



Compatibility studies with blends based on hydroxypropylcellulose and polyacrylonitrile

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Abstract

The solubility parameters of hydroxypropylcellulose (HPC) were measured with the viscosity method and estimated from proposing a group contribution approach for the prediction of δ based on the separation of the solubility parameter into three components (dispersion, polarity, and hydrogen bonding contributions). Hansen's three dimensional solubility parameters were calculated approximately through the contributions of the structural groups. The compatibility of the HPC and polyacrylonitrile (PAN) blend system was studied with theoretical calculations as well as experimental measurements. Viscometric methods, polarized optical microscopy (POM), scanning electron microscopy (SEM), infrared spectrophotometric and thermogravimetry analysis (TGA) were employed for this investigation. The viscometric methods and polarized optical microscopy results were obtained from the blended solution in the range of 0–1.0 weight fraction of PAN, and the scanning electron microscopy, infrared spectrophotometric and thermogravimetry analysis results were obtained from the transparent films cast from the blended solutions that contained 0.2, 0.5 and 0.8 weight fractions of PAN, respectively. The results showed an excellent compatibility existed in the blends system, which may due to the strong hydrogen-bonding interaction between the hydroxyl group of HPC and the nitrile group of PAN.

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1. Introduction

In recent years, much attention has been given to the intermediate properties of copolymers and blends with respect to homopolymers produced for some specific functions. Many interesting properties are attributed to the compatibility of these polymers (Drummond, Albers, & Furlong, 1992; Repka & McGinity, 2001). Characterization of the compatibility of polymer blends is very vital because manifestation of their superior properties depends on the compatibility of homopolymers at the molecular level (Kulshreshtha, Singhand, & Sharma, 1988). Homogenous mixing on a molecular scale is a required condition for

thermodynamic compatibility. However, most polymers lack thermodynamic compatibility, and they can reach a relative equilibrium because of higher viscosities of polyblends. Various blending procedures are available such as melting, dry mixing and solution blending. In these studies, solution blending was employed. The compatibility of polyblends was not only calculated theoretically by solubility parameters, but also measured experimentally by Fourier transform infrared spectroscopy (FTIR), polarized optical micrograph (POM), scanning electron microscopy (SEM), viscosimetry and thermogravimetry analysis (TGA).

HPC is an alkyl-substituted hydrophilic cellulose derivative that not only has a particular phase transition behavior in aqueous solution (Cai & Hu, 2003; Nymweya & Hoag, 2000; Suto & Umeda, 1999), and some solvents (Aden, Biab-

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chi, Ciferri, Conio, & Tealdi, 1984; Immaneni, Kuba, & Mchugh, 1997; Jiang, Li, Xiang, & Zhou, 1999), but also has many advantages such as excellent film forming properties, degradability, biocompatibility etc. (Khutoryanskiy et al., 2004; Nurkeeva, Mun, & Khutoryanskiy, 2001). HPC has been a focus of research because of these unusual and desirable properties, and its prospects in industrial applications (Adrados, Galaev, Nilsson, & Mattiasson, 2001; Fujii, Sasaki, & Nakata, 2001; Robitaille, Turcotte, Fortin, & Charlet, 1991). Most studies mainly focused on modified HPC (López-Velázquez, Bello, & Pérez, 2004), HPC hydrogel (Marsano & Bianchi, 2002), crosslinked HPC nanoparticles (Cai, & Hu, 2003) and HPC blends with PVA, PAA, PAA_{Na}, CH, PVME, PVP etc. based on hydrogen-bonding (EL-Zaher & Osiris, 2005; Marsano & Bianchi, 2002; Pawlak & Mucha, 2003; Mucha & Pawlak, 2005; Nurkeeva et al., 2005). However, studies on the miscibility of HPC with PAN are very rare. In this investigation, a solution blending system of polymers containing hydrogen bonding groups (Mamaza & Folarinmi, 1996; Mucha & Pawlak, 2005) was used with HPC and PAN. The compatibilities of the two polymers were investigated by means of FTIR, POM, SEM, viscosimetry, and TGA over the composition range. The compatibilities analyzed by the viscosimetry and POM were measured in the aqueous state, because polymer-polymer interactions are often more important than polymer-solvent interactions (Bonnet-Gonnet, Leikin, Chi, Rau, & Pardegian, 2001). Other forms of analysis were performed on transparent films. The blend of this polymer pair is not only a creative system but might have improved mechanical properties and broader applications.

2. Experimental section

2.1. Materials

A sample of dry hydroxypropylcellulose (HPC) was kindly supplied by Hercules Inc. (Wilmington, DE, USA). Its average molecular weight was 100,000 g/mol and molar substitution was 4. The sample was used without further purification. The sample of PAN containing more than 85% acrylonitrile, about 10% of the acid second monomer and about 5% of the ester third monomer was supplied by the Shanghai JINSHAN petrochemical construction company. DMSO analytical reagent (AR) was purchased from China National Medicines Corporation Ltd. (Shanghai, China).

2.2. Preparation of polymer blends

The two kinds of sample powder were dried in a vacuum oven for 24 h at 50 °C to remove the free water from the samples before use. The blend solutions were prepared by mixing the HPC/DMSO solution (10 wt%) and the PAN/DMSO solution (10 wt%) at the required ratios to obtain the blend solutions of various compositions. It is worth mentioning that in the preparation of the HPC solution, the HPC powder

must be slowly added to the solvent with gently stirring for about one week at room temperature to obtain a homogenous solution, otherwise the HPC tends to form sticky clumps when large amounts of the samples were added to the solvent.

2.3. Methods

2.3.1. Fourier transform Infrared Spectrophotometric analysis (FTIR)

The dry films used in FTIR were prepared as follows: the liquid blends were cast on clean glass plates, and incubated in a dustless oven at 40 °C for 7–10 days to evaporate the solvent. They were dried in a vacuum oven at 50 °C for 12 h and then the resultant membranes were peeled off the glass plate.

The infrared absorption spectra were recorded with Perkin-Elmer 1600 FT-IR spectrometer in the 4000–500 cm⁻¹ ranges from KBr disks after the samples were dried. All spectrums were recorded at ambient temperature at a resolution of 4 cm⁻¹ and 32-times scanning. Spectra diagrams were obtained through subtracting the spectra of DMSO and H₂O from the spectra of each sample with the FTIR software.

2.3.2. Polarized optical micrograph (POM)

Morphological analysis of the HPC/PAN blend solution was performed using a BX51 Olympus polarized microscope. The samples were observed at 400× magnification at room temperature.

2.3.3. Scanning electron microscopy (SEM)

The cross sections of the blended films were examined with a JEOL JSM-5600LV scanning electron microscope at an accelerating voltage of 15 kV. The images were taken at magnifications of 5000× after the samples were coated with a gold-palladium target using a vacuum evaporator for 30 min.

2.3.4. Viscometric method

Viscosity measurements of all the blend solutions were made in a thermostatic transparent water bath at 25 ± 0.1 °C using an Ubbelohde dilution viscometer. Every value was measured three times and then averaged.

2.3.5. Thermal gravimetric analysis (TGA)

Samples studied by thermal gravimetric analysis were measured in the form of films after being dried in a vacuum oven at 50 °C for 12 h. The films were scanned with a Netzsch 209F1 thermogravimetric analyzer at a scan rate of 20 °C/min in a nitrogen atmosphere from room temperature to 850 °C.

3. Results and discussions

3.1. Compatibility based on theoretical calculations

In this paper, ethanol, acetic acid, dimethylacetamide, dimethylsulfoxide, H₂O, *n*-Amyl alcohol, and dimethylform-

amide were chosen to measure the solubility parameters of HPC with an Ubbelohde dilution viscometer. The values of the intrinsic viscosity of HPC in different solvents were plotted against the solubility parameters of the solvents and are showed in Fig. 1. The solubility parameter corresponding to maximum intrinsic viscosity is considered as a solubility parameter of HPC. The Hansen Three Dimensional Solubility Parameters (δ_d , δ_p , and δ_h) were calculated theoretically through the contributions of the structure groups in the polymer using Eqs. (1)–(3) (Hansen, 2000; Liang, 2000). An ideal chemical structure of HPC with an average of three hydroxypropyl groups per anhydroglucose was referenced to calculate the three Hansen solubility parameters, and $\rho_{\text{HPC}} = 1.17 \text{ (g/cm}^3\text{)}$ (Mark, 1999). The $\delta_{\text{max}} = 26.81 \text{ (MPa)}^{0.5}$ of HPC measured with viscometric methods was approximately equal to the δ of PAN ($\delta_{\text{PAN}} = 26.39 \text{ (MPa)}^{0.5}$). Since $\Delta\delta = 0.42 < 0.5$, this was indicative of some extent of possible compatibility between HPC and PAN. The three Hansen Solubility Parameters (HSP) of HPC were $\delta_d = 20.56 \text{ (MPa)}^{0.5}$, $\delta_p = 4.34 \text{ (MPa)}^{0.5}$, $\delta_h = 16.16 \text{ (MPa)}^{0.5}$, and $\delta = 26.50 \text{ (MPa)}^{0.5}$, which was calculated based on Eq. (4). The three HSP of PAN were $\delta_d = 16.09 \text{ (MPa)}^{0.5}$, $\delta_p = 18.96 \text{ (MPa)}^{0.5}$, and $\delta_h = 8.84 \text{ (MPa)}^{0.5}$. It is well known that the compatibility of polymers requires a close δ as well as having similar values for the three Hansen Solubility Parameters (Liang, 2000). The three Hansen Solubility Parameters of HPC clearly did not match those of PAN. Because HPC contains more hydroxyl groups, there is a greater contribution of hydrogen bonding to the solubility parameters of HPC, and therefore, the δ_h value of HPC is greater than that of PAN. The fact that the δ_p value of PAN was greater than that of HPC was attributed to the strong polarity of the

nitrile group present in PAN. The differences in the three HSP values of these two molecules indicated impossible compatibility when they were blended.

$$\delta_d = \sum F_{di}/\bar{V} \quad (1)$$

$$\delta_p = \sqrt{\sum F_{pi}^2/\bar{V}} \quad (2)$$

$$\delta_h = \sqrt{\sum E_{hi}/\bar{V}} \quad (3)$$

$$\delta^2 = \delta_d^2 + \delta_p^2 + \delta_h^2 \quad (4)$$

Eqs. (1)–(3) were used to calculate δ_d , δ_p and δ_h . Eq. (4) was used to calculate δ , where δ_d is defined as the dispersion component, δ_p as the polarization component and δ_h as the hydrogen-bonding component, respectively. F_{di} , F_{pi} and E_{hi} are defined as the molar attraction constant of additive atoms or groups in the repeated structure. Subscript di represents the dispersion component, subscript pi represents the polarization component and subscript hi represents the hydrogen-bonding component. V_i is the molar volume constant.

3.2. Compatibility based on experimental estimation

3.2.1. Spectrophotometric analysis

The liquid blends were cast on clean glass plates, and as HPC and PAN blended, the useful information regarding the structural changes was collected by FTIR analysis. FTIR analysis was based on the identification of absorption bands associated with the vibrations of functional groups presented in HPC and PAN. Fig. 2 shows the FTIR transmittance spectrums for pure HPC, PAN and the HPC-PAN polyblends samples as functions of wave

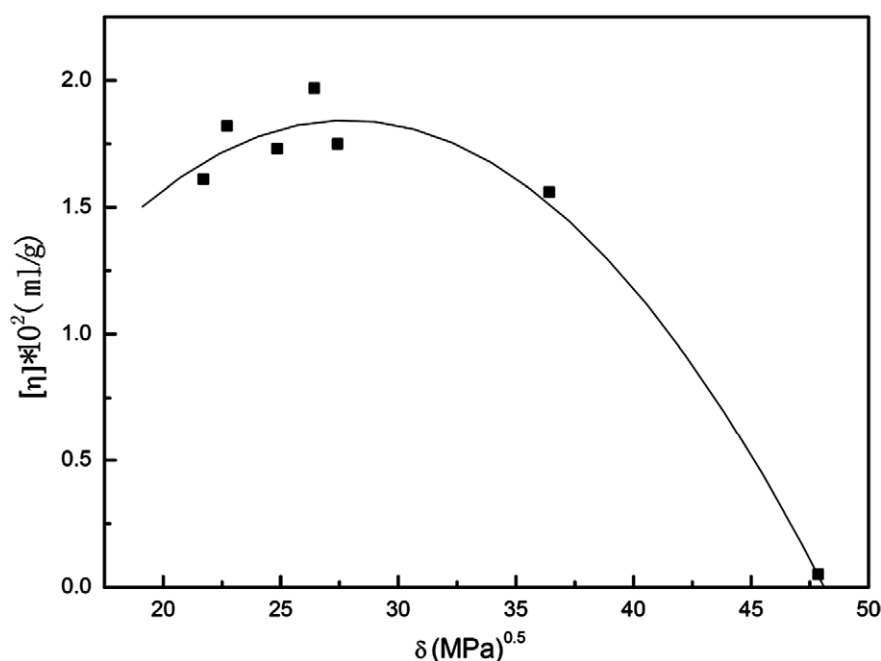


Fig. 1. The dependence of intrinsic viscosity on solubility parameters.

numbers in the range of $4000\text{--}500\text{ cm}^{-1}$. The formation of strong hydrogen bonds between HPC and PAN 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 $3600\text{--}3100\text{ cm}^{-1}$ 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 (Cai, & Hu, 2003). The broad band in the pure HPC spectrum at $3600\text{--}3100\text{ cm}^{-1}$, with a maximum at 3460 cm^{-1} , was assigned to stretching vibrations of the --OH groups. The difference among the curves in Fig. 2, a little broadening or shifting of the peaks at $3600\text{--}3100\text{ cm}^{-1}$ was observed in the transmission band of the HPC/PAN blends when they were compared with that of pure HPC, which suggested that a relative low amount of interaction was present between the polymers. The hydroxyl stretching vibrations band shifted to a lower wave number with increasing amounts of PAN. This could be associated with the hydrogen-bonded hydroxyl in HPC. The bands of hydroxyl stretching vibrations had a $6\text{--}14\text{ cm}^{-1}$ 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 $\text{C}\equiv\text{N}$ belonging to PAN. 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 constituents. A schematic hydrogen bond between the HPC and PAN is shown in Fig. 3.

3.2.2. Micrographs

A common qualitative technique for the estimation of blend compatibility is visual inspection. The morphology

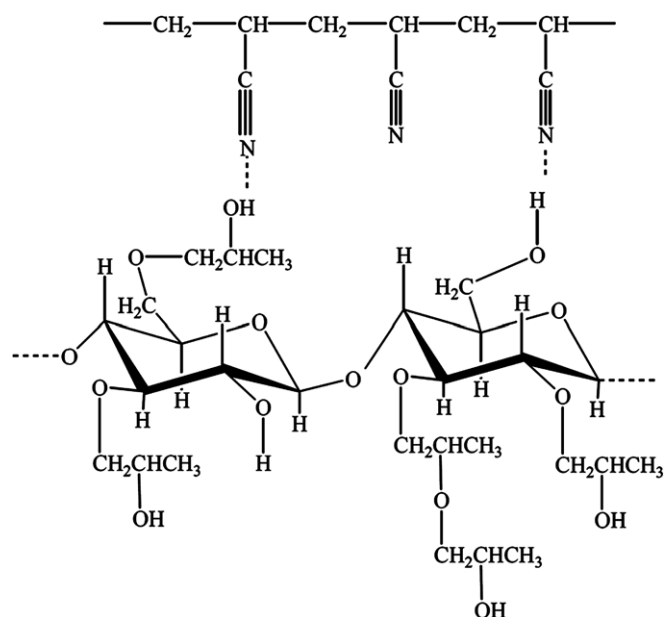


Fig. 3. Schematic representation of the hydrogen bonding between HPC and PAN.

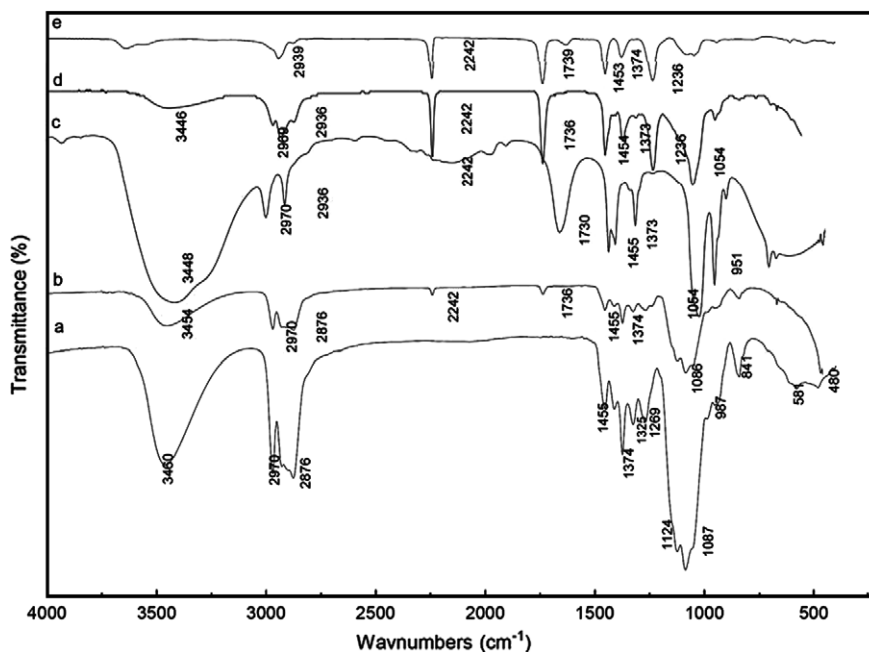


Fig. 2. FTIR spectra of the HPC/PAN blend system. The curve from a to e represents the various weight fractions of PAN, which are as follows: (a) 0; (b) 0.2; (c) 0.5; (d) 0.8; (e) 1.

of blends of two types of hydrogen-bonding systems was studied with a view to understanding the effects of blending on the extent of hydrogen bonding and changes in the morphology. A clearly visible and homogenous transparent solution could be observed under an optical microscope if the blends were compatible. Though, sometimes, optical transparency fails to assure compatibility, and big assemblies or phase separate boundaries were noticed when blends were incompatible. Both optical microscope measurements were done at room temperature (25 °C, 65% relative humidity). The blend system exhibited no large assemblies in the micrograph, except for a few particles as shown in Fig. 4. The color of these blended solutions gradually became yellow with increasing amounts of PAN and looked homogenous and transparent in a scan of the whole sample. No phase separation was observed after standing for one week. This result was also good evi-

dence that HPC and PAN are compatible or at less partially compatible.

3.2.3. Scanning electron microscopy

The films used for SEM measurements were the same as the films used in FTIR analysis. The images of the films containing different weight fractions of PAN were shown in Fig. 5. The morphology of pure HPC and pure PAN films were homogeneous. Besides, many bright stripes were observed in the image of pure PAN. With the addition of PAN to HPC, the morphologies of the blended films changed dramatically. The bright stripes presented in the PAN film disappeared, but no obvious phase separation boundary was visible. Interestingly, the morphologies of the blended films showed an increase in surface roughness, which indicated that the polymers were partially compatible but not incompatible.

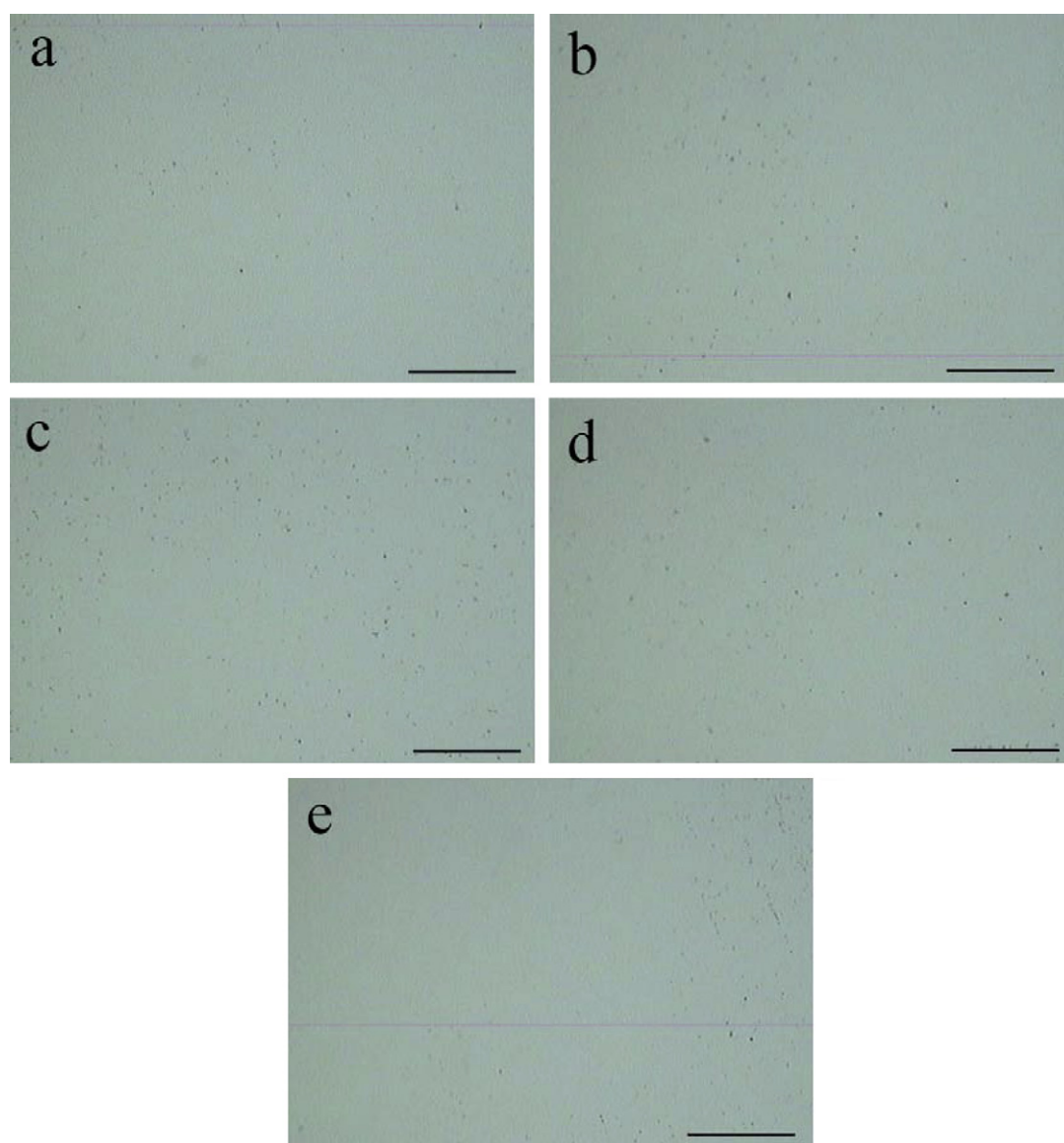


Fig. 4. POM micrographs of the HPC/PAN blend system. The graph from a to e represents the various weight fractions of PAN, which are as follows: (a) 0; (b) 0.2; (c) 0.5; (d) 0.8; (e) 1; bar = 60 μ m.

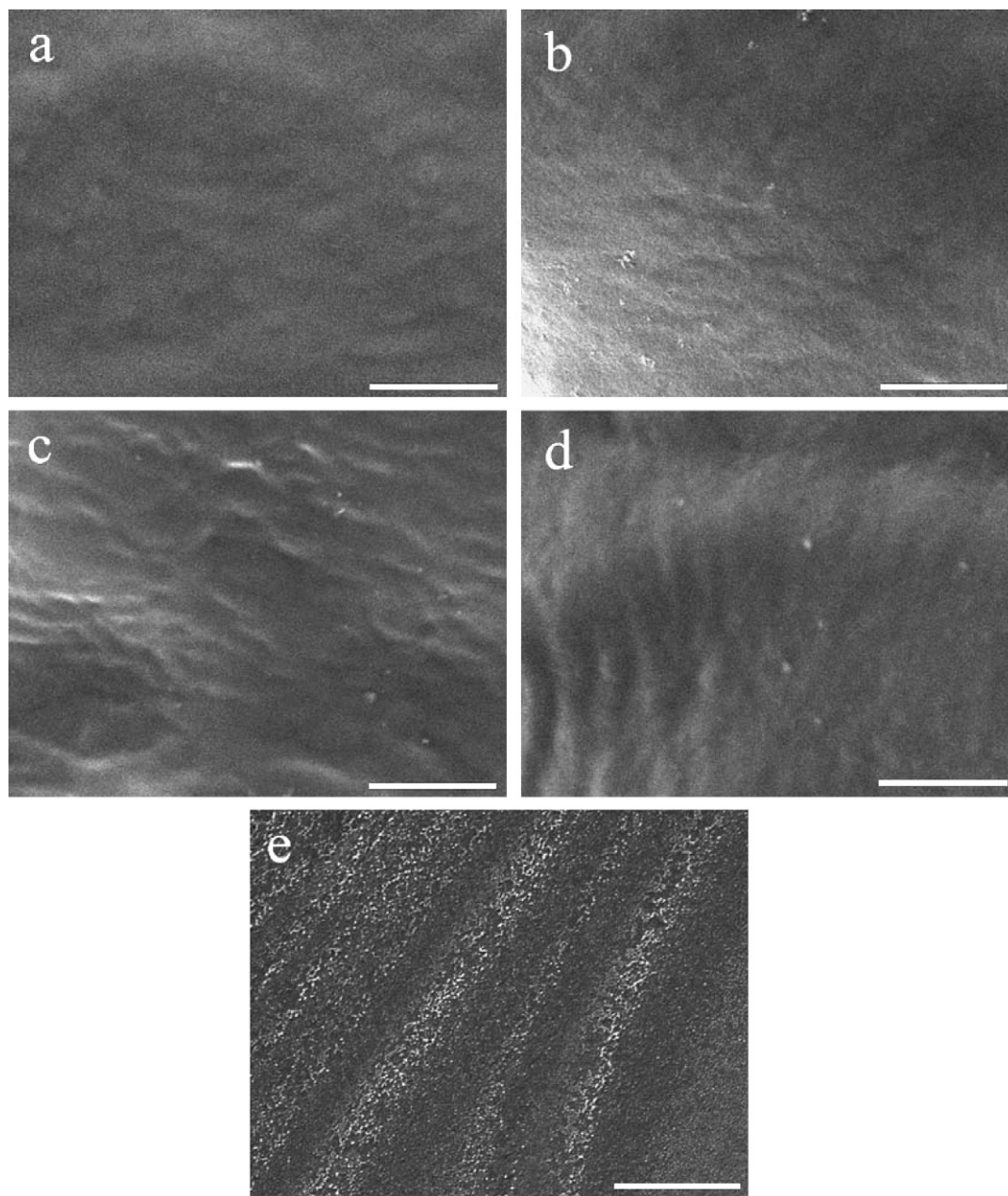


Fig. 5. SEM images of pure HPC film (a), and blended films containing 0.2 (b), 0.5 (c), 0.8 (d) weight fractions of PAN, pure PAN film (e); bar = 5 μm.

3.2.4. Viscosity method

Blends of HPC and PAN were prepared in DMSO with the different weight fractions of PAN 0, 0.2, 0.4, 0.5, 0.6, 0.8 and 1.0. It has been reported that the plots for compatible blends are linear and otherwise the plots are s-type. But for partially compatible blends the plots are between linearity and s-type (Mamaza, & Folarsnmi, 1996). The plots of relative viscosity versus concentration were neither linear nor s-type as shown in Fig. 6, and these curves all had a minimum relative viscosity value at the 0.5 weight fraction of PAN. This suggested that addition of the lower weight fractions of PAN to the solution of HPC was accompanied by a very marked decrease in the viscosity of the solutions and this continued to the 0.5 weight fraction of PAN. The decrease in viscosity is caused by the association between

the two components via hydrogen bonding of the hydroxyl-nitrile functional groups. Then when the weight fractions of PAN were above 0.5, there was an increase in the relative viscosity, which was caused by the presence of excessive amounts of PAN. This viscosity dependence with a minimum is typical for binary polymer systems that have specific interactions. The minimum viscosity corresponds to the stoichiometry of the interacting products. In our research, we have used Eqs. (5)–(7) for calculating viscosities.

$$\eta_{sp} = \eta_r - 1 \quad (5)$$

$$\eta_{sp/c} = [\eta] + K_H[\eta]^2 C \quad (6)$$

$$\eta_{sp/c} = [\eta] + 6K_m[\eta]C \quad (7)$$

$$K_m = K_H[\eta]/6 \quad (8)$$

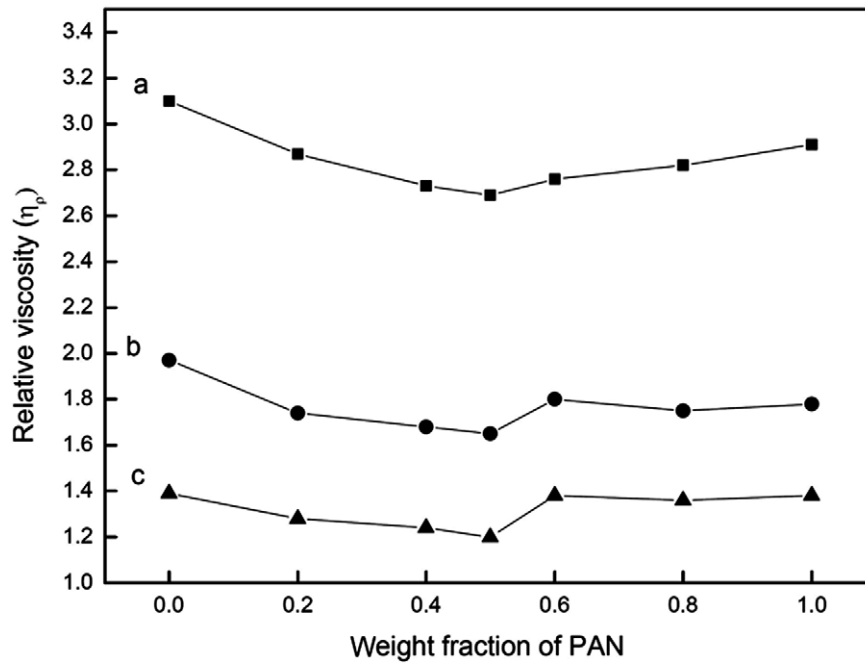


Fig. 6. Dependence of the relative viscosity on the composition plots for HPC/PAN blends (solvent:DMSO, temperature: 25 ± 0.1 °C). Curves from a to c represent various solution concentrations, which are as follows: (a) 0.62 g/100ml; (b) 0.31 g/100ml; (c) 0.16 g/100ml.

The slope b of the plot for $\eta_{sp}/c \sim C$ and the value of K_H were calculated from expressions (5), (6). The dependence of $\eta_{sp}/c \sim C$ on the concentration of the solution and K_H on the amount of PAN were plotted, and are shown in Figs. 7 and 8. The values for the interaction parameter Δb for the different blends are given in Table 1. Positive Δb values (Mahaveer, Udaya, & Jejrai, 2002) for all blend

compositions confirmed the presence of attractive molecular interactions and confirmed the compatibility of the HPC and PAN polymers. As shown in Fig. 7, K_H exhibited a positive deviation from the component's additivity that indicated that the two polymers could be compatible when they were blended. The ideal association constant in solution is calculated by the following Eq. (9).

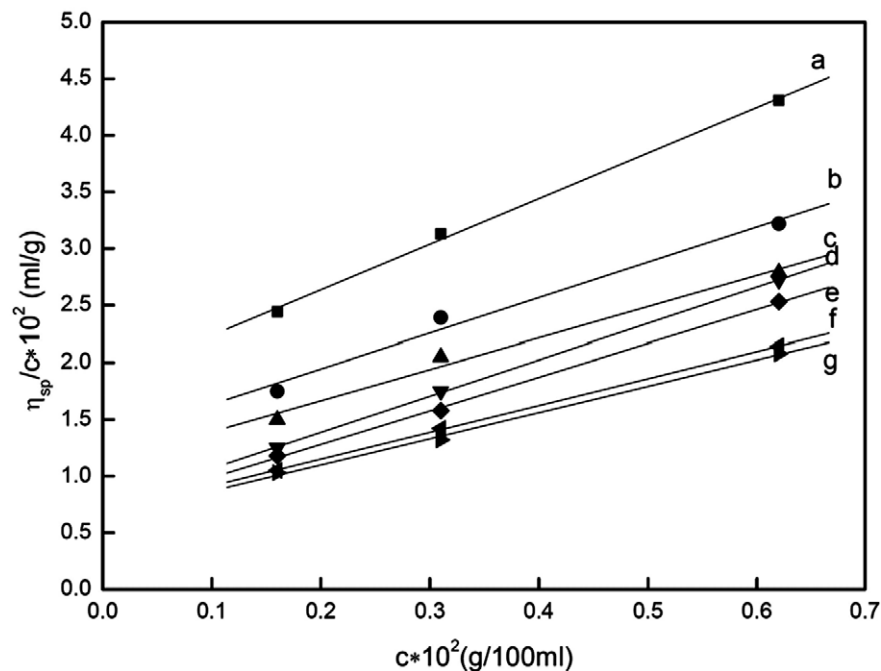


Fig. 7. The dependence of specific viscosity on concentration. The curves from a to g represent the various PAN weight fractions, which are as follows: (a) 0; (b) 0.2; (c) 0.4; (d) 0.5; (e) 0.6; (f) 0.8; (g) 1.

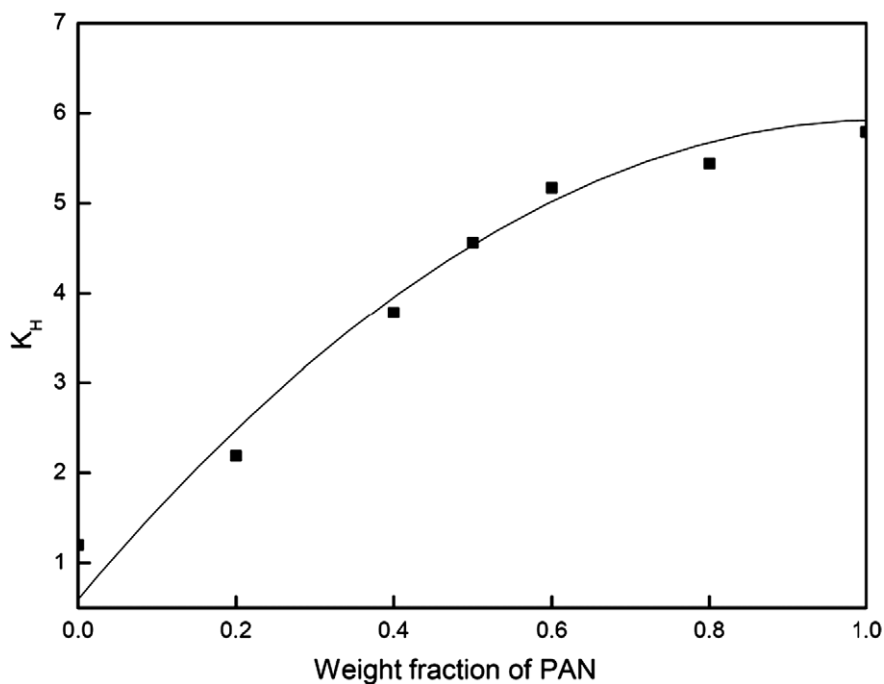
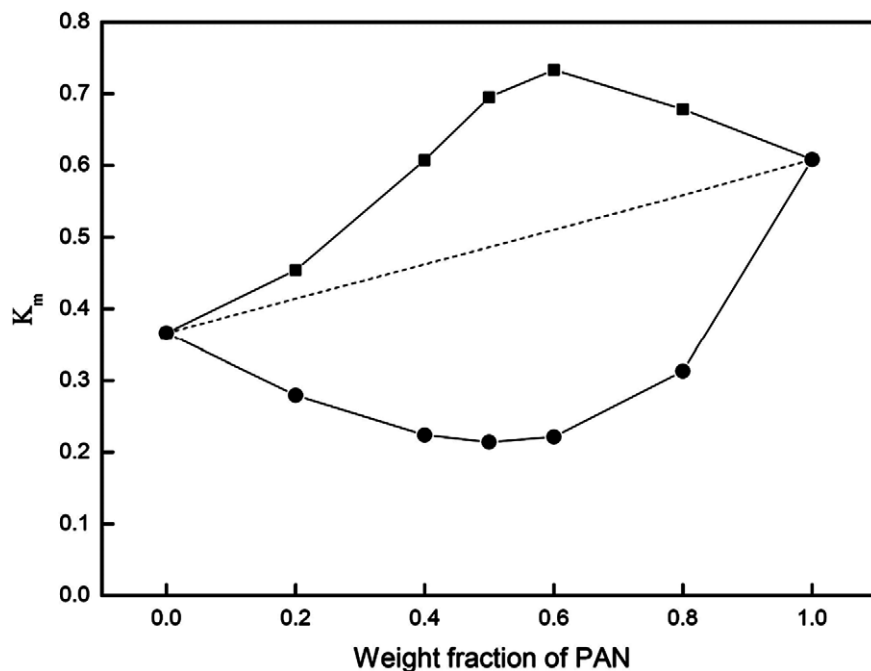
Fig. 8. The dependence of K_H on composition.

Table 1
The interaction parameters Δb values for different blend compositions

Weight fractions of PAN in HPC/PAN blend	Interaction parameter (Δb)
0.2	0.9
0.4	1.28
0.5	0.84
0.6	0.07
0.8	1.66

$$(K_m)_{id} = \frac{\sum_{i=1}^n k_{m_i} [\eta]_i w_i^2}{\sum_{i=1}^n [\eta]_i w_i} \quad (9)$$

The interactions between the constituents in the polyblend were determined with regard to whether $(K_m)_{exp}$ was positively deviated from $(K_m)_{id}$ or not. The association constant could be quantitatively predicted by considering the K_H and $[\eta]$ of the blend solution based on Eqs. (7)

Fig. 9. The dependence of the apparent association constant on the ideal association constant ■ $(K_m)_{exp}$ ● $(K_m)_{id}$.

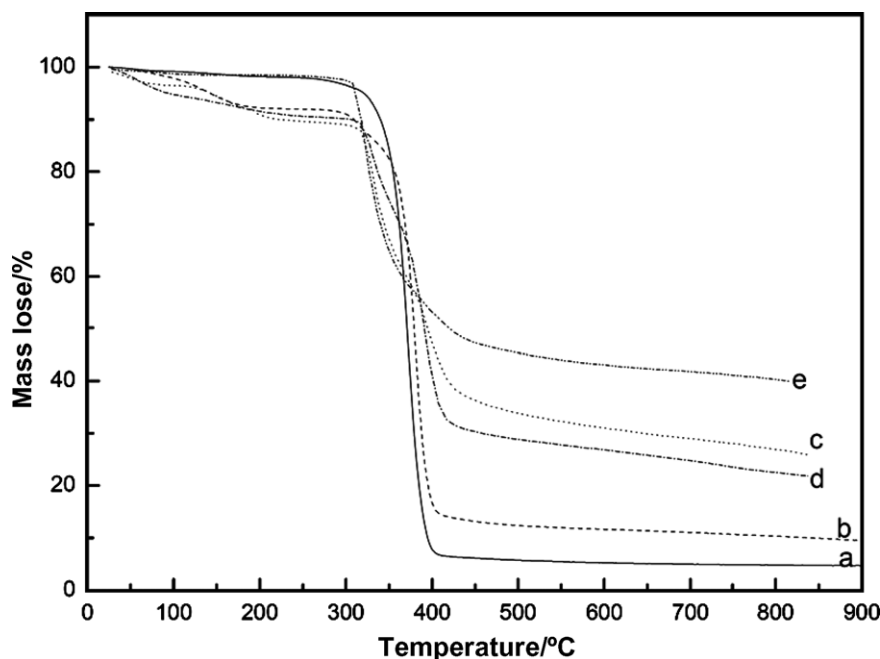


Fig. 10. The dependence of mass loss on temperature for TGA. The curves from a to e represent the different PAN weight fractions, which are as follows (a) 0; (b) 0.2; (c) 0.5; (d) 0.8; (e) 1.

and (8). K_H is Huggins constant, $[\eta]$ is the intrinsic viscosity, which is the value of specific viscosity extrapolated to a concentration of zero and this was obtained from Fig. 8. The observed and calculated values of $(K_m)_{\text{exp}}$ and $(K_m)_{\text{id}}$ were plotted as a function of the amount of PAN as shown in Fig. 9. The values of $(K_m)_{\text{exp}}$ positively deviated from $(K_m)_{\text{id}}$ in a way predicted by the principle cited above. The results supported the compatibility of the two polymers based on the molecular interactions that existed between the polymer blends.

3.2.5. Thermal analysis

TGA provided quantitative information on the process of weight loss. All of the samples were pyrolyzed at the heating rate of 20 °C/min in the temperature range of 25–850 °C. in an N_2 atmosphere. Fig. 10 shows the TGA curves for pure HPC, pure PAN, and different mixtures of the blended samples. The recorded TGA plots for pure HPC and pure PAN showed a one-stage degradation step within the range of 300–450 °C. This was characterized by a weight loss of about 90% for HPC and 50% for PAN, and suggested there is a single degradative process. The blended samples exhibited a three-step degradative process. An initial weight loss of about 5% occurred between 25 and 100 °C. A second weight loss of about 5% occurred between 100 and 200 °C. and the third weight loss of about 50% occurred between 300 and 450 °C. The first weight loss was caused by the loss of water and the second weight loss was caused by the loss of solvent that remained in the samples. The third degradative step was caused by the complete degradation of the macromolecule.

The third degradative step in blended samples demonstrated an intermediate between those of the two pure components. This suggested that the inter-macromolecular hydrogen-bonding caused interactions in the blends.

4. Conclusions

The idealized chemical structure of HPC was valid for calculating the three Hansen Solubility Parameters. The three HSPs were remarkably different between HPC and PAN. We concluded based on our theoretical calculations of the various solubility parameters that this binary blend system was incompatible. But the HPC and PAN blends were found to be compatible in our experimental results. Of the methods we used, the viscosity method was the most sensitive, simple and accurately reflected the compatibility changes in the polyblends.

The interaction parameters, Huggins constants, and association constants that were calculated based on the viscosity were very credible and along with viscosimetry were valuable tools for the characterization of the compatibility of polymeric blends. The same compatibility results were deduced from SEM, POM, TGA and FTIR analysis in this investigation. The compatibility of these blending polymers was partly produced by the hydrogen-bonding interactions between the hydroxyl groups and the nitrile groups during blending. This research increases our knowledge of the compatibility properties that can be measured in these systems and provides the promising possibilities of casting films and spinning hollow fiber membranes from novel polymeric blends.

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