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STUDIES ON MISCIBILITY OF SODIUM CARBOXY METHYL CELLULOSE/ POLY (ETHYLENE GLYCOL) BLENDS

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ABSTRACT

Miscibility studies have been conducted on solutions of blends of Sodium Carboxy Methyl Cellulose (NaCMC) and Poly (ethyleneglycol) (PEG) over an extended range of concentrations in water. The Viscosity , Ultrasonic velocity, density, and refractive index of the blends have been measured for different compositions viz., 0/100, 20/80, 40/60, 50/50, 60/40, 80/20 and 100/0 of NaCMC/PEG blends in water at 30^0 C. The interaction parameters such as ΔB, μ and α calculated from viscosity results using Chee and Sun methods indicate that NaCMC/PEG blend is miscible when NaCMC content is more than 50% in the blend. The obtained conclusions have been confirmed by ultrasonic velocity and refractive index results also. The NaCMC/PEG blend films are prepared by solution casting method and are analyzed by Fourier Transform Infrared Spectroscopy, Differential Scanning Calorimetry, X-ray diffraction and Scanning Electron Microscopic techniques that confirmed the semi miscibility nature of this blend.

INTRODUCTION

Miscibility of polymers plays an important role in the production and study of polymer blends. The properties of polyblends depend upon the formation of homogeneous system at a molecular level. At first sight, polymer blends may be classified into homogeneous or miscible, and heterogeneous or immiscible blends (Utraki, 1989; Olabisi *et al*., 1979). Homogeneous blends are relatively rare because the Gibb's free energy of mixing is positive due to the negligible change in entropy, as a consequence of high molecular weight of polymers and with enthalpy term being positive. The best method of enhancing, the miscibility of polyblends is to introduce specific interactions between the constituent polymers. The potentially useful specific interactions are random dipole, induced dipole, dipole-dipole, hydrogen bonding, acid-base interaction and charge transfer interactions (Liu *et al*., 2005). Mixing of solutions of interacting polymers produces an immediate precipitate, turbidity, or homogeneity, indicating, respectively, stronger, weaker, and no or very small interactions between the polymers (Paladhi and Sing, 1994).

For polymer-polymer miscibility investigations the most useful techniques are electron microscopy (Inoue and Ougizawa, 1989), spectroscopy (Colemann and Printer, 1984), thermal analysis (Hourston *et al*., 1997), and inverse gas chromatography (Mandal *et al*., 1989). These techniques have been very powerful one for such studies. However, most of them are very expensive. An alternative simple, inexpensive, and reliable techniques to analyze polymer miscibility in solution phase are viscometry (Paladhi and Singh, 1994), ultrasonic velocity (Paladhi and Singh, 1994), and refractive index (Basav Raju *et al*., 2006). Sodium Carboxy methyl cellulose, often called cellulose gum or NaCMC. NaCMC [β (1,4)-D-glucopyronose] is a semi synthetic water soluble polymer where CH_2COONa groups are substituted on the glucose units of the cellulose chain through ether linkage, has hydroxyl and carbonyl functional groups as well. It is colorless, odorless, non-toxic and water soluble but insoluble in organic solvents. Due to its anti-tumor and anti bacterial activity, as well as its high hydrophilicity, NaCMC hydrogels have been used in several biomedical applications including drug encapsulation, wound care industry and cosmetics. It is nontoxic, biocompatible, bio degradable, and abundant (Charpentier *et al*., 1997; Ito *et al*., 1986; Wach *et al*., 2001; Kim *et al*., 2006). NaCMC is a widely used component of food system; it may act as a suspending agent, thickener, protective colloid and for control of the crystallization of some

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other component (Elliot *et al*., 1974). Poly (ethylene glycol) (PEG) is a water soluble and hydrophilic polymer. PEG series and their fatty acid esters are widely used in cosmetic and pharmaceutical industries (Elliot *et al*., 1974). They have various characteristics such as water–solubility, non–volatility, physiological inertness, and lubricating properties. We can change the viscosity, moisture, absorption and structure of products according to their different molecular weights. The product with low relative molecular weight (not more than 2000) can be used as wetting agent and thickness modifier for producing cream, emulsion, toothpaste and having cream etc.

A combination of synthetic and natural polymers results in new materials, which have useful properties of synthetic component (good mechanical properties, easy processability, low production and transformation costs) and bio compatibility typical for biopolymers (Cascone *et al*., 2001; Cristallini *et al*., 2001). These blends have already been utilized as biodegradable biomaterials (Azevedo *et al*., 2003), drug delivery systems (Cascone *et al*., 1995), membranes (Yang *et al*., 2004), and materials for agricultural application (Chiellini *et al*., 2001).

As a part of our research work (Naveen kumr *et al*., 2010; Sudharshan Reddy *et al*., 2012) we have studied the miscibility of NaCMC/PEG blend in solution and solid state. We selected these polymers, because they have many pharmaceutical and biomedical applications. NaCMC is a typical polysaccharide and contains large amount of hydroxyl groups in its structure which can act as a proton donor with respective proton-accepting ether groups of PEG and form intermolecular hydrogen bonding. This concept will be of use for us to study this polymer blend miscibility. This has been carried out in the present work by studying the blend solutions through viscosity, ultrasonic velocity and refractive index measurements and also studying the solid blend films through FTIR, DSC, X-RD and SEM techniques.

MATERIALS AND EXPERIMENTAL TECHNIQUES

Sodium Carboxy Methyl cellulose (Mw = 90000) was purchased from E-Merck (India) Limited, Mumbai, India and Poly (Ethylene Glycol) (Mw=6,000) was purchased from Qualigens, Mumbai (India) and were used without further purification. Double distilled and deionized water having almost zero conductivity was used as solvent.

PREPARATION BLEND SOLUTIONS

The 1 wt % of NaCMC and 1 wt % of PEG solutions were prepared by dissolving 1 g of each polymer in 100 mL of distilled water in two separate Stoppard conical flasks. Eight different blend solutions of NaCMC and PEG were prepared by mixing NaCMC with PEG 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.

PREPARATION OF BLEND FILMS

Blend films of NaCMC with PEG were prepared by solution casting method. Required amount of NaCMC was dissolved in distilled water by stirring over a magnetic stirrer (model 1103; Jenway, UK) for 24 h. To this, 20, 40, 50, 60, and 80 wt % (with respect to NaCMC) of PEG were added. Solutions were mixed uniformly and filtered to remove any foreign floating or suspended particles. The respective solution was poured onto 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.

TECHNIQUES

Viscosity and density measurements were made at 30^0C using Ubbelohde suspended level viscometer (with a flow time of 95 sec for distilled water) and specific gravity bottle respectively. The required temperature at 30^0C was maintained within $\pm 0.05^0$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 (NaCMC/PEG) by weight were measured at 30^0C using ultrasonic interferometer. The constant temperature was maintained by circulating water from a thermostat with a thermal stability of $\pm 0.05^0$C through the double walled jacket of ultrasonic experimental cell. The experimental frequency was 2MHz and the velocity measurements were accurate to better than ±0.5%. The refractive indices of blend solutions with different compositions were measured directly with an abbe's refract meter (digital) with thermo stated water circulation system at 30^0C. The accuracy of the refractive index measurement is ±0.02%.

FOURIER TRANSFORMS INFRARED SPECTROSCOPY

Fourier transform infrared (FTIR) spectra of NaCMC, PEG 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 N_2 atmosphere at a scan rate of 21 cm^{-1}.

DIFFERENTIAL SCANNING CALORIMETRY

DSC curves of NaCMC, PEG 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 20^0C/min under N_2 atmosphere at a purge speed of 100ml/min.

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 1^0/min in a 2θ range of 5^0-40^0) at room temperature.

SCANNING ELECTRON MICROSCOPIC ANALYSIS

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 NaCMC and PEG of different compositions in water at

30^0C are shown in the Fig 1. It was well established earlier by many workers (Kuwahara, 1963; Solomon and Ciuta, 1962), that the variation of viscosity Vs concentration of blend composition plots are linear for compatible blends and non-linear for incompatible blends. On this basis in the present study it is noticed that absolute viscosity varied non-linearly with the concentration of blend composition upto 50/50 NaCMC /PEG and linearly beyond this composition. This indicates that the polymer blends of NaCMC/PEG are found to be miscible when NaCMC content is more 50% and immiscible below this critical concentration. From Figure 1, it is also observed that as the concentration of NaCMC increases in the blend composition the absolute viscosity also increases. With the increasing percentage of NaCMC in the NaCMC/PEG blend, PEG can interact with several chains of NaCMC and can form highly cross linked structure, which causes high solution viscosity. Further, with the increase in proportion of NaCMC in the blend along with cross linking effect, the effect of hydrogen bonding between the -OH groups of NaCMC and etheric oxygen atom of PEG also increases. These effects at higher contents of NaCMC of these blends leads to high viscosity of the blends as observed in Figure 1.

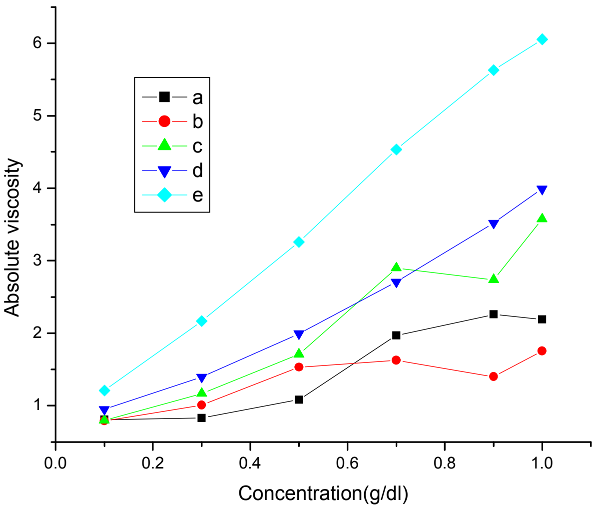


Figure 1. Plots of absolute viscosity Vs concentration for 1% w/v NaCMC/PEG blends of 20/80(a), 40/60(b), 50/50(c), 60/40(d), 80/20(e)

The Huggins plots of reduced viscosity against concentration of different (NaCMC/PEG) blend compositions, pure NaCMC and pure PEG in water at 30^0C and are shown in the Figures 2(a) to 2 (g). From these graphs on extrapolating to zero concentration, the intrinsic viscosities are determined and the values of NaCMC, PEG and (NaCMC/PEG) blends of different compositions (20/80, 40/60, 50/50, 60/40, 80/20) are 2.7346, 0.1373, 0.0008, 0.5690, 0.2056, 0.3576 and 1.2754 dl/g respectively. The intrinsic viscosity values of (NaCMC/PEG) blends for different compositions are in between the values of pure polymers. The reasonable interpretation may be that the attractive interactions are existing between NaCMC and PEG in water. These attractive interactions may lead to decrease the intermolecular excluded volume effect and decrease the hydrodynamic volume. As a consequence, the (NaCMC/PEG) blends expand, which causes an increase of the intrinsic viscosity of (NaCMC/PEG) blends in water.

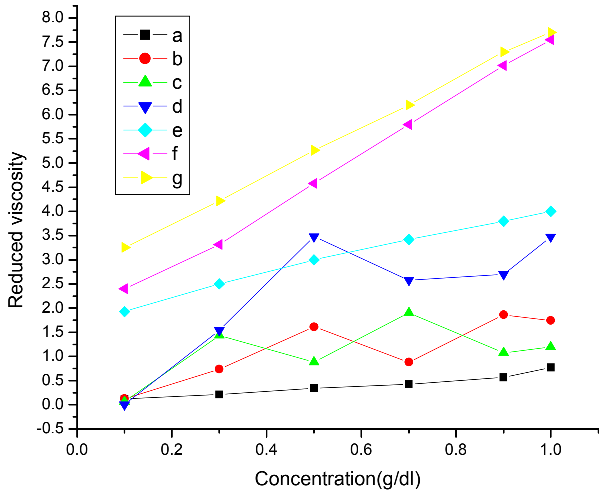


Figure 2. Reduced viscosity Vs Concentration curves for Pure PEG(a), NaCMC/PEG blends 20/80(b), 40/60(c), 50/50(d), 60/40(e), 80/20(f) and Pure NaCMC(g)

From these graphs (Figs. 2(a) to 2 (e)), it is clearly evident that the Huggins curves composed of two regions with varying slopes for the polymer blends upto 50/50 of (NaCMC/PEG) blend composition. The change in the slope of the curve may be attributed to the mutual repulsion of macromolecules in solution which favors the polymer immiscibility. But beyond the (50/50) composition, the curves are linear and this behavior is attributed to the mutual attraction of macromolecules in solution which favors the polymer miscibility. A Similar observation was also made by Basavaraju *et al.* (2007) from their viscosity study in case of Xanthane Gum/PEO blend and reported the blend is semi compatible.

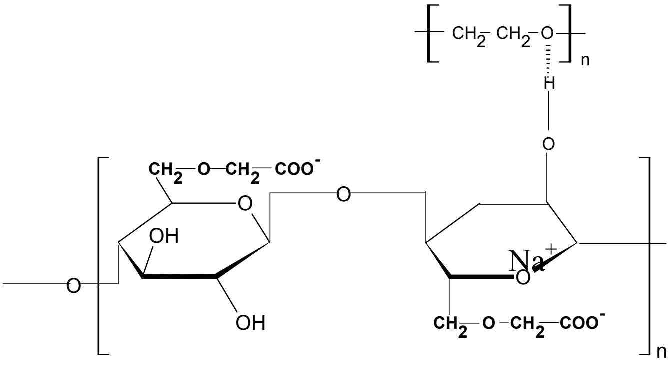
CHEE AND SUN INTERACTION PARAMETERS

In order to quantify the miscibility or immiscibility nature of thr present polymer blend studies, the interaction parameters (B and) suggested by Chee and () suggested by Sun are calculated for the NaCMC/PEG blend systems in water at 30^0C and are presented in the table 1.

Table 1. Chee and Sun's interaction parameters for different compositions of 1% (w/v) NaCMC/PEG blends in water at 30^0C

NaCMC/PEG Blend composition	Chee's Differential Interaction parameters		Sun's miscibility Parameter
	ΔB	μ	$\alpha \times 10^2$
20/80	-0.019	-0.681	-1.467
40/60	-0.097	-0.016	-0.041
50/50	-0.036	-1.249	-0.698
60/40	0.093	1.225	0.306
80/20	0.306	1.298	0.046

It is observed from the Table 1 that the values of ΔB, μ, and α are negative upto 50/50 NaCMC/PEG blend compositions and positive beyond this composition. In general if ΔB, μ, and α are positive for any poly-blend system, it is considered as a miscible blend whereas if these values are negative the poly-blends are considered as immiscible one. Based on this, it is concluded from the present study, as per the values given in the Table 1, (NaCMC/PEG) blend shows miscibility when the NaCMC content is more than 50% and immiscible below this composition. A similar observation was made by Varadarajulu *et al.* (1998) in the case of polyvinyl pyrrolidone/polystyrene blends. At the same time the available –OH groups of NaCMC are also less in number upto 50/50 NaCMC/PEG blend composition, hence the hydrogen bonding possibility is less and this may be the reason for immiscibility of these blends composition. Later as the NaCMC content increases beyond this composition, more no. of NaCMC molecules are responsible for having more no. of '-OH' groups which in turn gives a scope to form more H-bonding with etheric 'O' atoms of PEG molecules, and this leads for miscibility of this blend beyond 50/50 NaCMC/PEG blends. Scheme 1 shows the probable hydrogen bonding interactions between the NaCMC and PEG molecules.



Scheme 1

To further confirm this observation, we measured the ultrasonic velocity (u) and refractive index (n) of the blend under consideration at various compositions at 30^{0}C. The variation of the ultrasonic velocity and refractive index with the blend composition is shown in Figures 3 and 4, respectively. It was already established (Fadnis *et al.*, 2008; Rai *et al.*, 2006) that the variation is linear for miscible blend and nonlinear for immiscible blend. In the current case, on this basis it is noticed that ultrasonic velocity and refractive index varied non-linearly with the concentration of blend composition up to 50/50 NaCMC /PEG and linearly beyond this composition. This indicates that the polymer blends of NaCMC/PEG are found to be miscible when NaCMC content is more 50% and immiscible below this critical concentration due to possible H-bonding interactions taking place between the hydroxyl groups of PEG and the hydroxyl groups of NaCMC as shown in the Scheme 1. A similar observation was made by Demappa *et al.* (2008) in the case of miscibility of Hydroxy Propyl Methyl Cellulose (HPMC)/PVA blends.

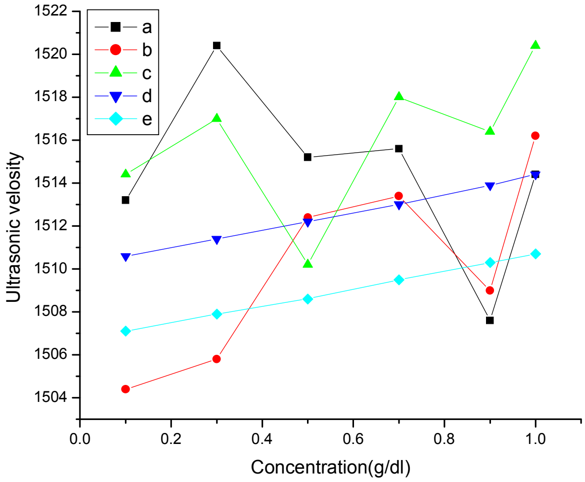


Figure 3. Ultrasonic Velocity Vs Concentration curves for NaCMC/PEG blends of 20/80(a), 40/60(b), 50/50(c), 60/40(d), 80/20(e)

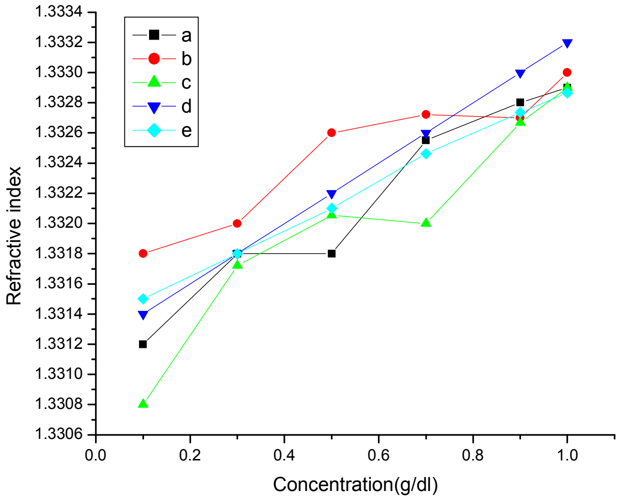


Figure 4. Refractive Index Vs Concentration curves for NaCMC/PEG blends of 20/80(a), 40/60(b), 50/50(c), 60/40(d), 80/20(e)

MISCIBILITY STUDIES OF BLEND FILMS BY CHARACTERISTIC TECHNIQUES

FOURIER TRANSFORM INFRARED SPECTROSCOPY STUDIES

Fourier transform infrared (FTIR) spectroscopy of blend films was carried out in order to detect any peak shift that could be attributed to interactions between the two polymers, such as hydrogen bonding or complexation (Paul *et al.*, 1978). The FTIR spectra of the interpolymer complexes show spectral features similar to those for the homopolymers, but the bands appear at shifted positions. In the present study, the type of H-bonding within NaCMC/PEG polymer blends may be complicated, (Mucha *et al.*, 1999; Kolhe *et al.*, 2003; Jiang and Han, 1998; Fang and Goh, 2000) because there are several groups that can form hydrogen bonds in NaCMC.

Figure 5 shows the FTIR spectra of the pure NaCMC, PEG and blend films in the wavelength ranges 4000-600 cm^{-1}. The characteristic band of PEG was observed at 843 cm^{-1} due to the C-O-C bending. On the other hand, the bands of NaCMC appeared at 3400 cm^{-1} for the hydroxyl groups and at 1613 and 1415 cm^{-1} for the asymmetric -COO- stretching vibration and symmetric -COO- stretching vibration, respectively. The spectrum of the PEG/NaCMC blend films was characterized by the presence of the absorption bands typical of the pure components, with the intensity roughly proportional the blending ratio. The characteristic bands of NaCMC appeared at 1611 and 3400 cm^{-1} were observed in all spectra of the blends. The spectrum of the PEG/NaCMC blend films shows a significant difference in the region of the C-O-C asymmetric stretch at 1100cm^{-1}. The blend films that have undergone the step transition show a broader C-O-C band compared with the pure components that have not. This broadening results in a band shift to lower wave number. The change in the C-O-C band in the spectrum, suggests that hydrogen bonding is the underlying mechanism in the interaction. In addition, hydrogen bonding has the strongest influence on the donor (in our case the -OH of NaCMC) and the absorption maximum of stretching vibration shifts toward lower wave numbers compared to that for the pure NaCMC. It is also noticed that the hydroxyl stretching bands became much broader with increasing NaCMC content. This strongly supports the idea that a hydrogen bonding can form between ether oxygen atoms of PEG and hydroxyl groups of NaCMC (Scheme1). This suggests that the NaCMC/PEG blends show miscibility when NaCMC content is more than 50% (i.e, higher content of NaCMC).

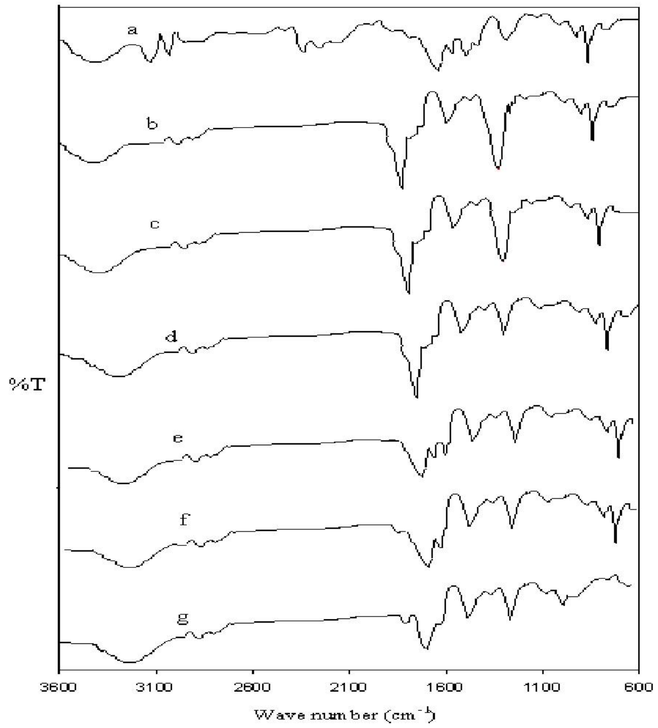


Figure 5. FTIR Spectra of PEG (a), 20/80 NaCMC/PEG (b), 40/60 NaCMC/PEG (c), 50/50 NaCMC/PEG (d), 60/40 NaCMC/PEG (e), 80/20 NaCMC/PEG (f), NaCMC (g)

DIFFERENTIAL SCANNING CALORIMETRY STUDIES

We can estimate the polymer-polymer miscibility by determining the glass transition temperature (T_g) of the blend and its comparison with T_g of component polymers, if one of the components is crystalline then depression in the melting temperature T_m can also be used to study the blend the compatibility. In the present study, DSC was used to estimate the T_m of the blends to investigate the compatibility of NaCMC and PEG blends. Fig. 6 displays the DSC thermo grams of NaCMC, PEG and their blends of different compositions. DSC thermo grams of PEG (g) shows an exothermic peak at 280^0C is related to the melting temperature (T_m), and melting temperature of NaCMC (a) shows at 300^0C. As the PEG content increases in the NaCMC/PEG blends, crystalline melting peak also decreases upto some extent after then it disappears. It indicates the semi compatibility nature of NaCMC/PEG blend.

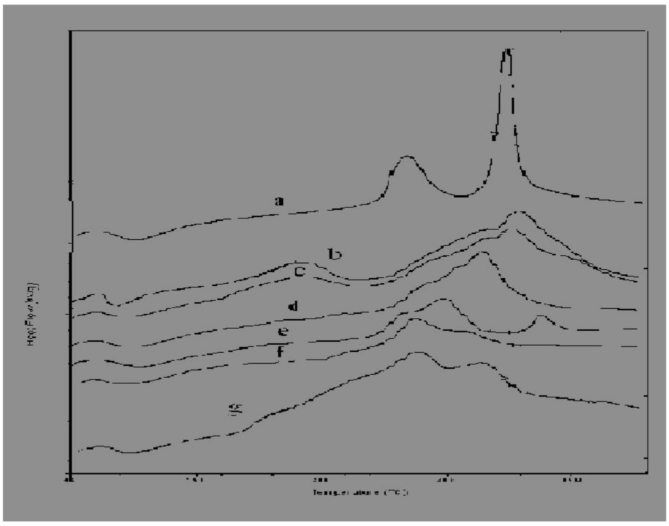


Figure 6. DSC thermograms of NaCMC (a), NaCMC/PEG 80/20(b), NaCMC/PEG 60/40(c), NaCMC/PEG 50/50 (d), NaCMC/PEG 40/60(e), NaCMC/PEG 20/80(f), PEG(g)

X-RAY DIFFRACTION STUDIES

X-Ray Diffraction was performed to investigate the crystalline portion of PEG and how it is influenced by mixing it with NaCMC. The scanning of the samples was carried with an X-ray diffractometer at room temperature. The typical X-RD patterns of PEG, NaCMC and their blend compositions (20/80, 50/50, 80/20 NaCMC/PEG blends) are shown in Fig 7.

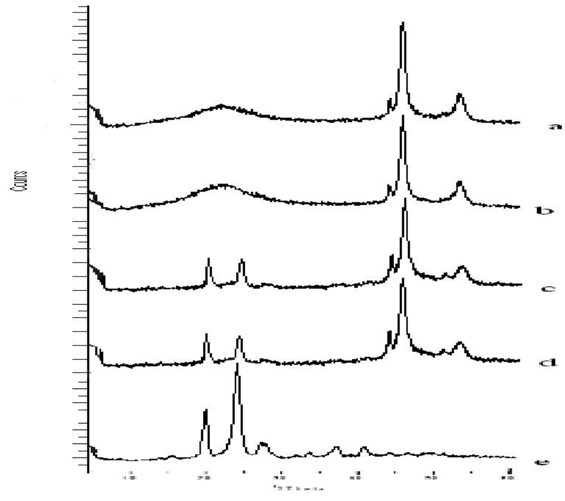


Figure 7. X-ray diffraction patterns of NaCMC (a), NaCMC/PEG (80/20) (b), NaCMC /PEG (50/50) (c), NaCMC/ PEG (20/80)(d), PEG (e)

For the pure NaCMC, there were two peaks around $2\theta=21.5^0$ and 44.6^0. The diffraction model of PEG showed peak around $2\theta=19.7$ and 23.9. Peaks of 20/80 NaCMC/PEG blend film are $2\theta=19.3^0$, 23.6^0 and 44.6, and peaks of 50/50 NaCMC/PEG blend films are $2\theta=19.3^0$, 23.6^0 and 44.6. Here each component has shown its own crystal region in the blend films of 20/80 NaCMC/PEG and 50/50 NaCMC/PEG blend composition. These X-RD patterns have expressed as simple mixed patterns. It indicates that there is no interaction between NaCMC and PEG molecules. This further indicates that these two polymers are not miscible in these two compositions. This means the 20/80 and 50/50 NaCMC/PEG blends show immiscible nature. But the peaks at 2θ values of 80/20

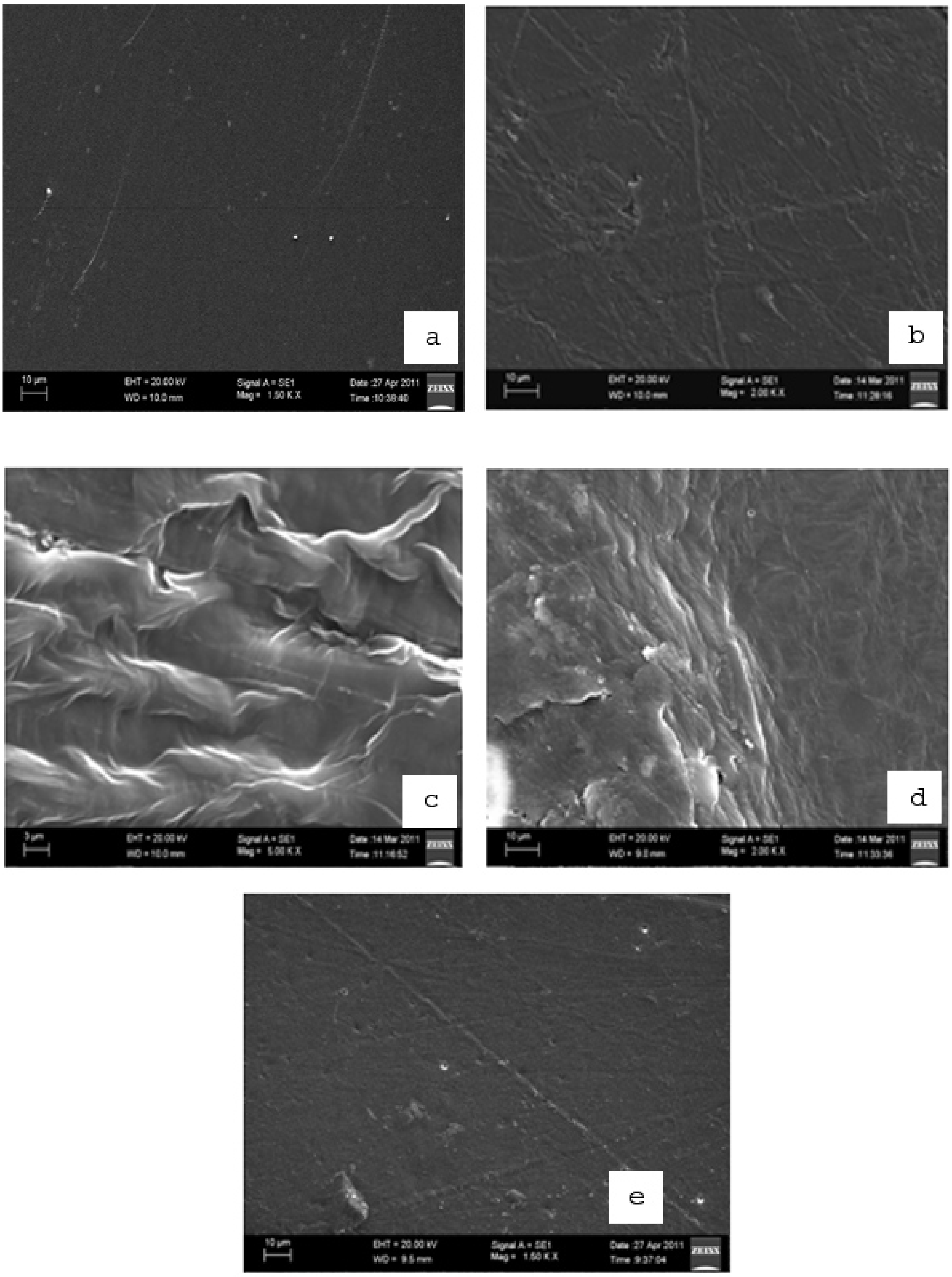


Figure 8. SEM images of NaCMC (a), PEG (b), 20/80 NaCMC/PEG (c), 50/50 NaCMC/PEG (d), 80/20 NaCMC/PEG(e)

NaCMC/PEG blend film are 21.6 and 44.6 and these peaks are similar when compared pure NaCMC peaks. This indicates that strong interactions occurred between NaCMC and PEG molecules in 80/20 NaCMC/PEG blends. This further confirms that this blend is miscible. This evidence further confirm that the NaCMC/PEG blends show miscibility when NaCMC is more than 50% content in the blend and it also confirms the blends are miscible beyond this composition. This result supports the conclusions drawn from other techniques for this blend.

SCANNING ELECTRON MICROSCOPY STUDIES

Figure 8 shows the SEM images of NaCMC (a), PEG (b) and their blends of different compositions 20/80 (c), 50/50 (d) and 80/20 (e). The surface morphology of NaCMC (a) and PEG (b) films were homogeneous. The bright strips presented in the image of pure PEG. With the addition of PEG to NaCMC, the morphologies of the blended films changed dramatically. Phase separation is observed in the 20/80 and 50/50 NaCMC/PEG blend films, but no phase separation is visible in the 80/20 of NaCMC/PEG blend. Hence we conclude that the blend of 20/80 NaCMC/PEG is miscible whereas the NaCMC/PEG blends of compositions 50/50 and 80/20 are immiscible. These results further support the semicompatibile nature of this blend as concluded by the other techniques employed above.

CONCLUSIONS

The miscibility studies of the polymer blends of sodium carboxy methyl cellulose/Poly (ethylene glycol) in water is studied at 30^0C. Viscosity, density, ultrasonic velocity and refractive index measurements are carried out and from the results, it is concluded that the NaCMC/PEG blends are found to be miscible only when the NaCMC content is more than 50% in the blend. It may be due to the hydrogen bonding to some extent between the hydroxyl groups of NaCMC and ether groups of PEG. The films prepared by solution casting method using water as solvent are characterized by FTIR, DSC, X-RD and SEM techniques. These results confirm the semi compatibility nature of the NaCMC/PEG blend concluded by the simple physical techniques.

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