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Miscibility studies on Guargum/Pectin blends

G. Satyamaiah[1], M. Prasad[1], M. Chandrasekhar[1], D.M. Suhasini[2], C. Suryanaraya[3], M.C.S. Subha[2], K. Chowdoji Rao[1]*

[1]Dept. of polymer science and Technology, S.K.University, Anantapuramu, A.P
[2]Deportment of Chemistry S.K.University Anantapuramu A.P
[3]Naval Science & Technological Laboratory (NSTL), Visakhapatnam,A.P
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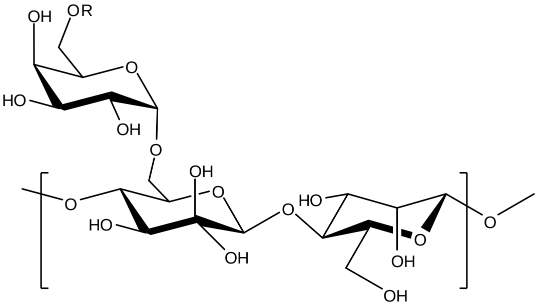
ABSTRACT
The miscibility of blends of Guargum (GG) and Pectin studied over an extended range of concentrations in water using the viscosity, ultrasonic velocity, and refractive index above blend solutions have been measured at 300C. The interaction parameters suggested by Chee (μ) and Sun (α) were computed using the viscosity data to probe the blend miscibility. These values revealed that GG/Pectin blend is immiscible when the Guargum content is more than 40% in the blend. The effects of composition of blends on miscibility have been studied. The miscibility results obtained from viscometric method have been confirmed by the ultrasonic velocity and refractive index studies. The GG/Pectin films were prepared by solution casting method using water as solvent. The blends thus obtained were characterized by FTIR, DSC, X-RD and SEM techniques and the results are also used to probe the miscibility of these blends and confirmed its semicompatibile nature which may be due to the interactions between -OH groups of Guargum and ether groups Pectin.

Keywords: *Guargum, Pectin, Blends*

1. INTRODUCTION

In search of new polymeric materials, either new monomers are polymerized or co-polymerization technique is used to tailor make a new product. An alternative method has been used to blend existing polymers to produce materials with desired properties. An obvious advantage of this approach is that usually requires little or no extra capital expenditure relative to new polymers. The miscibility between the constituents of polymer mixture is an important factor in the development of new materials based on polymeric blends [1]. During the past few years, researchers have paid considerable attention to the study of polymer blending [2-6].By considering blending of natural polymer with synthetic polymer which will be of application orientated to prepare biodegradable and pollution free materials [7-11]. But to have complete biodegradability and pollution free materials now a days researchers are concentrating on the preparation of natural /natural polymer blending[12,13]. In continuation of our research work [14-16] on this topic in this paper we have presented the miscibility of Guargum/Pectin blend system.

Guargum is a natural polymer which is used in food pharmaceuticals and other applications. An industrial grade of Guargum are usually free of cellulose and is available in various grades depending on color (white to grayish), mesh size, viscosity potential and rate of hydration. Guargum (GG) is one of the most abundantly and naturally occurring polysaccharide, consisting of 1,4-B-D-mannose backbone and 1,6-a-D-galactose side chain, and the ratio of galactose/mannose is 1:2 [17] . An industrial polysaccharide used in many fields, especially used as vehicles for oral controlled release purposes[18-21] and specific-colon system[18-20] because of its drug release retarding property and susceptibility . Its applications are innumerable such as in the ceramic industry as a binder, thickener and fixing agent for enamels, porcelain etc. In the pharmaceutical industries, dry Guargum is used as disintegrate and its solution as a binder in compressed tablets manufacture [22]. The structure of Guargum is shown scheme I



Scheme 1: Chemical structure of the guar gum.

Pectin is also a natural polymer and is polysaccharide consisting of linear and branched

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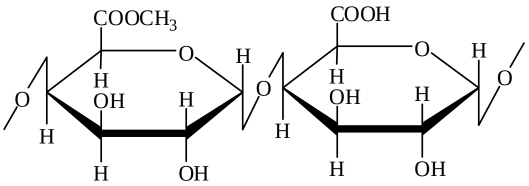
*Corresponding Author:
Email: chowdojirao@gmail.com
Phone: +91 9440533906

chains based on 1,4-α-D-galactosyloric acid residue [23-24]. It is commercially available and extracted from plants. Because of different physico-chemical properties, it can be used in varying fields like pharmaceuticals, coating films, food coating etc. The structure of the Pectin is shown in scheme II.



Scheme II: Chemical Structure of Pectine.

2. EXPERIMENTAL

2.1 Material

Guargum (Mw = 1,40,000) was purchased from s.d. fine chemicals, Mumbai, India and Pectin (Mw=3,60,000) was purchased from Sigma Aldrich, (USA) and were used without further purification for this work. The blends of Guargum /Pectin with 1:1 ratio of these Guargum /Pectin blends of different compositions 0/100, 20/80, 60/40, 50/50, 40/60, 80/20 and 100/0 (w/v) have been prepared by mixing solutions of the polymers in water at 30^0C.The total weight of the components in the solution is always maintained at 1.0 g/dl.

2.2. Preparation of blend films

Thin films of the polymers and their blends were prepared by solution casting method. Separate aqueous solutions of Guargum and Pectin were prepared and a solution of required two homopolymers solutions were added and mixed with constant stirring. The mixtures were stirred for 45 min at room temperature to ensure complete mixing. The total polymer concentration was kept at 1% (w/v). Stock solutions of Guargum and Pectin and their different blend compositions were then casted onto a Teflon-coated clean glass plate and dried using IR lamp in a dust free atmosphere. The dried thin films were peeled off from the glass plate and were found to be transparent. The prepared thin films of pure polymers and blends were characterized using FTIR, DSC, X-RD and SEM methods.

2.3. Techniques

The Viscosity and density measurements were made at 30^0C using Ubbelohde suspended level viscometer with the flow time of 95 sec for distilled water and specific gravity bottle respectively. The temperature was maintained in a thermostat bath, with a thermal stability of $\pm 0.05^0$C.The accuracy in the measurements of viscosity and density are ±0.5% and $\pm 0.0002 g/cm^3$ 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 30^0C using ultrasonic interferometer[25]. 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 using a Digital Abbe's Refractometer, with a thermostated water circulation system at 30^0C. The accuracy of the refractive index measurements are ±0.02%.

2.4. Fourier transformation Infrared Spectroscopy

Fourier transformation Infrared Spectroscopy (FTIR) spectra of Guargum, Pectin and their blend films were taken using Bomen MB-3000 FTIR spectrometer. Blend film were characterized at room temperature from $4000\text{-}400 cm^{-1}$ under an N_2atmsphear at a scan rate of $21 cm^{-1}$.

2.5. Differential scanning Calorimetry

DSC curves of GG, Pectin and their blend films of different composition were recorded using TA instrumentation 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 100 ml/min.

2.6. X-Ray Diffraction

The X-Ray Diffraction patterns of the blend samples were obtained with an Intel diffractmeter (Paris, France) monochromatized Cu Ka radiation scan speed of 1^0/min in a 2θ

2.7. SEM Analysis

Conventional SEM images are formed by scanning a focused beam of electrons across the surface of a specimen and detected the secondary electrons that are ejected by the specimen. A piece of film about 1 X 2 mm size was fixed on the sample holder using adhesive tape and was then coated with a thin layer of gold to improve image resolution. The magnification of the sample is printed on the corresponding SEM photomicrographs. Surface morphology of composites was analyzed on JEOL, Japan (JSM-840A) at an accelerating voltage of 20kV. Surface of the samples were gold coated before analysis.

3. RESULTS AND DISCUSSION

3.1 Viscosity Studies

The absolute viscosity Vs concentration curves for the blends of Guargum and Pectin of different compositions, at 30^0C in water is shown in the Fig 1. It was well established earlier by many workers [26-28] 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, the linear variation of the absolute viscosity with concentration where Guargum composition is 40% observed for the blends. Where Guargum is 40% blend composition whereas the variation is non-linear for blends in which Guargum composition is less than 40%. This indicate the immiscibility nature of the blend when the Guargum content is less than 40% and miscible beyond this composition.

From Fig.1, it is also observed that as the concentration of Guargum increases in the blend composition the absolute viscosity also increases. With the increasing percentage of Guargum in the Guargum/Pectin blend, Pectin react with several chains of Guargum and can form highly cross linked structure, which causes high solution viscosity. Further, with the increase in proportion of Guargum in the blend along with cross linking effect, the effect of hydrogen bonding between the hydroxyl groups of Guargum and Pectin also increases. This effect at higher contents of Guargum of these blends leads to the high viscosity of the blends as Fig. 1.

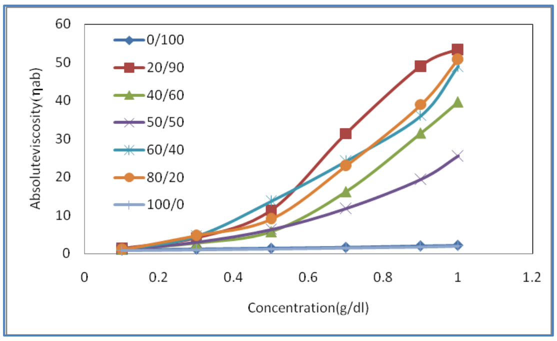


Figure 1: Variation of absolute viscosity with concentration of 1% (w/v) Guargum, Pectin and Guargum/Pectin blends of different compositions in water at 30^oC.

The Huggins plots of reduced viscosity against concentration of different compositions of 1% (w/v) Guargum/Pectin blends, pure Guargum and pure Pectin in water at 30^0C are shown in the Fig.2. From this graph the intrinsic viscosities are determined, on extrapolating to zero concentration and the values of Guargum, Pectin, and different compositions of (Guargum/Pectin) blends (20/80, 40/60, 50/50, 60/40, 80/20) are 1.3268, 0.0568, 0.185, 0.1983, 0.213, 0.155, and 0.124 dl/g respectively. The values indicate that the intrinsic viscosity of Guargum/Pectin blends for different compositions are in between the pure polymer values.

The reasonable interpretation for this may be due to the presence of attractive interactions between Guargum and Pectin molecules in water. These attractive interactions may lead to decrease the intermolecular excluded volume effect and decrease the hydrodynamic volume. As a consequence, the Guargum/Pectin blend coils expand, which causes an increase of the intrinsic viscosity of Guargum/Pectin 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.[29] from their miscibility studies of polymer blends through viscosity measurements.

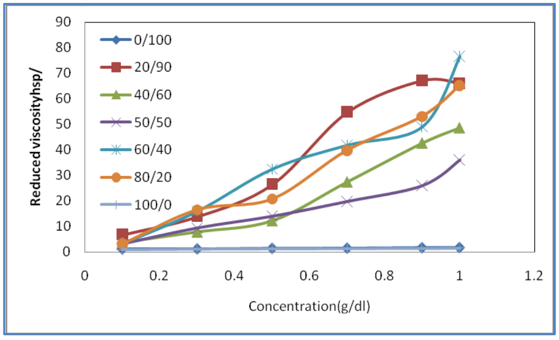


Figure 2: Variation of reduced viscosity with concentration of 1% (w/v) Guargum, Pectin and Guargum/Pectin blends of different compositions in water at 30^oC.

3.2. Chee and Sun interaction parameters

In order to quantify the miscibility of the polymer blends, Chee [30] suggested an interaction parameter μ calculated with the following equation and the values are included in Table.1.

$$\mu = \Delta B/\{[\eta]_2-[\eta]_1\}^2 \qquad (1)$$

Where $[\eta]_1$ and $[\eta]_2$ are the intrinsic viscosities for the pure component solutions. The values of μ are calculated and are also included in Table 1. The polymer blend is miscible if $\mu \geq 0$ and immiscible when $\mu<0$. Recently, Sun *et al.*[31] has also suggested a new formula for the determination of miscibility parameter of polymer blends.

$$\alpha = K_m - \frac{k_1[\eta]_1^2 W_1^2 + k_2[\eta]_2^2 W_2^2 \sqrt[2]{k_1 k_2 [\eta]_1 [\eta]_2 W_1 W_2}}{\{[\eta]_1 W_1 - [\eta]_2 W_2\}^2} \qquad (2)$$

Where k_1, k_2 and K_m are the Huggins constants for individual components 1, 2 and blend respectively. While deriving this equation the long range hydrodynamic interactions were taken into account. Sun *et al.*[31] suggested that a blend will be miscible if $\alpha \geq 0$ and immiscible when $\alpha < 0$. Based on this, the calculated 'α' values for the system has been included in Table 1. From Table 1, it is observed that the computed values of μ and α shows immiscibility nature when the Guargum content is less than 40% and miscible beyond this composition. A similar observation was reported by Rai *et* al [32] from the interaction parameters calculated from the viscosity studies on miscibility of HPMC/Pullulen blends in water.

Table 1: Chee and Sun's interaction parameters for Guargum, Pectin and for different composition of 1% (w/v) GG/Pectin blend in water at 30°C.

Blends (%)	**Chee's Differential Interaction Parameters μ**	**Sun's miscibility Parameter α x 10^3**
20/80	-3.4778	-7.02856
40/60	-1.1656	-10.2298
50/50	0.4057	10.8267
60/40	4.1915	5.62986
80/20	4.9507	4.52816

3.3. Ultrasonic velocity measurements studies

The variations of ultrasonic velocity (u) with concentration of different blend compositions at 30^0C are shown in Figs. 3(a to e) respectively.

From the Figs 3(a to e), it is noticed that Ultrasonic velocity vary nonlinearly upto 40/60 (Guargum/Pectin) blend composition and linearly with polymer blend concentration beyond these blend composition. It has already been established that if the variation is linear it is a miscible one and non-linear for immiscible blend [33-34]. This indicates the Guargum/Pectin blend is immiscible when Guargum content less than 40% and miscible when Guargum content is greater than 40% in the blend.

We can conclude that the ultrasonic velocity results also confirm the conclusions drawn from the viscosity results of these blend solutions as explained above. Hence the Guargum/Pectin blend is to be miscible only when the Guargum content is more 40% in the blend.

3.4. Refractive Index studies

It is evident from figure 4 that the refractive index varies nonlinearly with the concentration upto 40/60 (Guargum/Pectin) blend composition and linearly with polymer blend concentration beyond these blend composition. It has already been established that if the variation is linear it is a miscible one and non-linear for immiscible blend. A similar observation was reported by Thomas *et al* [35] and Chowdoji Rao *et alI* [36] from their ultrasonic refractive index investigations on the semi compatibility of polymer blends. These results support the conclusions drawn from the viscosity studies.

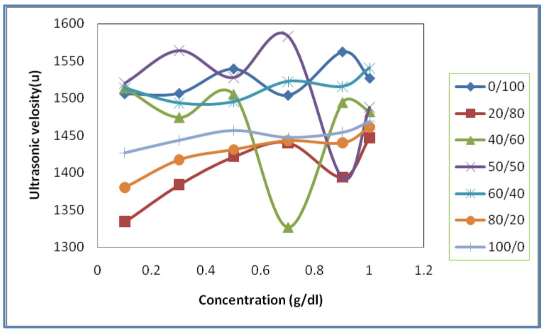


Figure 3: Variation of ultrasonic velocity (u) with concentration of 1% (w/v) Guargum, Pectin and Guargum/Pectin blends of different compositions in water at 30°C.

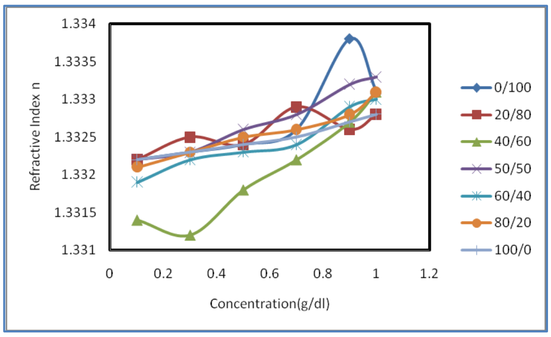


Figure 4: Variation of refractive Index with concentration of 1% (w/v) Guargum, Pectin and Guargum/Pectin blends of different compositions in water at 30°C.

4. Miscibility studies of blend films by characteristic techniques

4.1. Fourier transformation Infrared Spectroscopy

FTIR spectroscopy has been widely used by many researchers to study the formation of blends [37-38] FTIR spectrum provides information regarding intermolecular interaction via analysis of FTIR spectra corresponding to stretching or blending vibrations of particular bands, and the positions at which these peaks appear depends directly on the force constant or band strength. Hydrogen bonding or other secondary interactions between chemical groups on the dissimilar polymers should theoretically cause a shift in peak position of the participating groups. This kind of behavior is exhibited by miscible blends that show extensive phase mixing. Hydrogen bonding interactions usually move the stretching frequencies of participating groups, e.g., O-H towards lower numbers usually with increased intensity and peak broadening. The shift in peak position will depend on the strength of the interaction between the compositions.

The formation of strong hydrogen bonds between Guargum and Pectin 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 3600-3100cm^{-1} produced by stretching of the hydroxyl groups in the spectrum of Guargum can be remarkably distinguished. It can be seen from the spectrums that the peak intensity and peak shape were clearly different and these differences were induced by the different blend ratios. The broad band in the pure Guargum spectrum at 4000-4100 cm^{-1}, with a maximum at 3560 cm^{-1}, was assigned to stretching vibrations of the –OH groups in the range of 3414-3200cm^{-1}. The difference among the curves in Fig. 5, a little broadening or shifting or peaks at 3560-3200cm^{-1} was observed in the 20/80 and 40/60 (Guargum/Pectin) blend compositions when they were compared with that of pure Guargum, which suggested that a relative low amount of interaction was presented between the polymers indicating the immiscibility nature of this pair of polymers upto 40/60 (Guargum /Pectin). But, in the case of 50/50, 60/40, 80/20 and Guargum /Pectin blends there was no shifting in the peak, which it indicates that intermolecular interactions in between Guargum and Pectin. This further confirms that miscibility nature of these blends beyond 40/60 Guargum/Pectin blends. From the Figure 5 it is clear that 20/80, 40/60 Guargum/Pectin blends; there is no change (or) shift in -OH stretching vibrations. In these blends as the Guargum content is lower (40%), no of OH groups available also less to form the hydrogen bonds with ether groups of Pectin molecules. This is also responsible situation to for intermolecular H-bonding. Hence these blends show immiscible nature. By observing (Fig.5 d,e,f and g) spectrum it is clear that the hydroxyl stretching vibrations shifted to a lower wave number with increasing amounts of Guargum which are responsible for formation of more hydrogen bonding interactions between the -OH groups belonging to Guargum and –OH groups of pectin molecules. This is the situation where the H-bond formation due to the intermolecular interactions re possible. This leads to miscibility nature of Guargum/Pectin blends, where the Guargum content is more than 40% in the blend composition. Thus these FTIR results confirm the conclusions from viscosity, ultrasonic velocity and refractive index results.

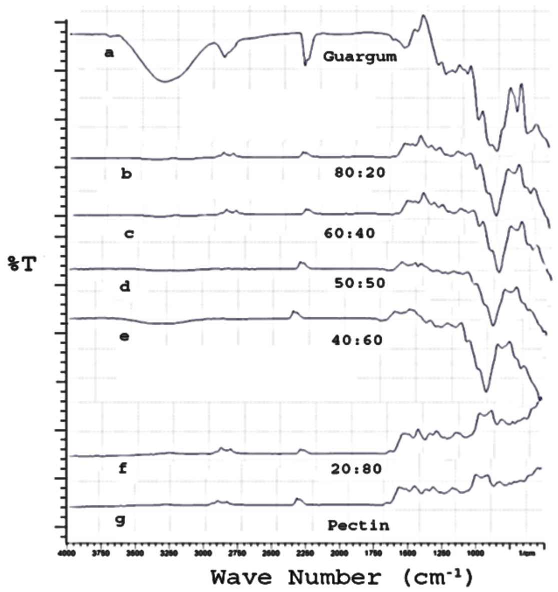


Figure 5: FTIR Spectra of blend membranes with different ratios of Guargum/Pectin (Guargum (a), 80:20(b), 60:40(c), 50:50(d), 40:60(e),20:80(f), pectin(g)).

4.2. Differential Scanning Calorometry Studies:

DSC was carried out to determine the compatibility of the polymer blend system under study. Special care must be taken during DSC measurements since Guargum and Pectin are apt to absorb moisture which strongly effects the DSC measurements. DSC thermograms of Guargum, Pectin and their blend films are displayed in Fig 6. DSC thermograms show that two sharp exothermic peaks at 180.7^0C and 175.9^0c for the pure Guargum, 280^0C and 340^0C for the pure Pectin. Similar two sharp peaks are obtained in the range of 225^0C-320^0C for the blend films of 20/80 and 40/60. This indicates that there are no interactions taking place in between Guargum and Pectin indicating the immiscibility nature of this pair of polymer upto 40/60 (Guargum/Pectin).

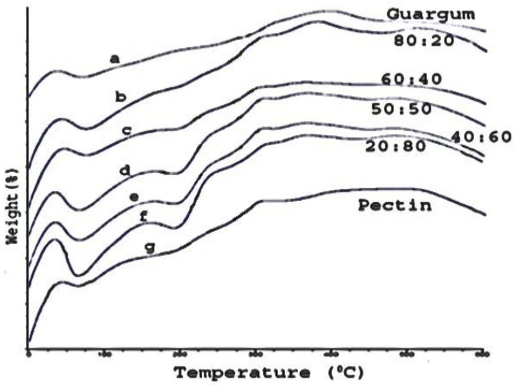


Figure 6. DSC thermograms of Guargum, Pectin and blends of different composition of Guargum/Pectin (Guargum (a), 80/20(b), 60/40(c), 50/50(d), 40/60(e), 20/80(f), Pectin(g)).

But in case of Guargum/Pectin polymer blends beyond 40/60 (Guargum/Pectin) i.e. for 50/50, 60/40 and 80/20 (Guargum/Pectin) blend films show only one broad exothermic peak, so that in these compositions there are intermolecular interactions taking place in between Guargum and Pectin. Hence we can conclude that 20/80, 40/60 blend films are incompatible and 50/50, 60/40, 80/20 Guargum/Pectin blend films are compatible.

4.3. X-Ray Diffraction Studies

The typical X-RD patterns of Guargum and Pectin their blend compositions are shown in Fig 7. For the pure Guargum, there were peaks around $2\theta= 44.6^0$. The diffractograms of Pectin there were three peaks showed at $2\theta=10$, 20, 25^0 and 44.6^0. Peaks of 20/80 (Guargum/Pectin) blend film are $2\theta=20^0$ and 44.8^0, these peaks are similar to Guargum. It indicates that no interactions occurred between Guargum/Pectin molecules in this blend composition. This confirms the immiscibility nature of this blend at this composition. But in case of 50/50, 80/20 (Guargum/Pectin) blend films, the X-RD patterns are $2\theta=10^0$, 20^0 and 44.6^0 which indicate that there are interactions present between Guargum/Pectin molecules in these blend films. Because here each component shows its own crystal region and X-RD patterns are expressed as simple mixed patterns.

It indicates that there are interactions between Guargum and Pectin molecules in the 50/50 and 80/20 (Guargum/Pectin) blends. This confirms the miscibility nature of these blends when Guargum content is more than 40 % in the blend. From these evidences we can further conclude that 20/80 Guargum/Pectin blend is immiscible and 50/50, 80/20 blend compositions are miscible. The X-RD results also support the conclusions drawn from the other techniques carried out for this system.

Figure 8 shows the SEM images of Guargum (a), Pectin (b) and their blends (Guargum/Pectin) of different compositions 20/80 (c), 50/50 (d) and 80/20 (e). The surface morphology of Guargum and Pectin films were homogeneous. The bright strips presented in the image of pure Pectin (b). With the addition of Pectin to Guargum the morphologies of the blended films changed dramatically. Phase separation is not observed in the 50/50 and 80/20 (Guargum/Pectin) blend films, but no phase separation is visible in the case of 20/80 (Guargum/Pectin) blend. Hence we can conclude that the blend of 20/80 (Guargum/Pectin) is immiscible, whereas blends 50/50 and 80/20 (Guargum/Pectin) are miscible in nature. These results also supporting the earlier conclusions stating that (Guargum/Pectin) is a semi compatible blend.

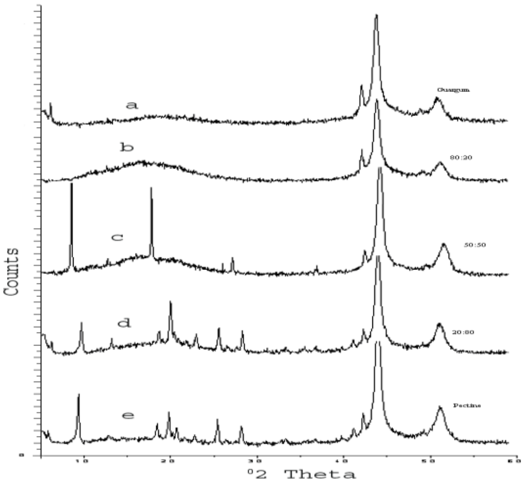


Figure 7: X-RD patterns of polymer blend membranes with different ratio of Guargum/Pectin (Guargum (a), 80/20(b), 50/50(c), 20:80(d), Pectin (e)) 1:1 ratio concentration in water at 30°C.

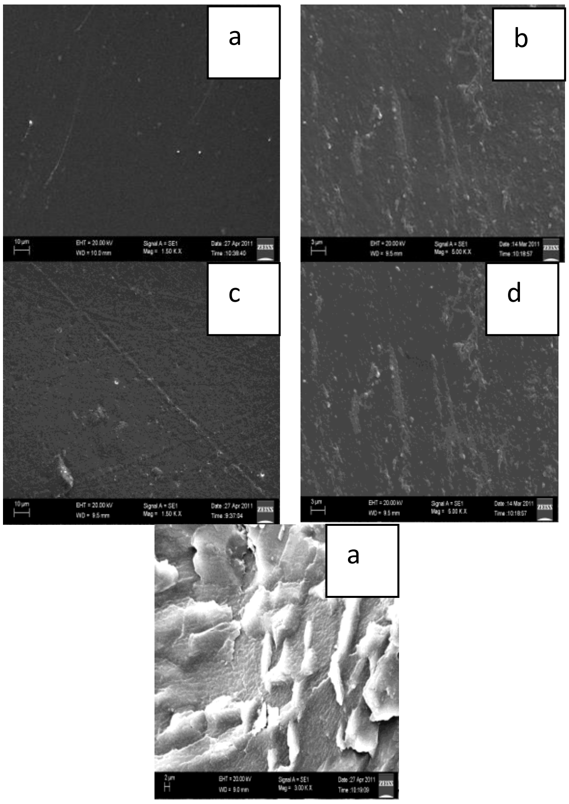


Figure 7: SEM images of Guargum (a), Pectin (b), (20/80) Guargum/Pectin (c), (50/50) Guargum /Pectin (d), (80/20), Guargum /Pectin (e).

5. CONCLUSIONS

The miscibility behavior of Guargum (GG)/Pectin blends in water has been studied by viscometric, ultrasonic velocity and refractive index techniques at 30^0C. Using viscometric data the interaction parameters (μ) suggested by Chee and (α) suggested by Sun are calculated. All these parameters are positive in the blends when the

Guargum content is 40% and negative in the blends when the Guargum content is greater than 40%. It indicates that these blends are immiscible when Guargum content is more than 40% in the blend whereas the blend are miscible when Guargum content is less than 40% in the blends. The miscibility may be due to hydrogen bond formation between hydroxyl groups of Guargum and ether groups of Pectin. This is further confirmed by ultrasonic and refractive index results.

Guargum/Pectin blend films prepared by solution casting using water as solvent are also characterized by FTIR, DSC, X-RD and SEM to support the miscibility data obtained from above simple methods. Analytical technique results support the miscibility window obtained by simple solution techniques.

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