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Development and Characterization of Hydroxy Propyl Cellulose/Poly(vinyl alcohol) Blends and Their Physico-Chemical Studies

K. Sudarsan Reddy[a], M.N. Prabhakar[a], K. Madhusudana Rao[b], D.M. Suhasini[b],
V. Naga Maheswara Reddy[a], P. Kumara Babu[a], K. Sudhakar[a], A. Chandra Babu[b],
M.C.S. Subha[b], K. Chowdoji Rao[a]*

[a]Department of Polymer Science and Technology, S.K. University, Anantapuram, Andhra Pradesh, India
[b]Department of Chemistry, S.K. University, Anantapuram 515003, Andhra Pradesh, India.
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ABSTRACT

The preparation of series of hydroxy propyl cellulose/Poly (vinyl alcohol) blends with different weight ratios and their miscibility studies using simple physical techniques such as viscosity, ultrasonic velocity, and refractive index were carried out at 30 °C. The effect of concentration, composition on the miscibility of the blends has been reported. The plots of reduced viscosity versus concentration of HPC/PVA blends vary linearly over the entire composition range. The interaction parameters ΔB and μ proposed by Chee and α proposed by Sun have been obtained using the viscosity data to probe the miscibility of the polymer blends and the results suggest that HPC/PVA blends were completely miscible in all proportions. The conclusions drawn from viscosity data have been confirmed by the ultrasonic velocity and refractive index studies also. The films of the blends were prepared by solution casting method using water as solvent. The prepared films have been characterized by analytical techniques such as FTIR, DSC, X-RD and SEM to probe the miscibility of the blends and concluded that HPC/PVA blends are miscible at all the compositions due to the strong intermolecular hydrogen bonding interactions between HPC and PVA.

Keywords: *Polymer blend miscibility, HPC, PVA, FTIR, DSC, X-RD, SEM*

1. INTRODUCTION

Properties of individual synthetic or natural polymers alone are often inadequate to produce materials with good chemical, mechanical, thermal, and biological performance properties. Blends of synthetic polymers with biological macromolecules have been prepared to obtain polymeric biomaterials [1] with special enhanced properties for certain applications. The blends were produced in different forms, like films, sponges or hydrogels and were evaluated as dialysis membranes[1], wound dressing [2] and drug delivery systems [3,4] etc.

To obtain polymer blends with desired & more useful properties polymer-polymer miscibility is important criteria which may arise from any specific interactions, such as hydrogen bonding, dipole-dipole forces and charge transfer complexes between the consistent homopolymers in the blend. There have been various techniques such as SEM, DSC, X-RD, & FTIR of studying the miscibility of the polymer blends [5-7]. Some of these techniques are complicated, costly and time consuming. Hence it is desirable to identify simple, low cost and rapid techniques also to study the miscibility of polymer blends. Chee [8] and Sun, Wang, and Feng [9] have suggested the viscometric method for the study of polymer-polymer miscibility in solution. Singh and Singh [10-12] have also suggested the use of ultrasonic velocity and viscosity measurements for investigating the polymer miscibility in solution. Palladhi and Singh [13] have shown that the variation of ultrasonic velocity and viscosity with blend composition is linear for miscible blends and non-linear for immiscible blends. Recently Chowdoji Rao et al. [14-16] used ultrasonic and refractive index techniques to study the miscibility of polymer blends.

HPC is an alkyl-substituted hydrophilic cellulose derivative that not only has a particular phase transition behavior in aqueous solution [17-18], and in some solvents [19-21], but also has many advantages such as excellent film forming properties, degradability, biocompatibility [22,23] etc. HPC has been a focus of research because of these unusual and desirable properties, and its prospects in industrial applications [24-26]. Poly (vinyl alcohol) (PVA) is a synthetic water-soluble polymer with good film forming property, which offers good tensile strength, flexibility and barrier properties to oxygen and aroma [27]. The biodegradability and water solubility of PVA

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*Corresponding Author:
E-mail address: chowdojirao@gmail.com

ensures its easy degradation and elimination after use.

Literature reveals that there were no reports on the compatibility studies of HPC and PVA blend. Besides this the author has selected these two polymers not only as they have H-bonding groups [28, 29], which are useful for preparing compatible blends but also they have many pharmaceutical and industrial applications. In continuation of our research on miscibility studies [14-16], in the present work, viscosity, ultrasonic velocity and refractive index techniques were used to determine the miscibility of HPC/PVA blends in water at 30 ^{o}C. Further HPC/PVA blend films were prepared from solution casting method, and also studied their miscibility by FTIR, SEM, X-RD and DSC techniques. These miscibility studies of blends of natural and synthetic polymers are useful for preparing biodegradable polymer blend materials which is further useful to avoid polymer pollution. The results are presented here.

2. EXPERIMENTAL

2.1 Materials

Hydroxy Propyl Cellulose (M_W =1,40,000) was purchased from sigma Aldrich, USA. Poly(vinyl alcohol) (M_W=57,600) was purchased from Molychem, Mumbai, (India) and were used without further purification. Double distilled water having almost zero conductivity was used as solvent.

2.2 Preparation of Blend Solutions

1 wt % of HPC and 1 wt % of PVA solutions were prepared by dissolving 1 g of each polymer in 100 mL of distilled water in two separate stoppared conical flasks. Different blend solutions of HPC and PVA were prepared by mixing HPC with PVA in the weight ratios of 0/100, 20/80, 40/60, 50/50, 60/40, 80/20, and 100/0. From each of these blend solutions, 0.1, 0.3, 0.5, 0.7, and 0.9 (w/v) concentrated solutions were used for the measurement of solution viscosity, ultrasonic velocity, and refractive index. The total weight of the two components in the solution was always maintained at 1.0 g/dl.

2.3 Preparation of Blend Films

Blend films of HPC with PVA were prepared by solution casting method. Required amount of HPC was dissolved in distilled water by stirring over a magnetic stirrer (Jenway, model 1103, UK) for 24 h. To this 20, 40, 50, 60 and 80 weight% (with respect to HPC) of PVA were added. Solutions were mixed uniformly and filtered to remove any foreign floating or suspended particles. The respective solution was poured on to a clean glass plate, leveled perfectly on a tabletop kept in a dust-free atmosphere and dried at room temperature. The dried films were peeled off carefully from the glass plate and used for further analysis and characterization by using different techniques.

2.4 Techniques:

2.4.1 Solution Techniques

Viscosity and density measurements were made at 30^{0}C using Ubbelohde suspended level viscometer (with the flow time of 95 sec for distilled water) and specific gravity bottle respectively. The required temperature was maintained within ±0.05 ^{o}C. The ultrasonic velocities of the blend solutions with different compositions, viz, 0/100, 20/80, 40/60, 50/50, 60/40, 80/20 and 100/0 by weight were measured at 30 ^{o}C using ultrasonic interferometer manufactured by M/s. Mittal enterprises, New Delhi. The constant temperature was maintained by circulating water from a thermostat with a thermal stability of ±0.05 ^{o}C through the double walled jacket of ultrasonic experimental cell. The experimental frequency was 2MHz and the velocity measurements were accurate to better than ±0.05%. The refractive indices of the solutions were measured directly with an Abbe's Refractometer (digital) with thermostated water circulation system at 30 ^{o}C as explained in the earlier publications of Chowdoji Rao et al.[14] The accuracy of the refractive index measurement is ±0.02%.

2.4.2 Fourier Transform Infrared Spectroscopy

Fourier transform infrared (FTIR) spectra of HPC, PVA and their blend films were taken using Bomen MB-3000 FTIR spectrometer to study the nature of molecular interactions. Blend films were characterized at room temperature from 4000 to 400 cm^{-1} under N_2 atmosphere at a scan rate of 21 cm^{-1}.

2.4.3 Differential Scanning Calorimetry

DSC was used for the assessment of thermal properties of the investigated materials. DSC curves of HPC, PVA and their blend films of different compositions were recorded using TA instruments differential scanning calorimeter (Model: SDT Q600, USA). The analysis of samples was performed at heating rate of 20 ^{o}C/min under N_2 atmosphere at a purge speed of 100ml/min.

2.4.4 X-Ray Diffraction

The X-ray diffraction (XRD) patterns of the blend samples were obtained with an Intel diffractomer (Paris, France) with monochromatized Cu Kα radiation (scan speed of 1^{o}/min in a 2θ range of 5^{o}-40^{o}) at room temperature.

2.4.5 Scanning Electron Microscopic Analysis

The scanning electron microscopic (SEM) micrographs of the blend samples were obtained under high resolution (magnification: 150-300X, 5 kV) using JOEL JSM 840 SEM equipped with phoenix energy dispersive system.

3. RESULTS AND DISCUSSION

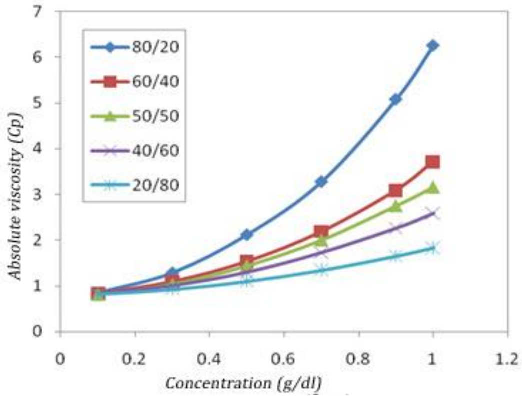


Figure 1: Plots of Absolute Viscosity Vs Concentration for 1% (w/v) HPC-PVA blends.

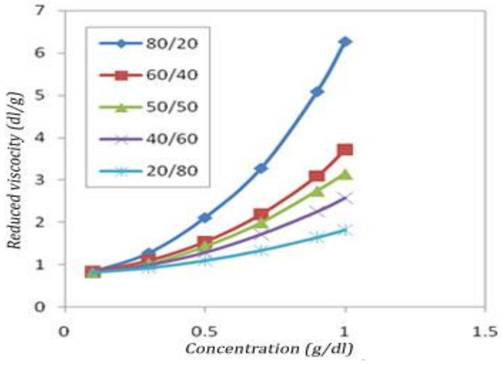


Figure 2: Plots of Reduced Viscosity Vs Concentration for 1% (w/v) HPC-PVA blends.

3.1 Viscometric Studies

The measured values of densities (ρ) and absolute viscosities (η_{ab}) were used to calculate the reduced viscosities (η_{red}) over the wide range of concentrations of the blend solutions for different compositions 20/80, 40/60, 50/50, 60/40 and 80/20 of 1% (w/v) of HPC/PVA blends in water at 30^0C. The absolute viscosity versus concentration curves for the blends of HPC and PVA of different compositions at 30^0C in water are shown in Figure 1. It was well established earlier by many workers [30, 31] that the variation of absolute viscosity versus concentration of blend composition plots are linear for compatible blends and nonlinear for incompatible blends. Based on this, in the present study, the linear variation of the absolute viscosity with concentration for all the blend compositions in water was attributed to the miscibility nature of this blend.

The Huggin's plots of reduced viscosity versus concentration of different compositions of HPC/PVA blends are shown in Figure 2. From these plots, it is also clearly evident that the Huggin's curves are linear in nature and this may be attributed to the mutual attraction of macromolecules in solution that favors the polymer miscibility. A similar observation was made by Ravi Prakash et al. [32] from their viscometric investigations on intermolecular interactions between methyl cellulose/Poly (Ethylene Glycol) (PEG) in water.

Chee and Sun interaction parameters:

To quantify the miscibility of the polymer blends from viscosity studies, Chee [8] suggested that the general expression for interaction parameter when polymers are mixed in weight fractions w_1 and w_2 is as follows:

$$\Delta B = \frac{b - \bar{b}}{2w_1w_2} \tag{1}$$

where b= w_1b_{11} + w_2b_{22} in which, b_{11} and b_{12} are the slopes of the viscosity curves for the pure components. The coefficient b is related to the Huggin's coefficient K_H as

$$b = K_H[\eta]^2 \tag{2}$$

for ternary systems The coefficient b is also given by

$$b = w_1^2b_{11} + w_2^2b_{22} + 2w_1w_2b_{12} \tag{3}$$

where b_{12} is the slope for the blend solution. Using these values, Chee defined a more effective parameter as follows:

$$\mu = \frac{\Delta B}{\{[\eta]_2 - [\eta]_1\}^2} \tag{4}$$

where η_1 and η_2 are the intrinsic viscosities for the pure component solutions. Recently, Sun et al.[9] have suggested a new formula for the determination of polymer miscibility as follows:

$$\alpha = K_m - \frac{K_1[\eta]_1^2w_1^2 + K_2[\eta]_2^2w_2^2 + 2\sqrt{K_1K_2}[\eta_1][\eta_2]w_1w_2}{\{[\eta]_1w_1 + [\eta]_2w_2\}^2} \tag{5}$$

where K_1, K_2 and K_m are the Huggin's constants for individual components 1, 2 and the blend, respectively. From Table 1, It is noticed that the values of ΔB, μ and α are positive for all the compositions of HPC/PVA blends. In general, if ΔB, μ and α are positive for any poly-blend system, it is considered as a miscible one, whereas if these values are negative, the poly-blends are considered as immiscible. Based on this and the values given in Table I, HPC/PVA blends show miscibility nature for all poly-blend compositions. A similar observation was made by Chowdoji rao et al. [33] in the case of xanthan gum/PVP blends. To further confirm this observation, we measured the ultrasonic velocity (u) and refractive index (n) of the blend under consideration at various compositions in water at 30 °C. The variation of the

Table 1. Chee and Sun's interaction parameters for different compositions of 1% (w/v) HPC/PVA blends in water at 30 ^{o}C.

HPC/PVA Blends composition	Chee's Differential Interaction parameters		Sun's miscibility Parameter
	ΔB	μ	α x 10^{2}
20/80	0.0637	0.7114	19.2886
40/60	0.0888	3.0451	2.3939
50/50	0.0375	31.5544	7.1792
60/40	0.0092	13.2295	0.8295
80/20	0.0824	12.8350	3.8986

ultrasonic velocity and refractive index with the blend composition is shown in Figures 3 and 4, respectively.

It was already established [33, 34] that the variation is linear for miscible blend and nonlinear for immiscible blend. In the present study, the variation is found to be linear for all compositions, and this observation is in confirmation with viscosity results. Hence, this study indicates the existence of miscibility over entire composition range due to possible H-bonding interactions taking place between the hydroxyl groups of both PVA and HPC as shown in the Scheme 1. A similar observation was made by Demappa et al. [34] in the case of miscibility of hydroxy propyl methyl cellulose (HPMC)/Poly(vinyl alcohol) PVA blends.

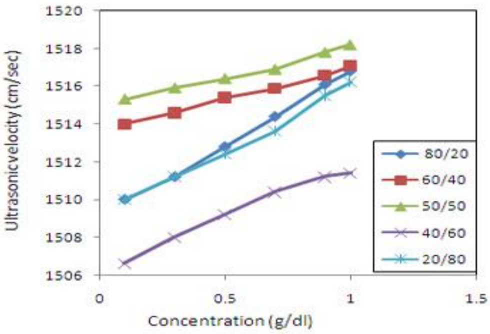


Figure 3: Plots of Ultrasonic Velocity Vs Concentration for 1% (w/v) HPC- PVA blends.

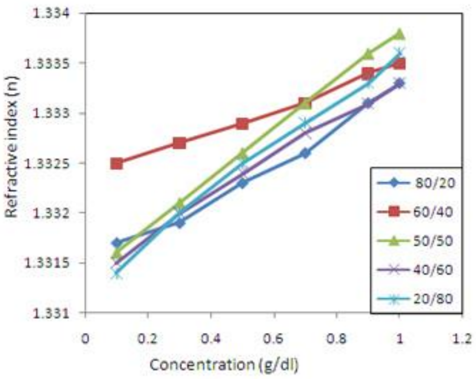
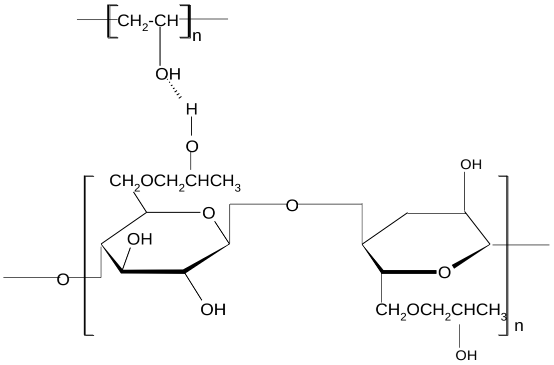


Figure 4: Plots of Refractive Index Vs Concentration for 1% (w/v) HPC- PVA blends.



Scheme I: Schematic representation of hydrogen bonding between PVA and HPMC.

3.2. Miscibility Studies of Blend Films by Characteristic Techniques

3.2.1.Fourier Transform Infrared Spectroscopy Studies

Figure 5. Shows the FTIR transmittance spectra for pure HPC, PVA and the HPC/PVA poly blends samples as functions of wave numbers in the range of 4000-500 cm^{-1}. The formation of strong hydrogen bonds between HPC and PVA was demonstrated by FTIR spectroscopy from the shifts of absorption bands showing hydroxyl stretching vibrations, which were sensitive to the hydrogen bonds formed during blending. The broad transmission bands at 3600-3100 cm^{-1} produced by stretching of the hydroxyl groups in the spectrum of HPC can be remarkably distinguished. It can be seen from the spectrums that the peak intensity and the peak shape were clearly different and these differences were induced by the different blend ratios. The presence of hydrogen-bond structures in some blends could be inferred from the peak shape and peak intensity of the absorption band of the hydroxyl stretching vibrations in the FTIR spectrums [35]. The broad band in the pure HPC spectrum at 3600-3100 cm^{-1}, with a maximum at 3460 cm^{-1}, was assigned to stretching vibrations of the -OH groups. The difference among the curves in Figure 5. a little broadening or shifting or a peak at 3600-3100 cm^{-1} was observed in the transmission band of the HPC/PVA blends when they were compared with that of pure HPC, which suggested that a relative low amount of interaction was presented between the polymers. The hydroxyl stretching vibrations band shifted to a lower wave

number with increasing amounts of PVA. This could be associated with the hydrogen-bonded hydroxyl in HPC. The bands of hydroxyl stretching vibrations had a 6-14 cm^{-1} red shift relative to a free hydroxyl, and the bands of hydroxyl stretching vibrations varied markedly with hydrogen bonding interactions between the -OH belonging to HPC and the -O- atom of -OH groups belonging to PVA. However, the free and associated hydroxyl groups in macromolecules drove to equilibrium via hydrogen bonds. The greater the amount of free hydroxyls in the structure of the HPC cellulose chains, the stronger the hydrogen bonds between the blending constitutes and the vice versa. A schematic hydrogen bond between the HPC and PVA is shown in Scheme 1.

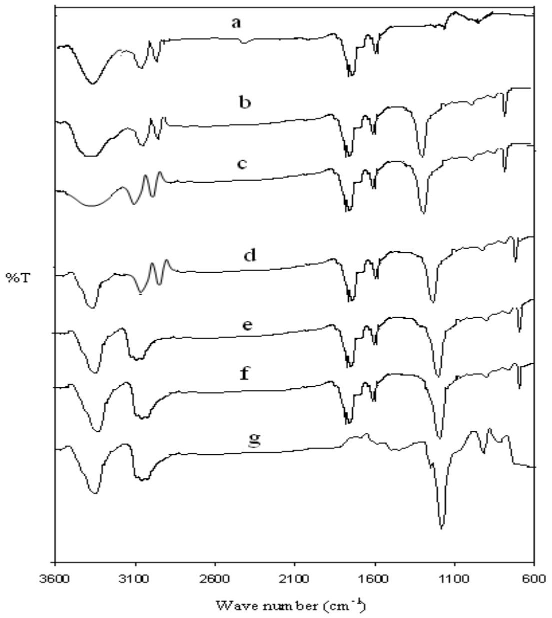


Figure 5: FTIR Spectra for the PVA (a), HPC/PVA 20/80 (b), HPC/PVA 40/60 (c), HPC/PVA 50/50 (d), HPC/PVA 60/40 (e), HPC/PVA 80/20 (f) and HPC (g).

3.2.2. Differential Scanning Calorimetric Studies

Figure 6. shows the DSC curves for pure PVA (g), pure HPC (a), and the HPC/PVA blend samples in different compositions (b to f). The difference in the shape and area of the melting endotherms were noticed. As pure HPC is more amorphous in nature it shows a broader endotherm compared to that of pure PVA which is a crystalline and shows sharp endotherm. The blends between pure HPC and pure PVA will vary the broad area of the endotherm peak depending on the ratio of the polymers. The area became larger and broader with a little sharpness occurred with increase in the content of HPC in the blends. This variation was attributed to the different degrees of crystallinity found in the samples with different blend concentrations. The observed change in endotherm area may have indicated the existence of polymer-polymer interactions between HPC and PVA molecules, through H-bonding between –OH groups of HPC and -O- atom of –OH groups of PVA.

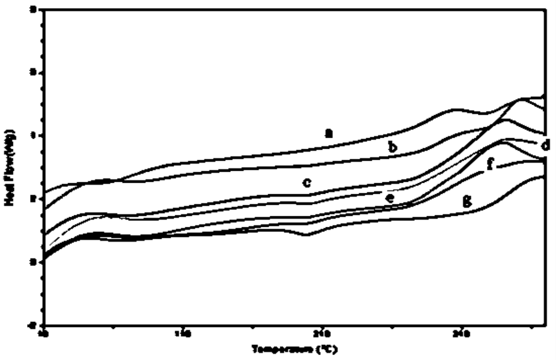


Figure 6: DSC thermograms of HPC (a), HPC/PVA 80/20 (b), HPC/PVA 60/40 (c), HPC/PVA 50/50 (d), HPC/PVA 40/60 (e), HPC/PVA 20/80 (f) and PVA (g).

It is also clear from Figure 6. that T_g for pure PVA is observed at 63.21^0C and the T_g for pure HPC is observed at 55.80°C. It is difficult to analyze the difference in the values of T_g's for the HPC/PVA blends, because the difference in T_g between two blend components is only about 8.6°C. We did not expect that two separate transition temperatures would be resolved. If T_g values of the blended polymer is intermediate between those of the parent polymers, this means that it is a miscible polymer blend. Such reports were there in the literature [36, 37] with regard to the miscibility studies of polymer blends by DSC technique. In the present study HPC/PVA blends show T_g values in between pure compounds hence the present blend is miscible one.

3.2.3. X-Ray Diffraction Studies

XRD has been used by many researchers to characterize changes in the crystal structure parameters, including the degree of crystal orientation, the apparent crystal size, and the lattice strain along the axis of the crystal unit cell. [38] Analysis of these diffractions yields a great deal of valuable information on the configuration of macromolecules and the size of the ordered regions of the material. XRD was used to check the crystalline formation [39] of pure PVA, pure HPC and 50/50 of HPC/PVA blend sample in the present study. Their typical XRD patterns are represented in Figure 7 and it is observed not much change in the intensity of the XRD peaks of the blended sample compared to the pure polymers. The peaks obtained in pure PVA at $2\theta=17.271$, pure HPC at $2\theta=17.021$ are similar to 50/50 HPC/PVA blend at $2\theta=17.950$. This can be attributed to the H-bonding interactions between the two polymers. This again

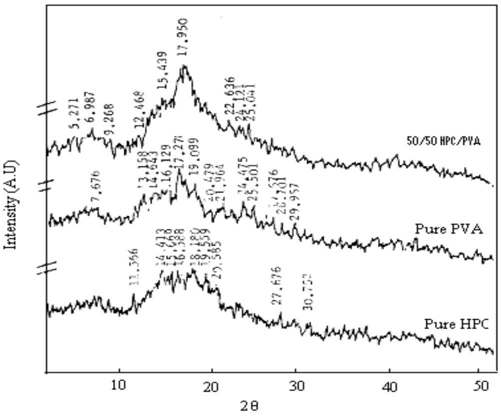


Figure 7: X-ray diffraction patterns HPC, PVA and its blend composition.

is an indication of complete miscibility of the blend.

3.2.4. Scanning Electron Microscopy Studies

Figure 8. shows the SEM images of HPC (a), PVA (b) and their blends of different compositions 20/80 (c), 50/50 (d), and 80/20 (e). The surface morphology of HPC and PVA films were homogeneous. It is also noticed from SEM studies that the morphology of the blends show homogeneity as observed in case of pure and HPC and pure PVA (Figure 8.). No phase separation is observed in the SEM images of the blends compared to SEM images of pure compounds. Hence, we can conclude that the blends of different formulations are miscible in nature.

4. CONCLUSIONS

Miscibility of HPC/PVA blend was investigated by different techniques. Miscible parameters derived from the classical Huggins equations were used to estimate the miscibility of these polymer pair. The effect of concentration of the blends, composition of the blends on the miscibility of HPC/PVA was studied, based on, the viscosity, ultrasonic velocity and refractive index measurements. It is concluded that HPC/PVA blend is found to be completely miscibility over the entire composition range.

The blends were also characterized by different analytical methods such as FTIR, DSC, X-RD and SEM methods. These results support the miscibility window obtained by simple physical techniques. As the prepared polymer blend films and their corresponding homopolymers are transparent in nature, these observations clearly supports the miscibility of the blends. This may be due to the formation hydrogen bond between the oxygen atom of -OH groups PVA and free hydroxyl groups of HPC.

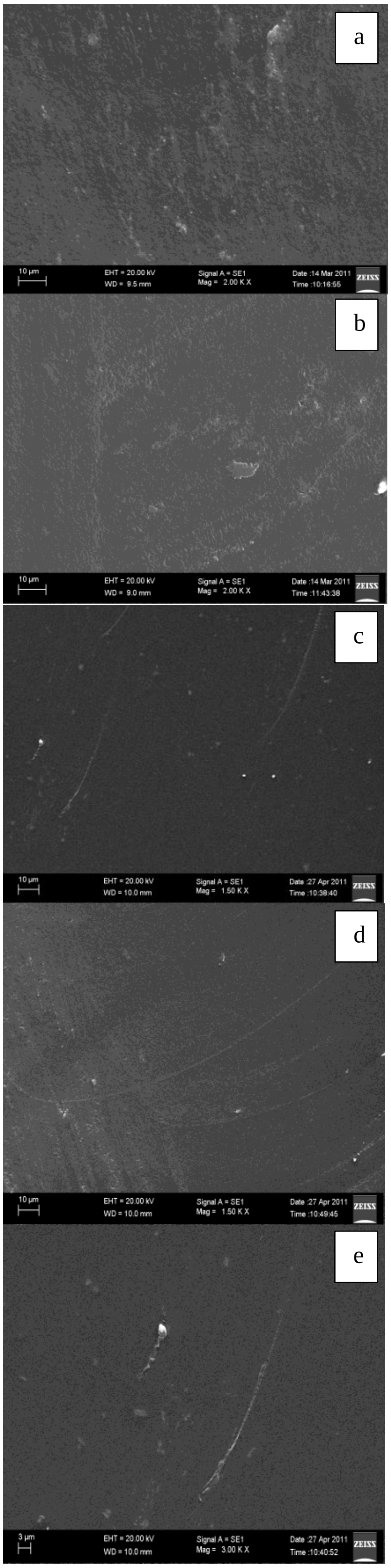


Figure 8: SEM images of HPC (a), PVA (b), 20/80 HPC/PVA (c), 50/50 HPC/PVA (d), 80/20 HPC/PVA (e).

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