spectrum of virgin PVC exhibits the characteristics frequencies of C-H stretching vibrations (2860 cm⁻¹), C-H aliphatic bending (1654 cm⁻¹), CH₂ wagging (1438 cm⁻¹), C-Cl stretching (864 cm⁻¹) and C-H wagging (657 cm⁻¹). The FTIR spectrum of CA shows characteristics frequencies O-H broad absorption band³⁸ (3473 cm⁻¹), C-H stretching absorption (2858 cm⁻¹), C-O-C characteristics band³⁹ (1089 cm⁻¹) as given in Table 4.

For PVC/CA blends (60/40) wt%, FTIR spectra show a peak position of C-H stretching vibration band at 2860 cm⁻¹ which is shifted to 2856 cm⁻¹. The absorption peak C-H aliphatic bending at 1654 is shifted to 1662 cm⁻¹. The wagging mode CH₂ at 1438 cm⁻¹ is shifted to 1435 cm⁻¹. The C-Cl stretching and C-H wagging mode at 864 cm⁻¹ and 657 cm⁻¹ is shifted to 875 and 611 cm⁻¹ as shown in Fig. 5(c). Therefore absorption peak is shifted due to higher loading wt% of PVC/CA (60/40).



Fig. 5 — FTIR spectra of (a) pure PVC, (b) Pure CA and (c) 60/40 PVC/CA blends

3.4 Microscopic analysis of PVC/CA blends

The surface morphology in polymer science is recorded by using scanning electron microscopy technique. To identify the phase separation of polymer systems, miscibility, microporosity, surface smoothness, presence of fillers is the key required information obtained by SEM micrographs. In the present investigation, Fig. 6(a,b) 10 wt% and (c,d) 30 wt% of CA loading clearly demonstrated the micropores. Basically PVC has low surface energy with major pores⁴⁰. The influence of CA in PVC blend shows decrease in microspores⁴¹. The micropores size may have the control upon the influence on several properties and it may act as a better membrane.

3.5 TGA of PVC/CA blends

The thermal stability of PVC/CA blends is tested by thermo gravimetric analysis (TGA). Figure 7(a-d) shows the TGA thermograph of different weight ratio of (a) 90/10, (b) 80/20 (c) 70/30 and (d) 60/40 of PVC/CA blends under nitrogen atmosphere at a heating rate of 20 °C/min. The TGA curves undergo two stage degradation processes. For PVC/CA blends,

Table 5 — TGA analysis PVC/CA blends			
Sample code	Sample details (wt%)	T_{g} (°C)	Total weight loss % at 800 °C
а	PVC/CA (90/10)	197	99.3
b	PVC/CA (80/20)	207	98.5
с	PVC/CA (70:30)	218	98.2
d	PVC/CA (60/40)	220	97.4

first weight loss is observed in the range of 232-343 °C, which may be due to influence of crystalline region. In this study it is observed that PVC/CA blends result in increase in thermal stability as compared to PVC. PVC content in the blend affects the thermal decomposition pattern⁴² of CA. The results revealed that value of T_g for PVC/ CA (90/10) (wt %) is 197 °C as shown in Table



Fig.6 — SEM micrographs of (a,b) 90/10 and (c,d) 70/30 PVC/CA blends



Fig. 7 — TGA of (a) 90/10, (b) 80/20, (c) 60/40 and (d) 70/30 PVC/CA blends



Fig. 8 — Dielectric constant of (a) virgin PVC, (b) virgin CA, (c) 90/10, (d) 80/20, (e) 70/30 and (f) 60/40 PVC/CA blends, as function of cellulose acetate (wt %) blending

5. It is due to presence of CA and the decreased slope is due to crystalline region of polymer PVC observed⁴³.

3.6 Electrical characterization of PVC/CA blends *3.6.1 Dielectric constant of PVC/CA blends*

Polymer composites are used in the domain of microelectronics devices, supercapacitors and modern electronic gazettes due to easy moldability and casting in desired shapes, size with moderate properties compare to virgin polymer system⁴⁴. The testing of

dielectric properties is observed in view of structural modification which is confirmed by XRD analysis. The effect of frequency shows the decrease in dielectric constant for varying weight fraction of CA (wt%). Basically in polymer blends or hyper branched network the dielectric property is crucial due to interfacial process⁴⁵. With this motivation the electrical ion exchange study is reported in dielectric properties studied in the present work. The dielectric properties of PVC/CA blends have been measured across frequency from 50 Hz to 10 MHz and temperature range



Fig. 9 — Dielectric loss tangent of (a) virgin PVC, (b) virgin CA, (c) 90/10, (d) 80/20, (e) 70/30 and (f) 60/40 PVC/CA blends as a function of frequency at various temperatures.

30-150 °C. The diameter of the sample under test is 10 mm and average thickness is 50 μ m. Figure 8(a-f) shows the plots of dielectric constant of (a,b) virgin PVC, CA, (c-f) with loading of CA (10, 20, 30, 40 wt%), respectively. The maximum value of dielectric constant (ϵ_r) for PVC/CA (90/10) wt % is 17.69 at 150 °C, 50 Hz. It is clear from the plots that the trend of ϵ_r decreases with increase in frequency due to capacitive discharge. The decrease in ϵ_r can be explained in terms of that due to increase in frequency, the dipole will no longer be

able to rotate rapidly due to capacitive discharge. So their oscillations lag behind those of the field. As the frequency is further increased, the dipole will be completely unable to follow the field and orientation stopped. Hence ε_r decreased at higher frequency⁴⁶.

3.6.2 Dielectric loss tangent of PVC/CA blends

Figure 9(a-f) shows the plots of dielectric loss tangent (tan δ) of PVC/CA blends across the frequency in the range of from 50 Hz to 20 MHz and temperature range

Table 6 — Comparative values of dielectric constant and dielectric loss			
PVC/CA blends (wt%)	Dielectric constant (ϵ_r)	Dielectric loss tangent (tanδ)	
Virgin PVC	8.48; 50 Hz; 150 °C	3.8; 50 Hz; 150 °C	
Virgin CA	7.23; 50 Hz; 30 °C	0.52; 20 MHz; 30 °C	
PVC/CA (90/10)	17.69; 50 Hz; 150 °C	0.53; 50 Hz; 140 °C	
PVC/CA (80/20)	11.24; 50 Hz; 150 °C	0.42; 50 Hz; 150 °C	
PVC/CA (70/30)	10.77; 50 Hz; 150 °C	6.06; 50 Hz; 150 °C	
PVC/CA (60/40)	15.47; 50 Hz; 150 °C	0.80; 50 Hz; 150 °C	



Fig. 10 — Discrete plot of dielectric constant and loss tangent vs CA weight fraction

of 30-150 °C. The maximum value of dielectric loss tangent (tan δ) for PVC/CA is 6.06 (150 °C, 20 MHz). It is clear from the figure that $tan\delta$ of PVC/CA blends decreases with increasing frequency. This can be attributed to low response of PVC dipole to follow the system variation at high frequency⁴⁷. It is due to interfacial polarization caused by heterogeneous of the system⁴⁸. An additional peak is observed in high frequency region at 1 MHz due to sudden capacitive discharge and produces phase change in I-V across the sample under test. Basically the micro porous membrane nature exhibits phase change from amorphous to crystalline nature that will influence on dielectric properties. The dielectric constant and loss tangent are given in Table 6. The passive effect on polarization is due to the restricted polaribility and interfacial phases⁴⁹.

Figure 10 shows the discrete plot of dielectric constant and loss tangent as function of cellulose acetate (wt%). As dielectric constant increases there is a decrease in loss. The nonlinear trend of polarization and loss tangent is due to the phase variation of polymer blends.



Fig. 11 — Shore 'A' test of PVC: CA: 90/10, 80/20, 70/30, 60/40 blends

3.7 Softness test of PVC/CA blends

In polymer science samples of rubbers, elastomers, fluoro-polymers, vinyl group functional polymers and polyolefin, biological samples and fibers softness property are tested by using the shore 'A' tester⁵⁰⁻⁵². The softness testing is based on the principle of durometer. A metal pin forced in to the polymer matrix and a numerical reading is reflected corresponding to the resistance of material under test.

Figure 11 demonstrated the softness by shore 'A' as a function of CA loading (wt%). The test reflects the 44 % increase in softness which is due to the decrease in interplanar network. This is confirmed and co-related by the XRD investigation. The softness of PVC/CA blends is influenced by CA due to the amorphous phase in blends.

4 Conclusions

A PVC/CA blends were successfully prepared by solution casting and characterised by different analytical techniques. The XRD spectra revealed the decrease in inter-planar distance. The CH₂ bond shift due to weight fraction of CA was confirmed by FTIR spectra. The direct band gap values decreased due to conjugation. The micro pores are identified by SEM analysis in PVC/CA blends and combined effect of phase misibility. In TGA the thermal stability of blends was increased due to weight fraction of CA. The dielectric constant decreased under broadband frequency. The dielectric loss tangent decreased (0.53 for 10 wt% to 0.42 for 20 wt %) with CA weight fraction. The shore 'A' test confirmed the 44% of increase in softness.

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