

# Study of Hydrophobic Nature of Fullerene-Based Poly (Methyl Hydro Siloxane) and Polyacrylonitrile Interpenetrating Polymer Network

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## Abstract

In this study, the effect of combining different molecular domains on single platform has been reported that revealed a proper packing and interpenetration of fullerene spheres with the monomeric species. The fabricated IPN system exhibits hydrophobic behavior in nature. An interpenetrating polymer network (IPN) of fullerene-based poly (methyl hydro siloxane) (PMHS) and polyacrylonitrile (PAN) was prepared. The synthesized polymer network was characterized using infrared (IR) spectroscopy, differential scanning calorimetric analysis (DSC), and scanning electron microscopic (SEM) technique. The IPN was analyzed by IR spectroscopy, which depicts presence of fullerene at 500  $\text{cm}^{-1}$  and 1632  $\text{cm}^{-1}$ , presence of PHMS at 1050  $\text{cm}^{-1}$ , 1250  $\text{cm}^{-1}$ , 2225  $\text{cm}^{-1}$ , and 3000  $\text{cm}^{-1}$  and presence of PAN at 3077  $\text{cm}^{-1}$ , 1299  $\text{cm}^{-1}$ , 1408  $\text{cm}^{-1}$  and 2083  $\text{cm}^{-1}$ . Shifting in band positions indicated the interpenetration of the reacting species. DSC endotherm showed crystalline peak ( $T_c$ ) at 117°C, which indicated the crystalline nature of the synthesized IPN. The absence of  $T_g$  peak and clear observable  $T_c$  peak revealed crystalline behavior of polymeric network. The microstructure of the polymer network was observed by SEM technique, which revealed transparent and dual-phase morphology of the IPN surface. The fluorescent emission spectra of polymeric network were recorded on a spectrofluorometer which revealed fluorescent excitation and emission spectra of the IPN. The Emission spectra generated by radiative decay of excitations exhibit a maximal peak at 450 nm, suggesting that the synthesized IPN nanosheets were typically high-intensity blue light emitting materials. The FTIR investigations revealed multiple non-covalent interactions

achieved by polymerization with physical anchoring on the polymeric network surfaces. Such interactions can be recognized as the driving force for the fabrication of hydrophobic flexible silicon-based materials with a self-cleansing action.

## Keywords

Polyacrylonitrile, PMHS, IPN, Polymeric Network

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## 1. Introduction

Interpenetrating polymer network (IPN); an overcome of researcher's hard work has proved to be a new domain of polymer chemistry [1] [2]. An IPN is a network of two or more polymers entangled in such that they cannot be pulled apart from each other. It usually reveals physical and mechanical characteristics of the reacting network species such as stiffer hydrogel to greater swellability [3] [4] to tune enhanced modulus properties. IPN is an equivalence combination of pliancy, modulus and robustness. Millar in [1] put forward idea of polymeric interpenetration, which provided a new branch of chemistry; the IPN chemistry. This is an applicable idea of researchers to form novel IPN system. Many scientists and co-workers provided their best for up gradation of IPN structure.

Idea of IPN was initially put forwarded by Sperling *et al.* Zahao and coworkers reported the synthesis of elastomer from IPN [5], Buist *et al.* and his team [6] fabricated IPN based on polyurethane. Patel *et al.* [7] reported hydrogel formation. Vlad and team members synthesized an IPN with an immiscible component. Lastly, Meet *et al.* contributed towards the innovation of electromagnetic properties of fullerene-based semi-IPN of PAN and polyaniline [8].

Another important nanomaterial towards the polymer research has been discovered. This is Fullerene molecule, which exists as a hollow sphere. Nowadays Buckyballs (C60) [9] [10] have been a subject of intense study. C60 is a truncated icosahedron, resembling football of twenty hexagons and twelve pentagons. Nanotechnology research study reveals a controlled reactivity of fullerene may be achieved by the addition of desired vinyl/reactive groups to their surfaces. This is good to see that Fullerenes are stable, but not totally unreactive.

Besides these structures, silicon polymers also exhibit some unique optical properties such as low-temperature flexibility, thermal and oxidative stability, high resistance to weathering, etc. PMHS [11], a silicon-based polymer shows a promising property of hydrophobicity means resistant to water, hydrophilic molecules get absorbed or dissolved in water, while hydrophobic molecules only dissolve in oil-based substances, along with flexibility in their molecular structure. Its approach may unfurl new promenade in the synthesis of hydrophobic together with the supple silicon-based polymeric network with self-cleansing ability [12]-[16]. Thus as discussed earlier fullerene-based IPN of PAN; PHMS may provide possibility for future explorations. A variety of research works have been

carried out to investigate thermal and morphological properties [17] [18] [19].

In the literature, enormous work has been carried out on fullerene, silicon polymers and vinyl monomers independently but fullerene-based novel vinyl polymers and silicone IPNs are still rare or none [14]. Thus, the present study put light on the formation of these fullerene-based IPNs of PAN, PHMS. The discussed polymeric network possesses enhanced physiochemical and thermal properties. Attempts have been made to provide flexibility and better thermal properties to such IPN [15]. The vinyl polymer provides good interpenetration whereas PMHS is one of the reasons for flexibility and optical characteristics and the fullerene surface is an important factor for toughness, crystalline behavior and conductivity of the IPN [20] [21] [22].

## 2. Experimental Work

### 2.1. Synthesis of Polymer of Fullerene-Based Poly (Methyl Hydro Siloxane) (F-PMHS)

The polymer was prepared by refluxing PHMS, fullerene and benzoyl peroxide (BPO) via in situ polymerization at 70°C for 2.5 hours in the water bath. Precipitation was done by methanol and dried.

### 2.2. Synthesis of IPN

IPN was synthesized via in situ polymerization by systematic variations of concentration benzoyl Peroxide (BPO), fullerene, acrylonitrile and divinyl benzene (DVB) as a cross-linker. The systems were kept over water bath for 2.5 hours at 70°C under an inert atmosphere of nitrogen. These polymer networks were precipitated and dried [23]-[28]. Our experimental results revealed the significant impact of interfacial tension of both droplets and the surrounding medium on the overall wettability of the solid-medium-droplet system. The predicted results were within  $\pm 25\%$  deviation from that of the experimental observation for most of the cases. In contrast, the application of the previously reported theories resulted in a larger deviation (above  $\pm 35\%$ ) for all samples.

### 2.3. Swelling Measurements

Cross-linked density of polymer network was calculated by measurements of solvent absorbency. The swelling data was calculated by soaking sample in different polar and nonpolar solvents such as dimethyl formamide (DMF), dimethyl sulphoxide (DMSO), dioxane, benzene or toluene until an equilibrium weight was achieved (~24 hrs.). Weight measurements were carried out by blotting the samples and immediately weighing them. The percentage swelling was calculated according to the following relationship as shown in **Table 1**.

$$\% \text{ Swelling} = \left( \frac{W_s - W_d}{W_d} \right) \times 100$$

where,  $W_s$  = weight of swollen IPN and  $W_d$  = Weight of dry IPN.

**Table 1.** Polymeric network revealing swelling behavior with various solvents.

Sample used	DMF (%)	Benzene (%)	DMSO (%)	Toluene (%)	IPN obtained (Extracted)
IPN 1	83	41	67	41	21.3
IPN 2	69	35	57	47	20.5
IPN 3	40	46	53	67	51.3
IPN 4	61	71	67	66	45.6
IPN 5	64	43	69	57	30.1
IPN 6	66	50	65	64	24.8
IPN 7	59	41	68	56	20.6
IPN 8	78	65	63	61	31.8

### 3. Characterization of IPN

#### 3.1. Infrared (IR) Spectroscopic Studies

The structural analysis of IPN was studied by IR spectroscopic technique using Vertex 70 (Bruker) instrument with KBr pellet, data acquisition was based on two channel delta-sigma ADCs.

#### 3.2. Differential Scanning Calorimetry (DSC) Analysis

DSC was carried on a V2.2 Dupont calorimeter, equipped with data station. Sample prepared by casting sample IPN into aluminum pans from 5% solution in chloroform. The specimen was subjected to further drying under vacuum at room temperature for one day. The sample size was 15 mg. Experiments were carried out at a heating rate of 10°C/min under nitrogen atmosphere.

#### 3.3. Morphological Analysis by SEM

The polymer network was analyzed using TESCAN MIRA-3 FESEM, at 10 Kv and under 2500× magnification.

#### 3.4. Fluorescence Analysis

The fluorescent emission spectra of polymeric network were recorded on a spectrofluorometer (Fluorolog 3, Model FL 3-22), for fluorescence properties.

#### 3.5. Hydrophobic Characterization

Contact angle measurement for hydrophobic or hydrophilic characterization of the synthesized IPN was carried out with the Dataphysics Instrument mode TBU-100 with distilled water as a droplet at 300C room temperature.

## 4. Results and Discussion

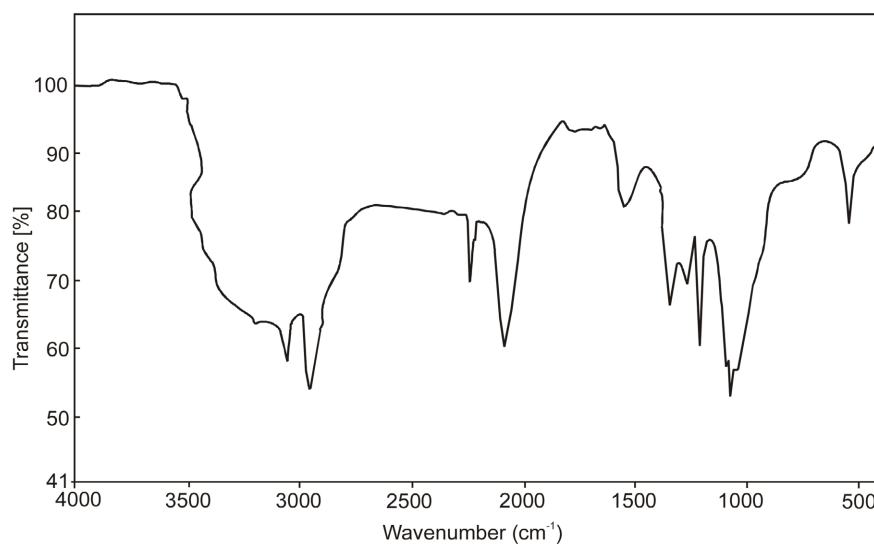
### 4.1. IR Spectroscopy

The detailed vibrational analysis of the synthesized IPN was performed by us,

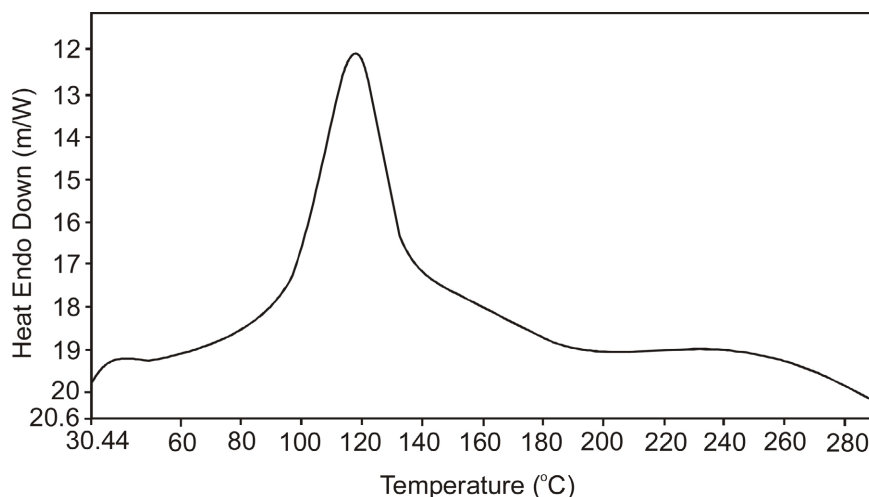
which reveals shifting in band positions of the reacting molecular structures. IR study for pure fullerene shows peaks at  $1430\text{ cm}^{-1}$ ,  $527\text{ cm}^{-1}$  (for C-C vibration mode) and  $1600\text{ cm}^{-1}$  (for  $\text{-C=C-}$  mode) respectively. For pure PAN it shows peak at  $2944\text{ cm}^{-1}$  (Aliphatic CH vibrations),  $1380\text{ cm}^{-1}$  (Aliphatic  $\text{CH}_3$ ),  $1450\text{ cm}^{-1}$  ( $\text{CH}_2$  Vibrations) and  $2224\text{ cm}^{-1}$  (CN) and for pure PMHS it shows the peak at  $1050\text{ cm}^{-1}$  (Si-O),  $1250\text{ cm}^{-1}$  (Si-C),  $2225\text{ cm}^{-1}$  (Si-H) and  $3000\text{ cm}^{-1}$  (C-H) respectively. While **Figure 1** shows IR spectra for the synthesized polymer network, which reveals the presence of fullerene at  $500\text{ cm}^{-1}$  (for caged vibrations),  $1632\text{ cm}^{-1}$  (for C=C mode), for polyacrylonitrile it shows peak at  $3077\text{ cm}^{-1}$  (Aliphatic CH vibrations),  $1299\text{ cm}^{-1}$  (Aliphatic  $\text{CH}_3$ ),  $1408\text{ cm}^{-1}$  ( $\text{CH}_2$  Vibrations) and  $2083\text{ cm}^{-1}$  (CN) and for PMHS it shows the peak at  $1069\text{ cm}^{-1}$  (Si-O),  $1262\text{ cm}^{-1}$  (Si-C),  $2168\text{ cm}^{-1}$  (Si-H) and  $2966\text{ cm}^{-1}$  (C-H) respectively. Thus, as evident from IR analysis that shifting in band positions has taken place, which was an indication of proper interpenetration among the reacting species [10] [11]. The FTIR investigations revealed multiple non-covalent interactions achieved by polymerization with physical anchoring on the polymeric network surfaces. Such interactions can be recognized as the driving force for the fabrication of hydrophobic flexible silicon-based materials with a self-cleansing action. This seems to be very interesting and vowing for the hydrophobic property of PMHS, which can do better intermingling with the swelling behavior of the IPN. It was observed that the fabricated IPN system completely protect the desired material from water and more or less the percolated water if enter the IPN surface, get entangled inside it in the form of hydrogel and lock the movement of water molecule completely at that position.

#### 4.2. DSC Analysis

It is evident from **Figure 2** (DSC endotherm curve) that the synthesized polymeric network exhibits an endotherm peak approximately at  $117^\circ\text{C}$ , which corresponds



**Figure 1.** IR spectra of IPN 5.



**Figure 2.** DSC curve of IPN 5.

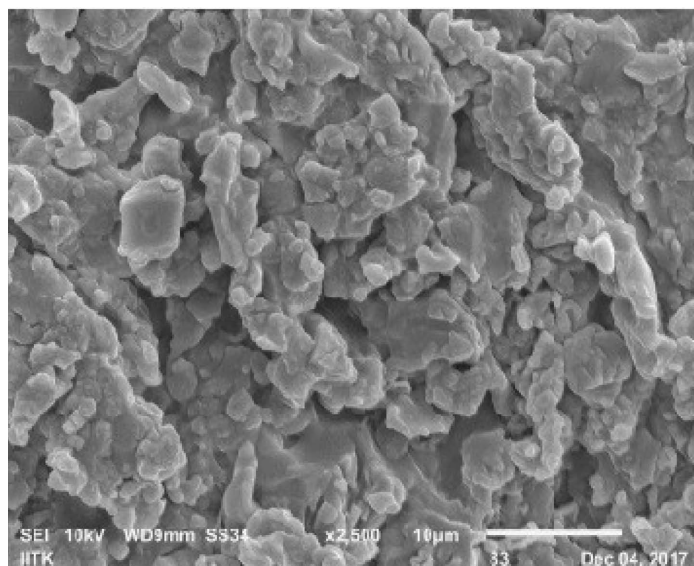
to the crystallization peak ( $T_c$ ) of the polymeric network. Such a peak indicates the crystalline nature of IPN.  $T_g$  value for pure polyacrylonitrile was found to be at 120°C. The synthesized IPN does not show any  $T_g$  peak. The absence of  $T_g$  peak and clear observable  $T_c$  peak indicates crystalline behavior of IPN [20] [21] [22]. This might have been a result of proper packing and interpenetration of fullerene spheres differently at different regions with the reacting species.

### 4.3. SEM Analysis

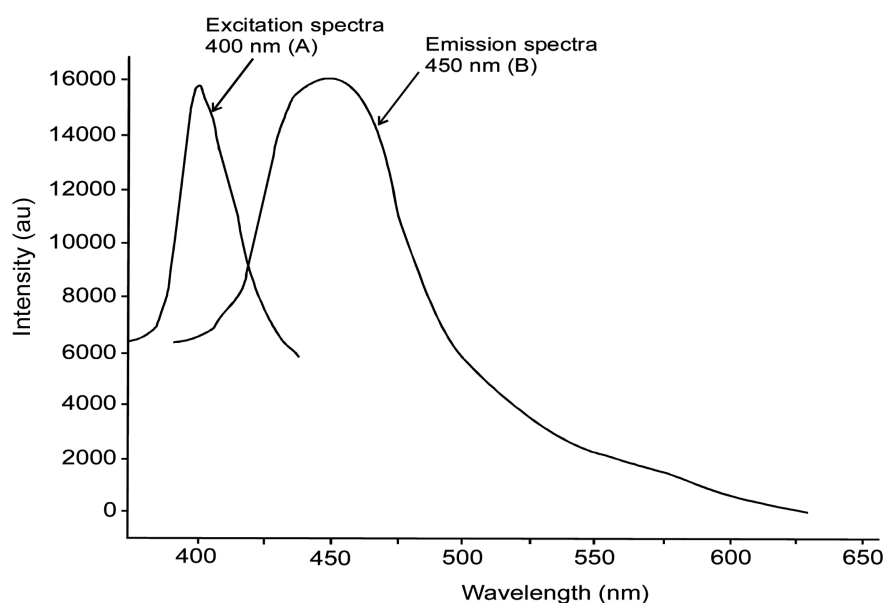
The structure of the fabricated fullerene-based interpenetrating polymer network of PMHS and PAN was detected by means of SEM technique which confides dual phase morphology and transparent nature (Figure 3). The network structure was variegated, which indicates sample heterogeneity of solution, used for the film preparation, which describes the happening of phase separation just after solvent evaporation. This was clearly prominent in the microscopic picture. The surface of IPN composed of fullerene, PMHS and PAN was not smooth. Some projections of different sizes were observable. This indicated that under the top layer, there was a heterogeneous region, depicting clear dual-phase morphology. Thus, the detailed morphological study of the IPN reveals a dual-phase morphology and transparent character.

### 4.4. Fluorescence

Figure 4 reveals fluorescent excitation and emission spectra of IPN 5. The fluorescent excitation and emission spectra of IPN Nano sheets depict that the polymeric network exhibits analogous excitation (A) and emission spectra (B). The emission spectra reveal a broad peak centered at about 400 nm, owing to  $n \rightarrow \pi^*$  electronic transitions found in polymeric networks. Emission spectra generated by radiative decay of excitations exhibit a maximal peak at 450 nm, suggesting that the synthesized IPN Nano sheets were typically high-intensity blue light emitting materials.



**Figure 3.** SEM image of IPN 5.



**Figure 4.** Fluorescent excitation and emission spectra of IPN 5.

#### 4.5. Hydrophobic Characterization

To establish the hydrophobic or hydrophilic nature of synthesized IPN, hydrophobic characterization using contact angle—a measurement through the liquid when the liquid interface meets a solid surface. The contact angle for hydrophilic (love water) is  $0^\circ < \theta < 90^\circ$ , hydrophobic (scared of water) is  $90^\circ < \theta < 150^\circ$  and superhydrophobic is  $\theta > 150^\circ$  [14] [29]. High contact angle shows low wettability and vice versa, was carried out with the Dataphysics Instrument mode TBU-100, 3.34  $\mu\text{L}$  distilled water droplet was used on the sample at  $30^\circ\text{C}$  room temperature, it was observed from the result, **Figure 5** that left contact angle of synthesized IPN was  $103.8^\circ$  and right contact angle was  $104.1^\circ$  which was greater



**Figure 5.** Contact angle measurement of IPN 5.

than 90° and confirm the hydrophobic nature of synthesized IPN for hydrophilic contact angle should be less than 90°.

#### 4.6. Evaluation of Extractable Polymeric Material

The solute component of IPN was removed with the help of the Soxhlet apparatus. The percentage of extractable material was calculated (Table 1) using the following equation.

$$\% \text{ Extractable material} = \left( \frac{W_b - W_a}{W_a} \right) \times 100 \quad (1)$$

where,

$W_b$  = Weight of IPN before extraction and

$W_a$  = Weight of IPN after extraction.

#### 4.7. Swelling Measurement and Calculation

The swelling was calculated in DMF, DMSO, benzene and toluene till a constant mass was achieved (nearly 24 hrs.) (Table 1). The percentage swelling (Tables 1-5) was calculated according to the following relationship.

$$\% \text{ Swelling} = \left( \frac{W_s - W_d}{W_d} \right) \times 100 \quad (2)$$

where,

$W_s$  = weight of swollen IPN and

$W_d$  = Weight of dry IPN.

#### 4.8. Crosslink Density Calculation

IPN sample was taken and its crosslink density was determined (Tables 2-5) by using the swelling data of IPN in DMF by using the Flory-Rehner equation.

$$\frac{1}{M_c} = - \frac{\ln(1 - V_p) + V_p + X_{12} V_p^2}{\rho V_1 (V_p^{1/3} - V_p/2)} \quad (3)$$



**Table 2.** BPO with varying concentrations and effects on IPN.

Sample Taken	BPO Mol in 100 ml	Swelling (%)	$M_c$
IPN 9	$1.0 \times 10^{-2}$	71	149
IPN 10	$2.0 \times 10^{-2}$	63	162
IPN 11	$3.0 \times 10^{-2}$	64	169
IPN 12	$4.0 \times 10^{-2}$	69	179

**Table 3.** Variation of concentration of fullerene and its effect on IPN.

Sample Taken	Fullerene (mol in 100 ml)	Yield	Swelling (%)	$M_c$
IPN 13	$2.1 \times 10^{-3}$	13.11	71	153
IPN 14	$4.2 \times 10^{-3}$	9.91	60	144
IPN 15	$6.3 \times 10^{-3}$	12.12	61	198
IPN 16	$8.4 \times 10^{-3}$	19.53	71	118

**Table 4.** Concentration variation effect of acrylonitrile monomer on IPN.

Sample Taken	Acrylonitrile (Mole in 100 ml)	Yield	Swelling (%)	$M_c$
IPN 17	$1.1 \times 10^{-3}$	11.40	53	144
IPN 18	$2.0 \times 10^{-2}$	16.8	57	189
IPN 19	$3.2 \times 10^{-2}$	17.13	77	147
IPN 20	$4.4 \times 10^{-2}$	17.79	78	189

**Table 5.** Concentration variation of DVB and its effect over IPN.

Sample Used	DVB (mol/100ml)	Yield	Swelling (%)	$M_c$
IPN 21	2.0	14.8	35	135
IPN 22	4.0	11.9	49	144
IPN 23	6.0	19.01	67	190
IPN 24	8.0	21.09	66	199

where,  $M_c$  = average molecular weight of network between crosslinks,  $p$  = density of the network,  $V_1$  = molar volume of solvent, and  $V_p$  = volume fraction of polymer in swollen gel,  $X_{12}$  = polymer solvent interaction parameter, calculated by the expression.

$$X_{12} = B + \frac{V_1 (\delta_p - \delta_s)^2}{RT} \quad (4)$$

where  $\delta_p$  and  $\delta_s$  = solubility parameters of polymer and swelling solvent, respec-

tively, and  $B$  = lattice constant, which was taken as 0.34.

For swelling measurement and crosslink density calculation of IPN seems to be very interesting and vowing for the hydrophobic property of PMHS, which can do better intermingling with the swelling behavior of the IPN. It was clear that the fabricated IPN system completely protects the material from water, more or less if water percolates it enter into the IPN surface, get entangled inside in the form of hydrogel and locks further movement of water molecule completely at that position.

## 5. Importance of Study

An interpenetrating polymer network (IPN) of fullerene-based poly (methyl hydro siloxane) (PMHS) and polyacrylonitrile (PAN) was prepared. PAN is a commercially very important organic polymer used in various applications such as rigid PAN. It is semi-crystalline and in some form, it may be a little brittle form. It is also used in adhesives, resins, printing ink, etc.

These important properties of the polymers such as the flexibility of PMHS and the stiffness and compactness of PAN may together be combined into a new and unique combination. Such an IPN synthesized reflects these combined properties with the increased value of  $T_g$  and TGA values. Such wettability adaptation evolves either due to the formation of a thin film of the surrounding medium over the solid surface, or the molecular reorganization at the solid-liquid interfacial region. Here we developed a theoretical framework and proposed a novel experimental approach to evaluate the solid-medium interfacial tension by implicitly involving the adaptation behavior of a solid surface in the presence of another liquid. We investigated the wettability of three solid surfaces, namely glass, IPN of fullerene-based (poly methyl hydro siloxane) (PMHS) and polyacrylonitrile (PAN) for a wide range of polar and non-polar oil droplets underwater. From our proposed two-liquid approach, we measured the polar and non-polar solid surface tension components.

Thus such polymeric networks may be applicable in vast domains such as defense, medicine, electronics, etc. Fullerene also played a great role in the improvement of the strength of IPN. Fullerene is also responsible for the conductive nature and electromagnetic properties of the synthesized IPN, and provides enhancement of the thermal properties as a result of percolation of these distinct species; fullerene, PAN and PMHS combination. Emission spectra generated by radiative decay of excitations exhibit a maximal peak at 450 nm, suggesting that the synthesized IPN nanosheets were typically high-intensity blue light emitting materials. Thus the polymeric network possesses some very important properties such as durability at higher temperature ranges, and high-intensity blue light emission along its hydrophobic nature. Although such a combination of opposite properties was not easily possible, we have tried to combine such different molecular domains on one platform, which revealed a proper packing and interpenetration of fullerene spheres with the monomeric species.

## 6. Conclusions

The present work has given rise to the formation of a novel flexible and crystalline interpenetrating polymer network of fullerene-based PMHS and PAN. Although such a combination of opposite properties was not easily possible, we have tried to combine such different molecular domains on one platform, which reveals a proper packing and interpenetration of fullerene spheres with the monomeric species. SEM picture depicts clear transparent and dual phase morphology of polymer network. The fluorescent emission spectrum of the polymeric network depicts typical high-intensity blue light emitting properties.

The emission spectra reveal a broad peak centered at about 400 nm, owing to  $n \rightarrow \pi^*$  electronic transitions found in polymeric networks. The wetting of a solid surface by a liquid droplet under a liquid medium not only includes the solid-droplet molecular interactions but also involves the interfacial interaction with the surrounding medium. It has been seen that the fabricated IPN system completely protect the desired material from water and more or less the percolated water if enter the IPN surface, it gets entangled inside in the form of hydrogel and lock the movement of water molecule completely at that position, *i.e.* hydrophobic behavior which was established from the hydrophobic characterization (contact angles  $103.8^\circ$  and  $104.1^\circ$ ). Besides these properties, the overall combination of the semi-IPN with PMHS reveals a perfect formation IPN swelling domain and the hydrophobic nature of the PMHS component.

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## Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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