MISCIBILITY STUDIES OF HYDROXYETHYL CELLULOSE AND POLY (VINYLPYRROLIDONE) BLENDS

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ABSTRACT

Miscibility characteristics of Hydroxyethyl Cellulose and Poly (vinyl pyrrolidone) inconnection solvent water were studies by density, viscometry, ultrasonic velocity and refractometric techniques at 30° C. Blend films of Hydroxyethyl Cellulose/PVP were prepared by solution casting method and characterized by differential Scanning Calorimetry (DSC), Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopic techniques. Using the viscosity data, the interaction parameters of Chee's (μ) and Sun's (α) were computed to determine their miscibility their miscibility. The obtained values revealed that blends were miscible in all compositions at 30° C. The results were then confirmed by ultrasonic velocity, refractive index, FT-IR, DSC and SEM techniques. Compatability in the above compositios may be due to the formation of hydrogen bonding between hydroxyl groups in the HEC and the carbonyl group in PVP.

Key Words: Hydroxyethyl Cellulose, Poly (Vinylpyrrolidone), Miscibility, Blends, Ultrasonic, SEM

INTRODUCTION

To satisfy the growing needs of new materials with specific properties such as engineering materials, new polymers have been synthesized (Krause *et al.*, 1978; Varnell *et al.*, 1983 and Varnell and Coleman, 1981) and chemical modifications in conventional polymers have also been proposed (Woo *et al.*, 1986 and Cabanclas *et al.*, 2005). However, the mixture of two or more polymers, forming a polymer blend, continues to be an economical method to obtain new polymeric materials (Crispim *et al.*, 1999). The final properties of a polymeric blend will commonly depend on the properties of its polymeric components, its composition and mainly on the miscibility of the constituent polymers (Jiang and Han, 1998). In some cases, by synergistic effects, the blend can exhibit better properties than the pure components (Crispim *et al.*, 1999 and Jiang and Han, 1998). Generally, the polymer – polymer miscibility is due to some specific interactions like dipole-dipole forces, hydrogen bonding and charge transfer complexes between the polymer segments (Crispim *et al.*, 1999 and Jiang and Han, 1998) and Jiang and Han, 1998.

Several works on polymer–polymer miscibility have been developed in the last 20 years (Chee, 1990 and Sun *et al.*, 1992). For such investigations, the techniques most commonly and widely used are electron microscopy (Singh and Singh, 1983), spectroscopy (Singh and Singh, 1983), thermal analysis (PAlladhi and Singh, 1994), and inverse gas chromatography (Palladhi and Singh, 1994). Other techniques using alternative properties or less expensive techniques, for instance, viscometry, ultrasonic velocity, and refractometry methods, have also been proposed (Naveen *et al.*, 2010; Defaye and Wong, 1986; Fennema, 1996; Hirano *et al.*, 1978; SBP Handbook, 1998 and Haiyand *et al.*, 1998). Chee (1990) and Sun *et al.* (1992) suggested the viscometric method for the study of polymer–polymer miscibility. Paladhi and Singh (1984; 1994) showed that the variation of ultrasonic velocity and viscosity with blend composition is linear for miscible blends and nonlinear for immiscible blends. Basavraju *et al.* (2007) also used refractive index method for the miscibility of polymer blends.

A combination of synthetic and natural polymers results in new materials, which have useful properties such as good mechanical properties, easy process ability, low production, transformation costs and biocompatibility typical for biopolymers (Guru *et al.*, 2010). These blends have already been used as

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biodegradable biomaterials (Thomas and Gopinathan, 1988), drug delivery systems (Nagmani *et al.*, 2002), membranes (Cai *et al.*, 2003), and materials for agricultural application.

As a part of our research programme (Sudarsan *et al.*, 2012; Sudarsan *et al.*, 2012 and Venkata *et al.*, 2011), we have studied the miscibility of Hydroxyethyl cellulose/PVP blends using viscometry, Ultrasonic velocity, density, refractometry, FT-IR, DSC and SEM techniques. We have selected these polymers because they have many pharmaceutical and food industrial applications. Hydroxyethyl Cellulose is water soluble cellulose ether and is non-ionic carbohydrate polymer, which is compatible with a wide range of other water-soluble polymers (Chanachai, 2000; Nicholson and Merritt, 1985 and Savge and Ethers, 1971). Poly (vinyl pyrrolidone) is an amorphous polymer, it has several pharmaceutical applications (Weber and Molenaar, 1970). In addition, PVP is able to form complexes with a wide range of compounds through H-bond formation between its carbonyl group and the hydroxyl groups of water, alcohol, and hydroxyl containing polymers like Poly (vinyl alcohol) (Mosakala *et al.*, 1985 and Goh and Sion, 1990).

In spite of the intensive investigation on inter polymer complexation between HEC and PVP, as well as their emerging perspective in medical applications, to the best of our knowledge there is no study devoted to the miscibility of these polymers. Consequently, in the present work the miscibility of these polymers were investigated.

MATERIALS AND METHODS

Materials

Two water soluble polymers, Hydroxyethylcellulose (Mw= 1,00,000) and Poly(vinyl pyrrolidone) (Mw= 40,000) purchased from s.d. fine chemicals, Mumbai and used without further purification for this work. Double distilled and deionized water having almost zero conductivity was used as solvent.

Preparation of blend solutions

Two kinds of polymer powders (HEC and PVP) were dried in a vacuum oven for 24 hrs at 50° C to remove the free water from the samples before use. HEC and PVP were separately dissolved in water to form 1% (w/v) solution. It is worth mentioning that in the preparation of HEC solution, the HEC powder must be slowly added to the solvent with gently stirring for about one week at room temperature to obtain a homogenous solution, otherwise HEC tends to form sticky clumps when large amounts of samples were added to the solvent. The blends of stock solutions (20/80, 40/60, 50/50, 60/40 and 80/20) of HEC/PVP were prepared by stirring the mixtures at room temperature for about 45 minutes. Using the above pure and blended stock solutions eight different blend solutions of HEC and PVP were prepared by mixing HEC with PVP 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 density, viscosity, ultrasonic velocity and refractive index.

Preparation of blend films

Blend films of HEC with PVP were prepared by solution casting method. Required amount of HEC was dissolved in distilled water by stirring over a magnetic stirrer (Model, 1103 and Jenway, UK) for 24 h. To this, 20, 40, 50, 60, and 80 wt % (with respect to HEC) of PVP were added. Solutions were mixed uniformly and filtered to remove any foreign floating or suspended particles. The respective solution was poured onto a Teflon-coated clean glass plate, leveled perfectly on a table top kept in a dust-free atmosphere, and dried using IR lamp at room temperature. The dried thin films were peeled off carefully from the glass plate and were found to be transparent. The prepared films of pure polymers and blends were characterized using FTIR, DSC and SEM methods. The procedure for the measurements for these techniques is explained in chapter 3.

Techniques

The Viscosity measurements were made at 30° C using Ubbelohde suspended level viscometer with the flow time of 95 sec for distilled water. Density measurements have been carried out at 30° C using specific gravity bottle. The temperature was maintained in a thermostat bath; with a thermal stability of $\pm 0.05^{\circ}$ C.

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The accuracy in the measurements of viscosity and density are $\pm 0.5\%$ and ± 0.0002 g/cm³ respectively. 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 (w/v) weight were measured at 30^oC using ultrasonic interferometer (Venkata *et al.*, 2011). The constant temperature was maintained by circulating water from a thermostat with a thermal stability of $\pm 0.05^{\circ}$ C through the double walled jacket of ultrasonic experimental cell. The experimental frequency was 2MHz and the velocity measurements were accurate to better than $\pm 0.5\%$. The refractive indices of blend solutions with different compositions were measured using a Digital Abbe's Refractometer, with a thermostated water circulation system (Chanachai *et al.*, 2000) at 30^oC. The accuracy of the refractive index measurements are $\pm 0.02\%$.

Fourier Transform Infrared Spectroscopy Studies

Fourier transform infrared (FTIR) spectra of HPC, PVP and their blend films were taken using Bomen MB-3000 FTIR spectrometer. Blend films were characterized at room temperature from 4000 to 400 cm-1 under a N2 atmosphere at a scan rate of 21 cm-1.

Differential Scanning Calorimetry Studies

DSC curves of HPC, PVP 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 200C/min under N2 atmosphere at a purge speed of 100ml/min.

Scanning Electron Microscope Studies

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

RESULTS AND DISCUSSION

Viscosity studies

The absolute viscosity Vs concentration curves for the blends of HEC and PVP of different compositions at 30° C in water is shown in the Fig. 1. It was well established earlier by many workers (Haiyand *et al.*, 1998 and Basavaraju *et al.*, 2007), that the variations of viscosity Vs concentration of blend composition

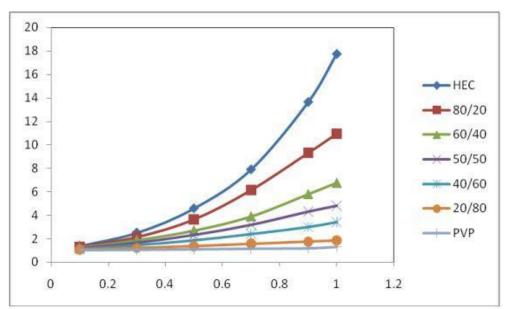


Figure 1: Plots of absolute viscosity □_{ab}(Cp) with concentration of 1% (w/v) HEC, PVP and (HEC/PVP) blends of different compositions in water at 30^oC

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plots are linear for compatible blends and non-linear for incompatible blends. On this basis in the present study the linear variation of the absolute viscosity with concentration for all the blend compositions in water has been attributed to the miscible nature of the blend.

From Fig. 1, it is also observed that as the concentration of HEC increases in the blend composition the absolute viscosity also increases. With the increasing percentage of HEC in the HEC/PVP blend, PVP can interact with several chains of HEC and can form highly cross linked structure, which causes high solution viscosity. Further, with the increase in proportion of HEC in the blend along with cross linking effect, the effect of hydrogen bonding between the OH groups of HEC and -OH groups of PVP also increases. These effects caused due to increasing the contents of HEC in these blends lead to the high viscosity of the blends as observed in Fig. 1.

The Huggins plots of reduced viscosity against concentration of different compositions of 1% (W/V) HEC/PVP blends, pure HEC and pure PVP in water at 30° C are shown in the Fig. 2. From this graph on extrapolating to zero concentration, the intrinsic viscosities are determined and the values of HEC, PVP and HEC/PVP blends of different compositions (20/80, 40/60, 50/50, 60/40, 80/20) are 1.3258, 0.1373, 0.0426, 0.1956, 0.555, 0.5184, and 0.7529 dl/g respectively. The intrinsic viscosity of HEC/PVP blends for different compositions are in between the values of pure polymers. The reasonable interpretation for this may be due to the presence of attractive interactions between HEC and PVP molecules in water. These attractive interactions may lead to decrease the intermolecular excluded volume effect and decrease the hydrodynamic volume. As a consequence, the HEC/PVP blend coils expand, which causes an increase of the intrinsic viscosity of HEC/PVP blends in water.

From this graph (Fig. 2), it is also clearly evident that the Huggins curves are almost linear in nature and this may be attributed to the mutual attraction of macromolecules in solution which favors the polymer miscibility. Similar observation was reported by Haiyand *et al.* (1998) from their miscibility studies of polymer blends through viscosity measurements.

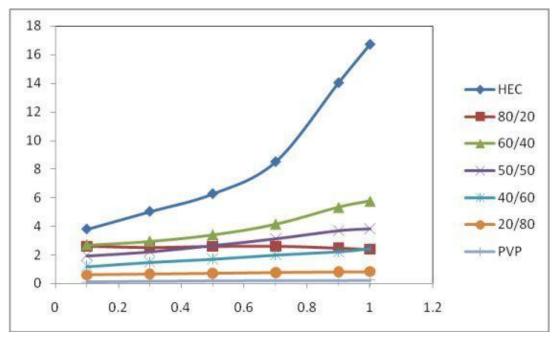


Figure 2: Plots of reduced viscosity \Box_{sp}/C with concentration of 1% (w/v) HEC, PVP and (HEC/PVP) blends of different compositions in water at 30^oC

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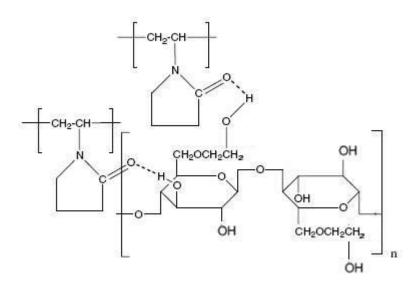
Chee and Sun interaction parameters

In order to quantify the miscibility or immiscibility nature of the present polymer blend studies, the interaction parameters ΔB and \Box suggested by Chee and Sun's interaction parameter α are calculated for the HEC/PVP blend systems in water at 300C and are presented in the Table 1. It is observed from the Table1 that the values of ΔB , \Box , and α are positive for all HEC/PVP blend composition and positive beyond this composition.

Table 1: Chee and Sun's interaction parameters	for different composition of 1% (w/v) HEC/PVP
blends in water at 30 [°] C	-

Blend Composition%	Chee's Differential Interaction parameters µ	Sun's miscibility Parameter α x 10 ³
20/80	1.4536	3.5177
40/60	0.8066	2.0995
50/50	1.2159	2.4143
60/40	0.8221	0.028
80/20	0.2023	0.8193

From the above study, it is concluded that the blends under study are fully miscible over entire composition range due to H-bonding interactions taking place between the carbonyl groups of PVP and the hydroxyl groups of HEC as shown in the scheme 1. A similar observation was reported by G.S. Guru *et al.* (1988) in case of viscosity study of GUM/PVP blends in water.



Scheme 1

Ultrasonic velocity and refractive index measurement studies

In order to confirm the exact nature of the HEC/PVP blends, ultrasonic velocity (u), density (\Box) and refractive index (n) for various blend compositions over a wide range of concentration of the blend at 30^{0} C were measured from the following equations.

$$\Box_{ad} = \frac{1}{\Box u^2} \qquad \dots \qquad (4.3)$$
$$Z = \Box \Box u \qquad \dots \qquad (4.4)$$

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where \Box and u are the density and ultrasonic velocity of the solutions.

The variation of u, \Box_{ad} , Z and n with concentration of different blend compositions at 30^oC is shown in Figs. 3 and 4 respectively. From these Figs., it is noticed that all the above mentioned parameters vary linearly with concentration of blend solution for all blend compositions.

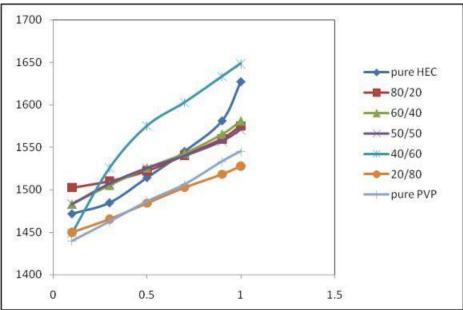


Figure 3: Ultrasonic velocity versus concentration curves for HPC/PEG blends of pure HEC, HEC/PVP (20/80), HEC/PVP (40/60), HEC/PVP (50/50), HEC/PVP (60/40), HEC/PVP (80/20)

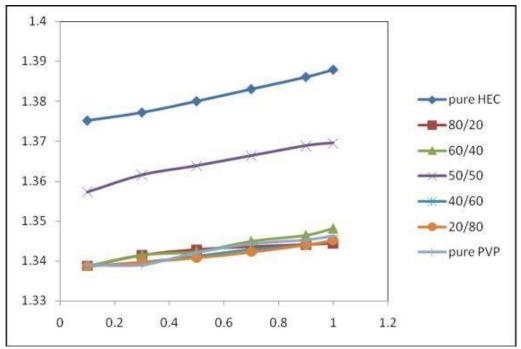


Figure 4: Refractive Index versus concentration curves for HPC/PEG blends of pure HEC, HEC/PVP (20/80), HEC/PVP (40/60), HEC/PVP (50/50), HEC/PVP (60/40), HEC/PVP (80/20)

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It has already been established that if these parameters variation is linear with concentration of blend it is a miscible one and non-linear it is a immiscible blend (Chee, 1990 and Basavaraju *et al.*, 2007). In the present investigation all the parameters $\Box \Box \Box \Box_{ad}$, Z and n) vary linearly with concentration of blend solutions in water for all blend compositions and this confirms the miscibility nature of this blend in water.

We can conclude that the ultrasonic velocity and refractive index results also confirm the conclusions drawn from the viscosity results of this blend solutions explained above. A similar observation was reported by Thomas *et al.* (1988) and Chowdoji Rao *et al.* (2012) from their ultrasonic velocity and refractive index investigations on the compatibility of polymer blends. These results support the conclusions drawn from the viscosity studies.

Miscibility studies of blend films by characteristic techniques

A series of HEC/PVP blend was processed into films by solution casting method by varying HEC content from 0 to 100 wt%. These films are studied by different characterization techniques such as FTIR, DSC and SEM.

FT-IR Studies

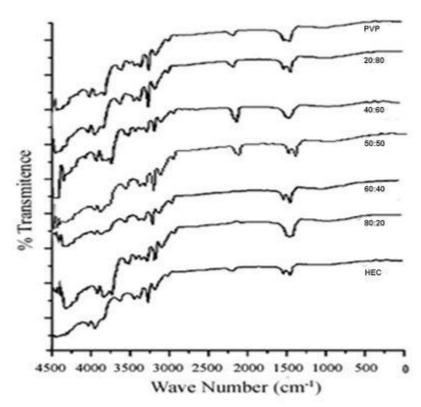


Figure 5: FTIR Spectra for the HEC/PVP (100/0) (a), HEC/PVP (80/20) (b), HEC/PVP (60/40) (c), HEC/PVP (50/50) (d), HEC/PVP (40/60) (e), HEC/PVP (20/80) (f), HEC/PVP (0/100) (g)

Fig. 4 shows the FTIR transmittance spectras for pure HEC, PVP and the HEC/PVP poly blend samples as functions of wave numbers in the range of 4000-500cm⁻¹. The formation of strong hydrogen bonds between –OH groups of HEC and C=O groups of PVP 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 3500-3100cm⁻¹ produced by stretching of the hydroxyl groups in the spectrum of HEC 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

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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 starching vibrations in the FTIR spectrums (Sudarsan et al., 2012). The broad band in the pure HEC spectrum at 3000-3250 cm⁻¹, with a maximum at 3250 cm⁻¹, was assigned to stretching vibrations of the -OH groups. The difference among the curves in Fig. 4, a little broadening or shifting or a peak at 3100-3600 cm⁻¹ was observed in the transmission band of the HEC/PVP blends when they were compared with that of pure HEC, which suggested that a relative low amount of interaction was presented between the polymers. The hydroxyl stretching vibration bands shifted to a lower wave number with increasing amounts of PVP. This could be associated with the hydrogen-bonded hydroxyl in HEC. 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 HEC and C=O groups belonging to PVP. However, the free and associated hydroxyl groups in HEC and C=O group of PVP drove to equilibrium via hydrogen bonds. The greater the amount of free hydroxyls in the structure of the HEC, the stronger the hydrogen bonds between the blending constitutes and the vice versa. A schematic representation of hydrogen bond formation between the HEC and PVP is shown in Scheme. 4.3.1. DSC Studies

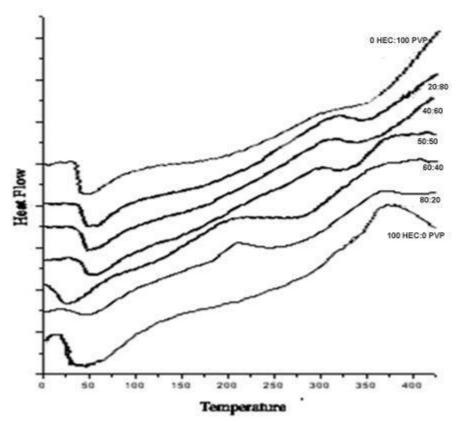


Figure 6: DSC thermograms of HEC/PVP (100/0), HEC/PVP (80/20), HEC/PVP (60/40), HEC/PVP (50/50), HEC/PVP (40/60), HEC/PVP (20/80), HEC/PVP (0/100)

The thermal behavior of the blend was analyzed by DSC method. The DSC thermograms for pure HEC, PVP and their blends are shown in Fig. 4. From DSC thermograms it is noticed that a sharp melting point at 60° C for PVP and broad melting point at 180.7° C for HEC. The broad melting curve for HEC may be

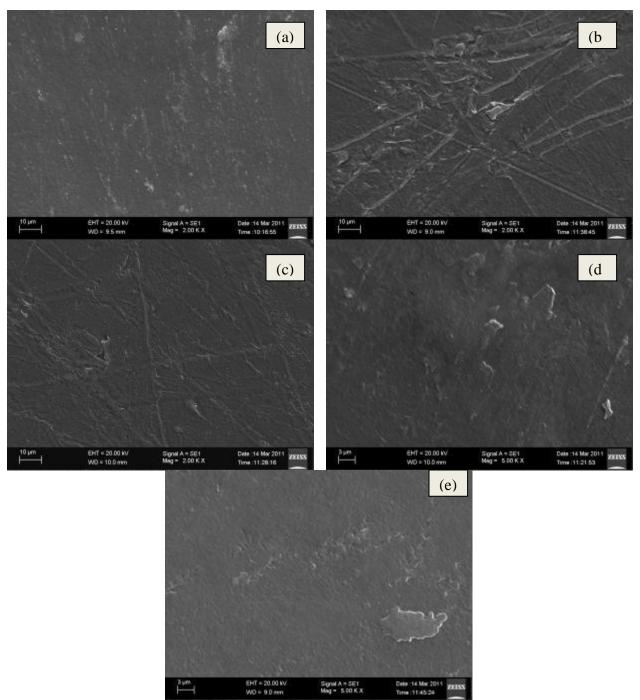


Figure 7: SEM micro photographs of SEM images of (a) pure HEC (b) pure PVP (c) 20/80 HEC/PVP (d) 50/50 HEC/PVP (e) 80/20 HEC/PVP

due to the presence of hydrogen bond and strong secondary forces of attraction. The addition HEC into PVP matrix changes the position of PVP peak significantly. In the same way, the introduction of PVP into HEC the transition temperature peak of HEC significantly shifts as can be seen from Fig. 4. This result

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clearly indicates interaction between PVP and HEC network and this data supports the miscibility window of HEC/PVP.

SEM Studies

Fig. 4 shows the SEM images of HEC (a), PVP (b) and their blends of different compositions 20/80(c), 50/50(d), and 80/20(e). The surface morphology of HEC and PVA films were homogeneous. It is also noticed from SEM studies that the morphology of the blends show homogeneous nature as observed in case of pure HEC and pure PVP (Fig. 4). No phase separation is observed in the SEM images of the all the compositions of the blends compared to SEM images of pure compounds. Hence, we can conclude that the blends of different formulations are miscible in nature.

Conclusions

Miscibility of HEC/PVP was investigated by different techniques. Miscible parameters derived from the classical Huggins equation were used to estimate the miscibility of these polymer pairs. The effect of concentration of the blends, composition of the blends on the miscibility of HEC/PVP blends was studied, based on, the viscosity, ultrasonic velocity and refractive index measurements. It is concluded that HEC/PVP 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 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.

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