# Indian Journal of Advances in Chemical Science

# Miscibility Studies of Poly(acrylonitrile)/Poly(vinyl chloride) Blends in Dimethylformamide by Viscometry

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# ABSTRACT

Miscibility of poly(acrylonitrile) (PAN) with poly(vinyl chloride) (PVC) was investigated by dilute solution viscometry. Dilute solution viscosity measurements were made on ternary systems, polymer (1)/polymer (2) solvent dimethylformamide for different average molecular weights of PAN and PVC. The intrinsic viscosity and viscometric interaction parameters were determined for binary (solvent/polymer) systems as well as the ternary systems by classical Huggins equation. Degree of miscibility of these polymer systems was estimated on the basis of the three following criteria: (1) The sign of  $\alpha$ , (2) the sign of  $\Delta B$ , and (3) the sign of  $\mu$ . Based on the sign convention involved in these criteria, miscibility was observed in all of the systems under study. The miscibility of all these systems in accordance with interaction between the unlike polymer chains rather than polymer–solvent interactions was investigated depending on molecular weight of polymer sample.

Key words: Viscosity, Hydrophilic polymers, Miscibility.

# I. INTRODUCTION

The production of polymer materials has grown rapidly in the past 50 years. The versatility of plastics, not exceeded by any other class of materials, guarantees that polymers will continue to be very important in the future. However, at present, a distinct change is taking place in polymer research and development. In the pioneering days of plastics, new polymer properties were determined by the choice of suitable new monomers. Today, the commercialization of polymers from new monomers is restricted to few specialties. On the other hand, the number of new polymer blends and alloys based on known polymers is increasing very rapidly. The market for polymer blend-based materials has increased continuously during the past two decades. Polymer blending is a convenient route for the development of new polymeric materials, able to yield materials with property profiles superior to those of the individual components. This method is usually cheaper and less time consuming for the creation of polymeric materials with new properties than the development of new monomers and/or new polymerization routes. An additional advantage of polymer blends is that the properties of the materials can be tailored by combining component polymers and changing the blend composition [1-3].

Three different types of blends can be distinguished: Completely miscible blends, partially miscible blends, and completely immiscible blends. As the gain in mixing entropy is negligible due to the high molecular weight of polymers and the mixing is endothermic in the majority of cases, miscible polymer blends are the exception rather than the rule. Indeed, only few miscible blends have been identified. Most polymer mixtures form immiscible blends, in which the interphase is sharp. Since adhesion between the two polymer components is poor in such blends, they are usually they can be compatibilized.

On the other hand, it has become clear through recent work that a large class of polymers forms miscible blends with appropriate partners through the formation of inter-associated hydrogen bonds; consequently, it is now a well-known strategy to enhance the compatibility of the immiscible blends by the incorporation of interassociated hydrogen bonds and other intermolecular attractions between constituent polymers.

To satisfy the growing needs of new materials with specific properties, such as engineering materials, new polymers have been synthesized [1-3] and chemical modifications in conventional polymers have also been proposed [4-6]. However, the mixture of two or more polymers, forming a polymer blend, continues to be an economical method to obtain new polymeric materials. 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 polymers. In some cases, by synergistic effects, the blend can present better properties than the pure components [7,8].

In recent years, polymer blends have gained considerable attention due to their cost-effectiveness and the relative ease with which new tailormade materials can be produced to meet specific applications. Polymer blends are a matter of active interest in recent years, mainly due to being a versatile way to develop new materials with designed properties that cannot be reached using single polymers. The development of new useful blends, however, is severely limited by the incompatibility of many polymer pairs of interest due to entropic reasons. Specific interactions, such as hydrogen bonding [9], dipole-dipole forces [10],

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**ISSN NO:** 2320-0898 (p); 2320-0928 (e) **DOI:** 10.22607/IJACS.2019.704003

**Received:** 02<sup>nd</sup> February 2019; **Revised:** 26<sup>th</sup> November 2019; **Accepted:** 04<sup>th</sup> December 2019 and charge-transfer complexes [11] for homopolymer mixtures or segment-segment repulsion inside the blends as well as dilution of repulsive interactions by less favorable ones, can produce negative heats of mixing. By this way, the chemical structure of the polymeric components plays an important role in enhancing interactions that can promote miscibility. The role of polymer-polymer interactions in determining the phase behavior and final properties of polymer blend materials is of interest in the prediction of miscibility in polymer blends.

The miscibility of polymer blend is calculated theoretically as well as being estimated experimentally by dynamic mechanical, thermal, electron-microscopic, neutron scattering, spectroscopic, and viscometric techniques [12-19]. Due to its simplicity, viscometry is an attractive and very useful method for studying the miscibility of the polymer blends [19]. Additional advantages (i.e., that no sophisticated equipment is necessary and that the crystalline or the morphological states of the polymer blends do not affect the result) make the viscometry more convincing for characterizing polymer mixtures. Furthermore, the retarded diffusion of polymers in the solid state makes it difficult to attain a condition of true thermodynamic equilibrium and so the behavior of a polymer mixture in solution is the best method of assessing the miscibility [20,21]. In addition, dilute solution viscometry method provides information about both polymerpolymer interactions and polymer-solvent interactions in solution. The effectiveness of dilute solution viscometry method is based on the assumption that mutual interactions of macromolecules in solution have a great influence on the viscosity in the ternary systems. A lot of work has been reported on systems involving neutral or uncharged polymers in organic solvents, complex mixtures of polymers and microemulsions, neutral and water-soluble polymers in water, and mixtures of poly-anions in aqueous solvents [9,10,22,23].

#### 1.2. Theoretical

Miscibility parameters obtained by diluted solution viscometry and derived from the classical Huggins equation are given by

$$\eta_{sp} = [\eta] c + bc^2 \tag{1}$$

Where,  $\eta_{sp}$  is the specific viscosity of a single-solute solution, c is the mass concentration,  $[\eta]$  is the intrinsic viscosity, and b is related to the Huggins coefficient k by

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

Where, b is believed to reflect the binary interactions between polymer segments. Huggins coefficient is a measure of the interpenetration of polymer coils, the extent of which depends on the segment-segment and segment-solvent interactions. This, in turn, affects the inter-molecular hydrodynamic interaction and the molecular dimensions. Krigbaum and Wall derived an expression for the ideal mixture viscosity by redefining  $\eta_{sp}$  in classical Huggins equation as follows:

$$\eta_{\text{spm}} = [\eta]_{\text{m}} C_{\text{m}} + b_{\text{m}} c_{\text{m}}^{2}$$
(3)

Where,  $[\eta]_m$  is the intrinsic viscosity of the mixture,  $C_m$  is the total polymer concentration, and  $b_m$  is the Huggins slope coefficient which characterize the interactions of all polymer species. The slope  $b_m$  for the mixture of polymer A/polymer B is given by

$$b_m = b_A W_A^2 + b_B W_B^2 + 2b_{AB} W_A W_B$$
 (4)

Where,  $W_A$  and  $W_B$  are weight fractions of the polymers,  $b_A$ ,  $b_B$ , and  $b_{AB}$  are the terms characterizing the interactions of the same (A-A, B-B) and different (A-B) polymer molecules, respectively, and the  $b_{AB}$  term, which is a complex parameter including the thermodynamic and hydrodynamic interactions in the system is given by

(5)

$$_{\rm B}=k_{\rm AB}[\eta]_{\rm A}[\eta]_{\rm B}$$

Where,  $k_{AB}$  is the Huggins coefficient between different (A-B) polymer molecules

b<sub>A</sub>

In contrast, Equation (4) yields

$$b_{AB} = [b_m - (b_A W_A^2 + b_B W_B^2)]/2W_A W_B$$
(6)

Combining Equations (5) and (6) give  $k_{AB}$  with all experimental parameters

$$K_{AB} = [b_m - (b_A W_A^2 + b_B W_B^2)]/2W_A W_B$$
(7)

In the presence of only hydrodynamic interactions, theoretical values of  $b_{AB}$  or  $k_{AB}$  are calculated as geometric means of  $b_A$  and  $b_B$  or  $K_A$  and  $k_B$ 

$$b_{ABt} = (b_A b_B)^{1/2}$$
 (8)

$$K_{AB} = (K_A K_B)^{1/2}$$
 (9)

Because experimental  $b_{AB}$  or  $k_{AB}$  values reflect both hydrodynamic and thermodynamic interactions between polymer segments, the difference between experimental and theoretical values  $\Delta k_{AB} = k_{AB} - k_{ABt}$  could give information on the thermodynamic interaction of the polymers. Accordingly,  $\Delta k_{AB} \ge 0$  signifies attractive interactions and miscibility, whereas  $\Delta k_{AB} \le 0$  indicates repulsion and immiscibility.

Sun *et al.* [24] suggested new criteria for polymer-polymer miscibility in terms of thermodynamic parameters,  $\alpha$ , based on the classical Huggins coefficient km in the blends using the equation

$$\begin{split} &K_{m} = \{k_{A}[\eta]^{2}{}_{A}w^{2}{}_{A} + k_{B}[\eta]^{2}W^{2}{}_{B} + 2(k_{A}k_{B})^{1/2}[\eta]_{A}[\eta]_{B} \\ &W_{A}W_{B}\}/\{[\eta]_{A}W_{A} + [\eta]_{B}W_{B}\}^{2} + \alpha \end{split}$$
(10)

According to Sun *et al.* [24] approach for a ternary, polymer A/polymer B/solvent systems, three types of interactions might contribute to the value  $k_m$ . These are long-range hydrodynamic interactions of pairs of single molecules, defined by  $k_{m1}$  which is the same as  $k_m$  given in Equation (10), the formation of double molecules gives by  $k_{m2}$  and intermolecular attraction or repulsion gives by  $k_{m3}$ . Thus, the overall  $k_m$  turns out to be

$$k_m = k_{m1} + k_{m2} + k_{m3}$$
 (11)

The second term  $k_{m2}$  can be neglected at sufficiently low concentration and in the absence of strong specific interactions that would be encouraged aggregation. Reabbreviating  $k_{m3}$  as  $\alpha$  and rearranging Equation (11)  $\alpha$  is given as follows:

$$\alpha = k_{\rm m} - k_{\rm mt} \tag{12}$$

Based on the term  $k_m$ , Sun *et al.* [24] thought that  $\alpha$  can be used to determine the miscibility of polymer blends when  $\alpha < 0$ , immiscible and  $\alpha \ge 0$ , miscible.

Contrast Chee proposed another criterion terming polymer-polymer miscibility described as follows:

$$\Delta B = (b_m - b^*)/(2W_A W_B) \tag{13}$$

$$b^* = W_A b_A + W_B b_B \tag{14}$$

Where,  $b_m$  is the observed interaction parameter for the polymer mixture,  $b_m$ ,  $b_A$ , and  $b_B$  values can be obtained experimentally. Accordingly,  $\Delta b \ge 0$  indicates miscibility and  $\Delta b=0$  indicates phase separation in the blend system. Chee [18] suggested a more effective parameter,  $\mu$  for blend solutions having sufficiently far apart  $[\eta]_A$  and  $[\eta]_B$  values

$$\mu = \{(b_m b_A)/([\eta]_m [\eta]_A\}(b_B b_A)/([\eta]_B [\eta]_A)\}/2([\eta]_B [\eta]_m)$$
(15)

A positive or zero  $\mu$  value indicates miscibility and a negative  $\mu$  value indicates phase separation.

#### **2. EXPERIMENTAL**

#### 2.1. Materials

The poly(acrylonitrile) (PAN) polymer samples studied in this work were obtained from HiMedia Laboratories Pvt. Ltd., Mumbai. The molecular weight characteristics are determined by 150,000. Poly(vinyl chloride) (PVC) polymer samples studied in this work were obtained from Albert Victor, India. Molecular weight characteristics are determined by 48,000. Dimethylformamide (DMF) is supplied by SD Fine Chemicals, Mumbai.

# 2.2. Experimental Method

The viscometric behavior of PAN/PVC/DMF solutions was performed at 30°C using an Ubbelohde-type capillary viscometer. The temperature of the thermostat was controlled with an accuracy of  $\pm 0.1$ °C and the flow times were measured with a digital stopwatch with an accuracy of  $\pm 0.01$ s. The stock solution of each binary and ternary system was made by dissolving polymer samples to a polymer concentration of 0.1 g/dl. Dilutions to yield six lower concentrations were made by adding solvent. For every measurement, the reading was taken after an equilibrium time of 10 min. The average elution times of solutions were determined after several measurements.

#### **3. RESULTS AND DISCUSSION**

Viscometry is a simple and effective technique for monitoring complexation and interaction of polymer blend solutions. In general, if no specific interaction exists in polymer pair, the component polymer coils spatially isolate from each other in dilute solutions, and the reduced viscosity of the polymer pair is close to additive law of the component viscosity. However, positive or negative deviations from the additive law may occur if the specific interactions between the polymer pairs are strong enough, suggesting intermolecular complexation. H-bond complexation is dependent on many factors such as molecular weight and its distribution of the component polymers, the level of the interaction and the solvent used. In these polymer systems, H-bond complexation was investigated depending on the molecular weight of the PAN and PVC. Miscibility is generally known to be enhanced by specific interactions between the polymer pairs (i.e., Van der Waals type interactions between polymers, due to the presence of relatively polar and polarizable groups and hydrogen bonding if there are sufficient hydrogen bonding sites present in the structures of polymer pairs).

Before discussion of data on PAN-PVC mixtures, it is necessary to characterize the viscometric behavior of each polymer separately in aqueous solutions. Using the Huggins equation, intrinsic viscosities, the data, and for the binary systems at 30°C are tabulated in Table 1.

Based on the experimentally observed [ $\eta$ ] for the ternary (polymer 1/polymer 2/DMF) systems, the parameters of the miscibility criteria proposed by Sun *et al.* [24] and Chee [18] were computed using related equations. The values of the miscibility parameters  $\mu$ ,  $\alpha$ , and  $\Delta k_{AB}$  for the pure and blend systems fraction of PAN are obtained from Figures 1 and 2, respectively. According to Figures 1 and 2, viscometric study shows that  $\Delta B$  and  $\Delta k_{AB}$  values are positive for the PAN/PVC blend for all weight fractions. This indicates that these blends are miscible only for these molecular weights under study.

From the measured values of densities ( $\rho$ ), absolute viscosities ( $\eta_{ab}$ ), the calculated values of relative viscosities ( $\eta_r$ ), specific viscosities, and reduced viscosities ( $\eta_{red}$ ) over a wide range concentrations of blend solutions for different compositions 0/100, 20/80, 40/60, 50/50, 60/40, 80/20, and 100/0 of 1% (w/v) for PAN/PC blends in DMF at 30°C are presented in Table 1.

From Figure 1, it is observed that as the concentration of PAN increases in the blend, the absolute viscosity also increases. With

the increasing percentage of PAN in the PAN/PVC blend, PVC can interact with several chains of PAN and can form highly cross-linked structure, which causes high solution viscosity. Further, with increase in proportion of PAN in the blend along with cross-linking effect, the effect of hydrogen bonding between the cyano groups of PAN and  $\alpha$ -hydrogen of PVC also increases. These effects at higher contents of PAN in the blends enhance the cross-linking effect as well as hydrogen bond effect, leading to high viscosity as observed in Table 1 and Figure 1.

The Huggins plots of reduced viscosity against concentration for a different composition of 1% (w/v) PAN/PVC blends in DMF at 30°C are shown in Figure 2. On extrapolating to zero concentration, the intrinsic viscosities are determined (Figure 2) and the values of PAN, PVC, and PAN/PVC blends of different compositions (0/100, 20/80, 40/60, 50/50, 60/40, 80/20, and 100/0) are 0.9756, 2.1914, 2.2982, 2.3972, 2.6055, 3.0236, and 0.7120 dl/g, respectively. These values indicate that the intrinsic viscosity values of PAN/PVC blends for different compositions are in between the pure polymer values. The reasonable interpretation may be that the attractive interactions between PAN and PVC existed in DMF. These attractive interactions may lead to decrease the intermolecular excluded volume effect and decrease the hydrodynamic volume. As a consequence, the PAN/PVC blend coils expand, which causes an increase in the intrinsic viscosity of PAN/PVC blends in DMF compared to intrinsic viscosity values of pure polymers.



**Figure 1:** Variation of absolute viscosity with a concentration of different compositions of poly(acrylonitrile)/poly(vinyl chloride) blends in dimethylformamide at 30°C.



**Figure 2:** Variation of reduced viscosity with a concentration of different compositions of poly(acrylonitrile)/poly(vinyl chloride) blend in dimethylformamide at 30°C.

**Table 1:** The values  $(\eta_{sp})$  of density  $(\rho)$ , absolute viscosity  $(\eta)$ , relative viscosity $(\eta_{rel})$  specific viscosity, and reduced viscosity  $(\eta_{rel}/C)$  for different compositions of 1% (w/v) (PAN/PVC) blend in DMF at 30°C.

Concentration (g/dl)	Density (ρ) (g/cm <sup>3</sup> )	Absolute viscosity (η) Cp	Relative viscosity (η <sub>rel</sub> )	Specific viscosity η <sub>sp</sub>	Reduced viscosity $(\eta_{sp/c}) \times 10^{-2} (dl/g)$
0/100 PAN/PVC					^
0.1	0.9469	0.7822	1.0976	0.0976	0.9756
0.2	0.9471	0.9404	1.3195	0.3195	1.5975
0.4	0.9473	1.0685	1.4993	0.4993	1.2482
0.6	0.9475	1.2193	1.7108	0.7108	1.1847
0.8	0.9479	1.1652	1.6349	0.6349	0.7055
1.0	0.9482	1.5817	2.2193	1.2193	1.2193
20/80 PAN/PVC					
0.1	0.9349	0.8689	1.2191	0.2191	2.1914
0.2	0.9352	1.0029	1.4072	0.4072	2.0358
0.4	0.9359	1.1969	1.6794	0.6794	1.6985
0.6	0.9363	1.5247	2.1393	1.1393	1.8988
0.8	0.9365	1.6292	2.2859	1.2859	1.6074
1.0	0.9371	1.7046	2.3918	1.3918	1.3918
40/60 PAN/PVC					
0.1	0.9351	0.8765	1.2298	0.2298	2.2982
0.2	0.9359	1.0185	1.4291	0.4291	2.1453
0.4	0.9363	1.2272	1.7219	0.7219	1.8047
0.6	0.9367	1.5700	2.2029	1.2029	2.0048
0.8	0.9368	1.6669	2.3388	1.3388	1.6735
1.0	0.9372	1.8239	2.5592	1.5592	1.5592
50/50 PAN/PVC					
0.1	0.9347	0.8835	1.2397	0.2397	2.3972
0.2	0.9351	1.0473	1.4695	0.4695	2.3477
0.4	0.9358	1.2786	1.7940	0.7940	1.9849
0.6	0.9359	1.5984	2.2427	1.2427	2.0712
0.8	0.9362	1.7179	2.4104	1.4104	1.7630
1.0	0.9364	1.8670	2.6196	1.6196	1.6196
60/40 PAN/PVC					
0.1	0.9347	0.8984	1.2606	0.2606	2.6055
0.2	0.9349	1.0768	1.5109	0.5109	2.5545
0.4	0.9351	1.2999	1.8239	0.8239	2.0597
0.6	0.9352	1.6269	2.2827	1.2827	2.1379
0.8	0.9354	1.7684	2.4813	1.4813	1.8516
1.0	0.9355	1.9247	2.7005	1.7005	1.7005
80/20 PAN/PVC					
0.1	0.9348	0.9282	1.3024	0.3024	3.0236
0.2	0.9349	1.1065	1.5526	0.5526	2.7629
0.4	0.9352	1.3520	1.8971	0.8971	2.2426
0.6	0.9354	1.6718	2.3458	1.3458	2.2429
0.8	0.9357	1.8210	2.5551	1.5551	1.9439
1.0	0.9361	1.9705	2.7648	1.7648	1.7648
100/0 PAN/PVC					
0.1	0.9423	0.7634	1.0712	0.0712	0.7120

(*Contd*...)

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#### Table 1: (Continued).

Concentration (g/dl)	Density (ρ) (g/cm <sup>3</sup> )	Absolute viscosity (η) Cp	Relative viscosity (η <sub>rel</sub> )	Specific viscosity η <sub>sp</sub>	Reduced viscosity (η <sub>sp/c</sub> )×10 <sup>-2</sup> (dl/g)
0.2	0.9425	0.9733	1.3656	0.3656	1.8281
0.4	0.9532	1.4765	2.0717	1.0717	2.6792
0.6	0.9545	1.9183	2.6915	1.6915	2.8192
0.8	0.9605	2.3499	3.2972	2.2972	2.8716
1.0	0.9655	2.6996	3.7879	2.7879	2.7879

PAN: Poly(acrylonitrile), PVC: Poly(vinyl chloride), DMF: Dimethylformamide

**Table 2:** Chee and Sun's interaction parameters for different compositions of 1% (w/v) (PAN/PVC) blend in DMF at 30°C.

Blend composition (PAN/PVC)	Chee's differential interaction parameters		Sun's miscibility parameter	
	ΔB	μ	α	
20/80	0.0281	1.5449	0.0048	
40/60	0.3895	2.1366	0.1905	
50/50	0.8402	4.6082	0.3116	
60/40	1.4987	8.2200	0.4514	
80/20	4.3573	2.3898	0.7871	

PAN: Poly(acrylonitrile), PVC: Poly(vinyl chloride),

DMF: Dimethylformamide

From these graphs, it is clearly evident that the Huggins curves are linear in nature for all compositions; this indicates that mutual attraction of macromolecules in solution favors the polymer miscibility. Similar observation was also made by Heiyang *et al.* in case of PVC/PCL blend and reported that the blend is miscible. In the present study from Figure 2, it is concluded that the PAN/PVC blend shows miscibility for all the blend compositions. This may be due to the formation of hydrogen bonding between  $\alpha$ -hydrogen of PVC and cyano group of the PAN as expected. The occurrence of protondonating and proton-accepting groups in these polymers governs the nature of the intermolecular bonds responsible for the formation of associates between macromolecules of these polymers of different nature. This miscibility could be connected with the proton donorproton acceptor interactions between PVC as a proton donor and PAN as proton acceptor.

A similar observation was repeated by Marentetle and Brown [23] from their studies of components of poly(ethylene oxide)/PVC blends.

# 3.1. Chee and Sun Methods

To quantify the miscibility and immiscibility nature of the present polymer blend system, the interaction parameters  $\Delta B$  and  $\mu$  suggested by Chee [18] and " $\alpha$ " suggested by Sun *et al.* [24] calculated using the equations. The values  $\Delta B$ ,  $\mu$ , and  $\alpha$  of PAN/PVC blend systems in DMF at 30°C are presented in Table 2.

In general [25], if  $\Delta B$  and  $\mu$  are positive for any polyblend system, it is a miscible one, whereas if these values are negative the polyblends are considered as immiscible one. It is concluded from the present study based on the values given in Table 2, the values of  $\Delta B$  and  $\mu$  are positive for the systems under study and this indicates that PAN/PVC blend shows miscibility. A similar observation was made by Rajulu *et al.* [22] in the case of poly(vinylpyrrolidone)/poly(styrene) blends.

The values of " $\alpha$ " calculated for the present system are presented in Table 2. According to Sun *et al.* [24], a blend will be miscible if  $\alpha$  is

 $\geq 0$  and immiscible if  $\alpha$  is <0. In the present study, the values of " $\alpha$ " are found to be positive for all PAN/PVC blend compositions, indicating that the blends are miscible in all compositions.

This supports the conclusions drawn regarding the miscibility nature of PAN/PVC blend in DMF at 30°C from the intrinsic viscosity data as explained above.

# 4. CONCLUSION

The present investigation clearly indicates that the interaction in the blends studied by simple measurements based on viscometry and interactions provide valuable information about the miscibilities of the blends.

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