

Synthesis and swelling properties of NR/PVP/CB semi IPN composites and its insecticide removal efficiency

Manju R Nath¹, Mohanan A^{2*}

¹PG and Research Department of Chemistry, Payyanur College, Payyanur, India

²Department of Chemistry, Nehru Arts and Science College, Kanhangad, India

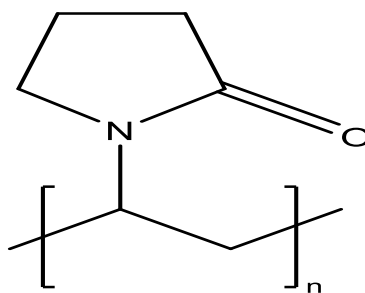
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Abstract: A novel NR/PVP/CB semi IPN composite film of different composition were synthesized by solution casting process using glutaraldehyde (GA) as the crosslinking agent. The semi IPN blends were characterized by Fourier transform infrared spectroscopy (FTIR), thermogravimetry, differential scanning calorimetry. Swelling kinetics of semi IPN blends in toluene were studied by weighing blends swollen with toluene at various time points. Swelling rate decreases linearly with the incorporation of PVP demonstrated the removal efficiency of toxic insecticide bendiocarb due to the development of entangled networks in the semi-IPN synthesized. It is evident that theoretically calculated S_{max} values and the equilibrium swelling ratio obtained experimentally are in good agreement. The swelling exponent (n) values of all system were found to be in between 0.47 and 0.58 signifies that diffusion mechanism follows a Fickian mode of transport. The effect of thermal stability on the in cooperation of PVP and carbon black into the natural rubber latex was studied. A notable bendiocarb removal efficiency is seen from the developed semi IPN composites, along with PVP-induced swelling capacity in water medium that enhances pesticide diffusion.

I. Introduction

Natural rubber, an impressive polymer distinguished by outstanding mechanical, elastic, and flexible properties beneficial to many industrial applications, has been employed in the creation of numerous IPNs [1]. Reports from various studies demonstrate that these polymers have enhanced mechanical characteristics, as well as improved capacities in toxic chemicals such as metals, pesticides and dye adsorption, water absorption and biodegradation when blended with other polymers [2–4].

Polyvinyl pyrrolidone (PVP) is a polymer of N-vinyl pyrrolidone whose structure is depicted in scheme 1 below. PVP is a hydrophilic polymer having repeating units of vinylpyrrolidone. It has various useful properties like film-forming and adhesive properties, solubility in different solvents including water and alcohols, low toxicity etc. In solution, it has good wetting properties and hence readily forms film. This makes it as a good coating material [5–7]. Application of PVP is diverse including pharmaceutical, agricultural, cosmetics, adhesives, textiles etc [8–10].



Scheme 1 Structure of PVP

Since PVP is hydrophilic in nature, blending it with NR can bring the polymer matrix suitable for pesticide removal property [11, 12]. The incorporation of NR and PVP in a semi-IPN system brings potential for the growth of advanced materials. The purpose of this work is to develop an effective semi-IPN composite material composed of NR, hydrophilic PVP and CB as the reinforcing filler for removing toxic chemical bendiocarb from wet soil environment using glutaraldehyde as the crosslinking agent. The investigation presents findings concerning the synthesis, swelling properties and pesticide removal efficiency of semi-IPN system.

Experimental

Materials

Natural rubber latex (NR) polymer with 60% dry rubber content (DRC) was purchased from Indraje Latex Centrifugal factory in Sullia, India. Polyvinyl pyrrolidone was supplied by Merck in Mumbai. Carbon black (CB) was provided by Alfa Aesar, located in Mumbai, India. Crosslinking agent, Glutaraldehyde (GA, 25%) was procured from Shah Scientific (Mumbai, India). Merck Chemicals Pvt. Ltd. in India was the source of the imported sodium periodate and dinitro phenyl hydrazine. Sodium hydroxide, toluene, and ethanol were the reagents supplied by Qualigens Fine Chemicals (Mumbai, India). Bendiocarb, an insecticide, was supplied by Sri Durga Laboratory Equipment Supplies (Mangalore, India). All chemicals were used as such supplied, without any modifications.

Preparation of semi-IPN composites

Originally separate aqueous PVP solutions of 10%, 20%, 30%, and 40% were prepared by stirring at ambient temperature. Then the two polymers, natural rubber latex and PVP were combined in various ratios to create the following composition: NR/PVP (w%/w%): 90/10, 80/20, 70/30, and 60/40. To make homogeneous, the resultant mixture was stirred for 1h. Then, 10 ml 2% GA was added, and is thoroughly agitated again for about half an hour to develop semi-interpenetrating polymer network systems. The solutions were then cast on a petri dish and left at 50°C for 48 hours. The films that developed with 2.5 mm thickness were taken off and stored. Additionally, variance in GA content has been examined by fixing the composition of the polymer. Table 1 compiles the preparation conditions and various sample codes in detail. CB at different formulations (0.002g, 0.02g and 0.2g) were incorporated into the NR_{90%}PVP_{10%}GA_{2%} composition, which is again prepared as before and stirred for 1 h. The solutions were then cast on a petri dish and left there at 50°C for 48 hours. Later the films were separated and to assess the properties of the newly synthesized semi IPN films, various characterization techniques were employed. Removal efficiency of bendicarcab, an insecticide from aqueous solutions were investigated in the present study.

Table 1. Preparation conditions and codes of semi-IPN films

Sl. No.	Code	NR (%)	PVP (%)	GA (%)	CB(g)
1	NR _{100%} GA _{2%}	100	0	2	0
2	NR _{90%} PVP _{10%} GA _{2%}	90	10	2	0
3	NR _{80%} PVP _{20%} GA _{2%}	80	20	2	0
4	NR _{70%} PVP _{30%} GA _{2%}	70	30	2	0
5	NR _{60%} PVP _{40%} GA _{2%}	60	40	2	0
6	NR _{90%} PVP _{10%} GA _{1%}	90	10	1	0
7	NR _{90%} PVP _{10%} GA _{3%}	90	10	3	0
8	NR _{90%} PVP _{10%} GA _{2%} CB _{0.002g}	90	10	2	0.002
9	NR _{90%} PVP _{10%} GA _{2%} CB _{0.02g}	90	10	2	0.02
10	NR _{90%} PVP _{10%} GA _{2%} CB _{0.2g}	90	10	2	0.2

Fourier Transform Infrared Spectroscopy

The Nicolet iS50 FTIR Spectrometer was used to examine the functional group that was present in the samples. IR spectra were obtained in the transmission mode between 4000 cm⁻¹ and 100 cm⁻¹. Samples were put inside the sample container and exposed to the infrared beam. At room temperature, the experiment was run 45 times at a resolution of 4 cm⁻¹.

Thermal characterization

Using a PerkinElmer STA 6000 TG/DTG, the thermal behavior (TGA, DTG) of the produced composite films was measured. The samples were heated in a nitrogen environment from 40.00°C to 730.00°C at a rate of 20.00°C/min. NETZSCH DSC 204F1 Phoenix was used for DSC analysis. The samples were heated to -80°C/10.0(K/min)/500°C in a nitrogen atmosphere.

Swelling measurements

Square shaped samples were used for swelling studies. After being placed in test bottles containing toluene as the solvent, samples with a thickness of 2.5 mm were allowed to reach room temperature. At regular intervals, samples were taken out of the test bottle, and moist surfaces were rapidly dried with filter paper. An electronic balance was used to quickly record the specimens' weight. When the samples were put back into the test container, the swelling process continued until the point of equilibrium swelling was attained. The percentage of swelling (S%) was calculated using the following calculation [13].

$$\text{Swelling percentage (S\%)} = \left[\frac{W_t - W_0}{W_0} \right] \times 100 \quad (1)$$

where W_t is the weight of the swollen sample at time t and W_0 is the weight of the dry sample.

The percentage of a swollen sample that can hold the solvent molecule at equilibrium is called the equilibrium solvent content, or % ESC. The following equation is used to calculate it [14].

$$\text{Equilibrium solvent content percentage(ESC\%)} = \left[\frac{W_e - W_0}{W_e} \right] \times 100 \quad (2)$$

where W_e is the weight of the swollen sample at equilibrium and W_0 is the weight of the dry sample.

Removal efficiency of Bendiocarb pesticide

62.5 mg of bendiocarb were dissolved in 5 ml of ethanol, and the mixture was then topped off to 100 ml with distilled water to create a 0.003M stock solution. Bendiocarb was synthesized at various concentrations by hydrolysing it in an alkaline medium (NaOH) after the stock solution was diluted. According to the reference [15], bendiocarb solution in its coloured form is created for the current experiment. One millilitre of 0.03M NaIO₄ and 1.2 millilitre of 0.001M DNP solution were added to a series of ten millilitre calibrated flasks. After that, 5-20.0 µg/ml hydrolysed bendiocarb solution was added followed by 2.5 millilitre 0.2 M NaOH solution. Then, the appropriate amount was diluted using distilled water. The maximum red colour that appeared persisted for as long as 48 hours. The absorbance was measured at 515 nm using a UV-Visible spectrophotometer in comparison to a blank reagent that was prepared identically but without bendiocarb. For this experiment, a 17ppm solution of bendiocarb was employed and 0.05g of IPN films were soaked in 10ml of bendiocarb solution at room temperature for 48 hours. Following the removal of the films holding the bendiocarb, the amount of bendiocarb consumed by each sample was determined by spectrophotometrically measuring the concentration of the remaining solution at 515 nm. The amount of pesticide eliminated by the IPN films can be computed using the beginning and final absorbance values[16].

$$\text{Amount of bendiocarb loaded (L)} = \frac{(C_0 - C)V}{M} \quad (3)$$

where C_0 is the concentration of bendiocarb in solution prior to loading (mg/L), C is the concentration of bendiocarb in solution after loading (mg/L), V is the volume of solution treated (L), and M is the mass of the dried sample (mg).

$$\text{Bendiocarb removal (\%)} = \left[\frac{C_0 - C}{C_0} \right] \times 100 \quad (4)$$

where the initial and final bendiocarb concentrations are denoted by the letters C_0 and C (mg/L).

II. Results and Discussions

FTIR studies

Figure 1 displays the FTIR spectra of three different polymer films such as; (a) GA vulcanized NR film (b) NR/PVP semi IPN films and (c) NR/PVP/CB semi IPN composite films. The peak corresponding to the carbonyl stretching of aldehyde group is not observed in figure 1a indicating that GA has undergone a complete vulcanization process with NR latex molecules [17]. The peak at 3469cm⁻¹, shows the N-H stretching vibrations of secondary amines, is in good agreement with the above results. Moreover, the C-H stretching of CH₂, non-conjugated C=C stretching, C-H bending of CH₃, and C-N stretching of aliphatic amines all exhibit peaks in the spectra of GA vulcanized NR latex at 2929 cm⁻¹, 1634 cm⁻¹, 1384 cm⁻¹, and 1024 cm⁻¹, respectively. From the figure 1b and c, the peak at 1654 cm⁻¹ represents the carbonyl stretching vibration present in the pyrrolidone ring [18]. Peak at 2957 cm⁻¹ is ascribed to the asymmetric stretching of pyrrolidone ring CH₂ group [19]. Also, peak at 2915 cm⁻¹ and 2856 cm⁻¹ indicates the symmetric stretching of chain CH₂ and ring CH₂ group respectively [20]. The characteristic peak at 1444 cm⁻¹ as well as 1374 cm⁻¹ are ascribed to the C-H bending of CH₂ and CH₃ groups. In addition, 1286 cm⁻¹ peak indicates C-N bending vibrations of pyrrolidone ring [20]. A broad band ranging 3317-3475cm⁻¹ is due to the hydrogen bonding occurring in the carbonyl group of PVP component. This is for the reason that PVP molecule can form hydrogen bond with two water molecules because of the existence of two lone pair of electrons on the C=O group present in the pyrrolidone ring [21]. Therefore, FTIR analysis offers good indication for the existence of both NR and PVP in the prepared semi IPN films.

IR frequency	Functional Group
3317-3475cm ⁻¹	H-bonding by C=O group of PVP
2957 cm ⁻¹	asym. Str. of pyrrolidone ring CH ₂ group
2915 cm ⁻¹ & 2856 cm ⁻¹	symm. Str. of chain CH ₂ and ring CH ₂ group
1654 cm ⁻¹	Str. vibr. of C=O group in the pyrrolidone ring
1286 cm ⁻¹	C-N bend. vibr. of pyrrolidone ring
1444 cm ⁻¹ & 1373 cm ⁻¹	C-H bend. of CH ₂ and CH ₃

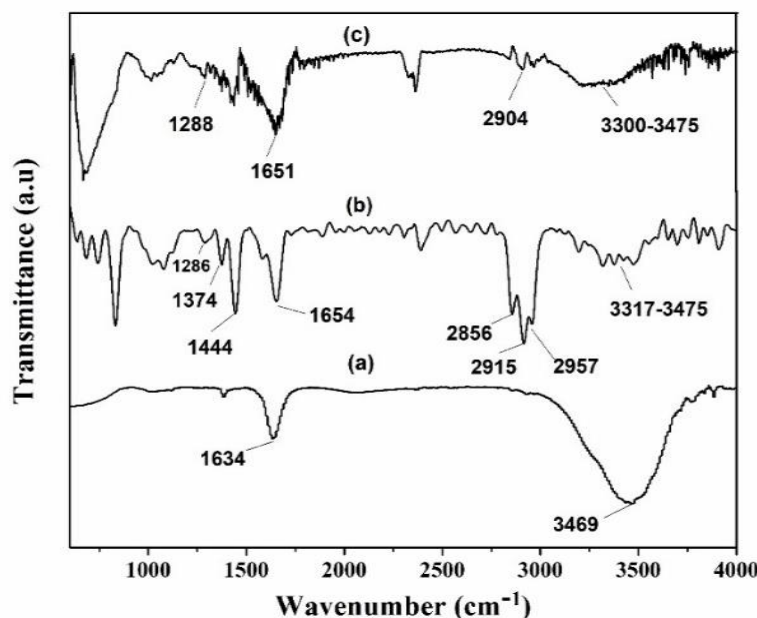


Figure 1. FTIR spectrum of (a) NR (b) NR/PVP semi IPN films (c) NR/PVP/CB semi IPN composite films

Thermal studies

a) Thermogravimetric Analysis

Thermal stability as well as the decomposition pattern of polymeric materials could be studied using thermogravimetric analysis. Both TGA and DTG plots for GA vulcanized NR, NR/PVP semi IPN as well as NR/PVP/CB semi IPN composites are represented in figure 2a and b. From the TG curve it can be seen that an apparent weight reduction is seen after 280°C in the case of pure NR films. But semi IPN film shows a slight weight loss between 100 and 170°C [22]. This can be the moisture content adsorbed on the surface of the films due to the hygroscopic nature of PVP. This observation is reflected in the DTG curve of semi IPN at 134.15°C. Major decomposition has transpired between 300°C and 450°C, resulting in an approximate 85% weight loss. This phenomenon is characterized by a distinct DTG peak observed at 377.28°C. Also, a small peak at 424.21°C is observed in semi IPN curve, which confirm the presence of PVP in the prepared sample. This is different from the DTG peak of pure NR films at 394.96°C indicating a decreased thermal stability of NR latex with PVP molecule. But addition of CB has improved the thermal stability compared to pure NR/PVP semi IPN system. It is clear from the sharp DTG peak at 385°C obtained for the composite film. This indicates a well dispersion of CB into the NR/PVP interpenetrating network system, thereby improving its thermal stability.

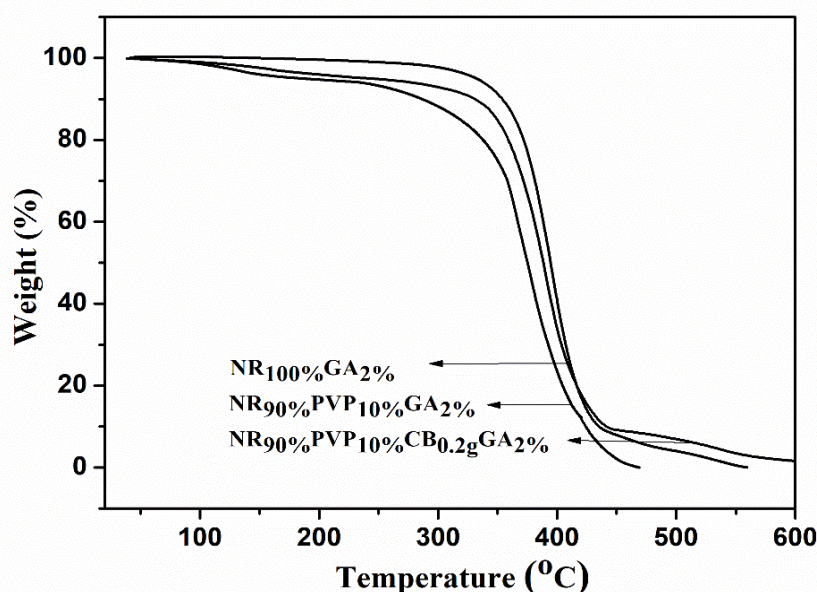


Figure 2a TGA curves of NR, NR/PVP semi IPN and NR/PVP/CB semi IPN composites

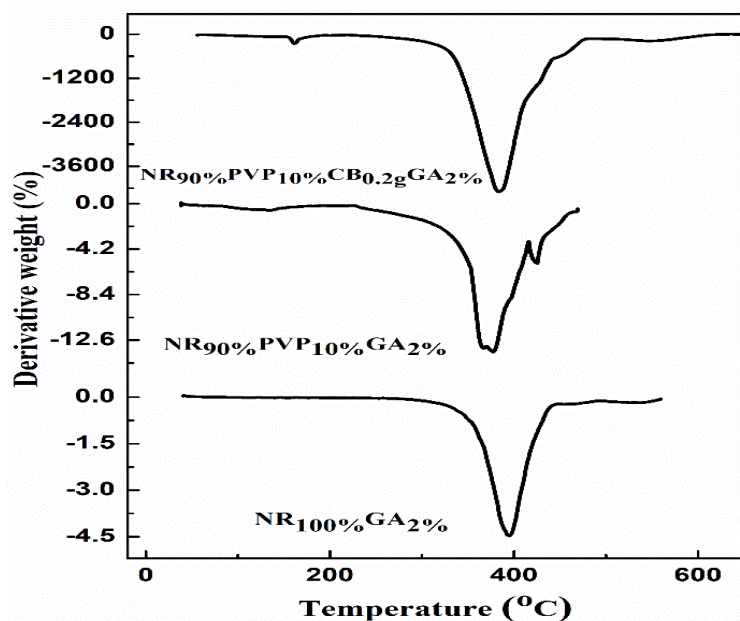


Figure 2b DTG curves of NR, NR/PVP semi IPN and NR/PVP/CB semi IPN composite

b) Differential scanning calorimetry

Figure 3 depicts the DSC thermogram of NR, NR/PVP semi IPN and NR/PVP/CB semi IPN composite. According to DSC analysis of semi IPN film, it is inferred that both polymers are forming a thermodynamically compatible one phase network system showing only one T_g value. The T_g value of glutaraldehyde vulcanized NR is -62.0° C. Though semi IPN exhibits a lower T_g value at -62.9° C, its composites show a higher T_g value at -59.9° C. This happens because of the strong interaction occurring among the active functional group present on the polymeric chains as well as on the CB particles [23].

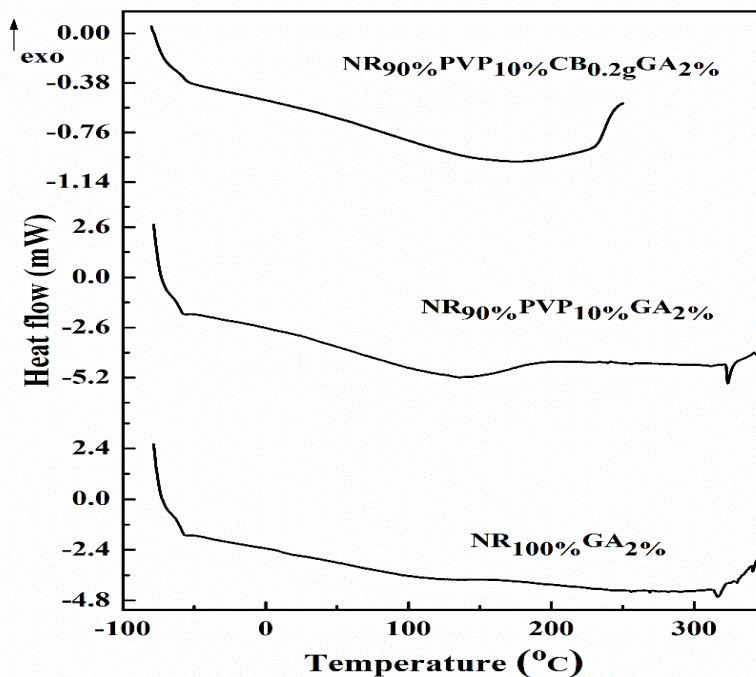
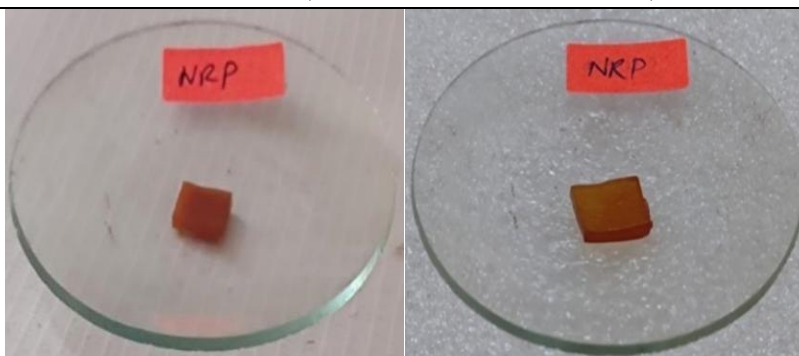


Figure 3 DSC Thermograms of NR, NR/PVP semi IPN films and NR/PVP/CB semi IPN composite films

Swelling studies

Toluene was used as the solvent at room temperature to examine the liquid sorption behaviour of each sample. Swelling studies of NR, NR/PVP semi IPN and NR/PVP/CB semi IPN composites are carried out. The results obtained are summarized in Table 2. The images of dry and swollen NR/PVP semi-IPNs are displayed in Scheme 2.



Dry sample

Swollen sample

Scheme 2 Images of dry and swollen NR/PVP semi-IPN samples

Equilibrium swelling

a) Polymer composition

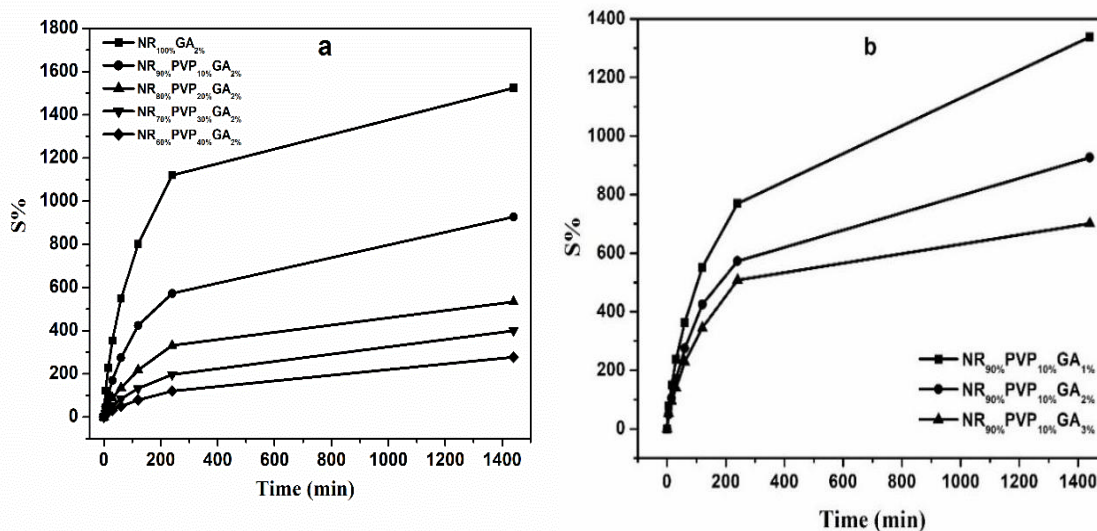
Table 2 represents the swelling parameters of all samples studied in toluene as solvent medium. It is seen that pure NR film swells to its maximum and that as PVP concentration increased, the percentage of swelling dropped. It is clear from figure 4a that the S% of NR_{90%}PVP_{10%}GA_{2%} film declines about 39% from NR_{100%}GA_{2%} film. This suggests that NR and PVP have formed strong interpenetrating networks that are stable, developing a tortuosity path for the entry of solvent molecules. Further addition of PVP has again declined the swelling capacity of films. The least swollen NR_{60%}PVP_{40%}GA_{2%} semi-IPN contains the maximum PVP of 40%. It demonstrates how additional PVP molecules form a robust entangled network system, which raises solvent resistivity. Additionally, PVP being hydrophilic in nature will repel the non-polar solvent molecules. The same trend is observed from ESC% and S_{max} results.

b) GA concentration

Choosing NR_{90%}PVP_{10%}GA_{2%} film as the base composition, GA concentration at 1%, 3% and CB loadings at 0.002g, 0.02g and 0.2g has been examined. When the glutaraldehyde concentration in the IPN system increases from 1 to 3%, a declining trend for S%, ESC%, and S_{max} was observed (figure 4b). Swelling reflects the amount of crosslinking of a network structure[24]. An increased cross-linked network results with increasing the GA concentration. As a result, there is only less room available for the permeation of the penetrant inside the polymer medium. This is the reason for the decreasing behavior of S%, maximum equilibrium swelling and equilibrium solvent content with increasing GA concentration. With 1% GA, a more swollen behavior is observed due to the lack of effective crosslinking. This highlights that while GA function as a cross-linking agent, its effectiveness depends upon its concentration.

c) CB loadings

The impact of carbon black on NR/PVP semi-IPN films' ability to swell is signified in figure 4c. A decrease in swelling behavior is seen with increase in CB content from 0.002 to 0.2g in NR_{90%}PVP_{10%}GA_{2%} film. This is due to the well dispersion of CB into the polymer matrix with strong polymer-CB interaction which restrict the entry of toluene molecules through it.



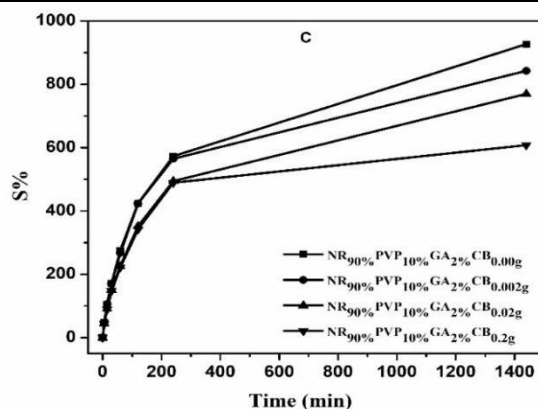


Figure 4 Swelling isotherm of NR/PVP semi IPN blends representing the effect of (a) polymer composition (b) GA concentration & (c) CB content

Equilibrium solvent content

The percentage of equilibrium solvent content for all films are shown in Table 2. From the table it is clear that a declining pattern is observed as PVP content increases. It can be the aggregation of PVP molecules in the NR matrix at its higher concentration, which prevent the films from swelling. Similar trend is observed with GA and CB variation study. It is a clear indication of the formation of a compact IPN films by increasing these parameters. So, the pore size available for the penetration of solvent molecule get reduced, which improves the solvent resistivity.

Kinetics of swelling

The theoretical equilibrium swelling (S_{max}) is computed from the corresponding slopes derived from ' t/S ' versus ' t ' plot, and the swelling rate and swelling rate constant are calculated from the intercept of their respective curves. Plots are depicted in figure 5. Swelling rate decreases with the incorporation of PVP into the NR latex due to the development of entangled networks in the semi-IPN synthesized. Table 2 makes it evident that theoretically calculated S_{max} values and the equilibrium swelling ratio obtained experimentally are in good agreement. A decrease in swelling rate is seen with increase in GA conc. and CB loadings. This is a clear indication of uniform dispersion of CB into the polymer matrix developing a stronger crosslinked structure restricting the solvent molecule to diffuse into the interpenetrated network films.

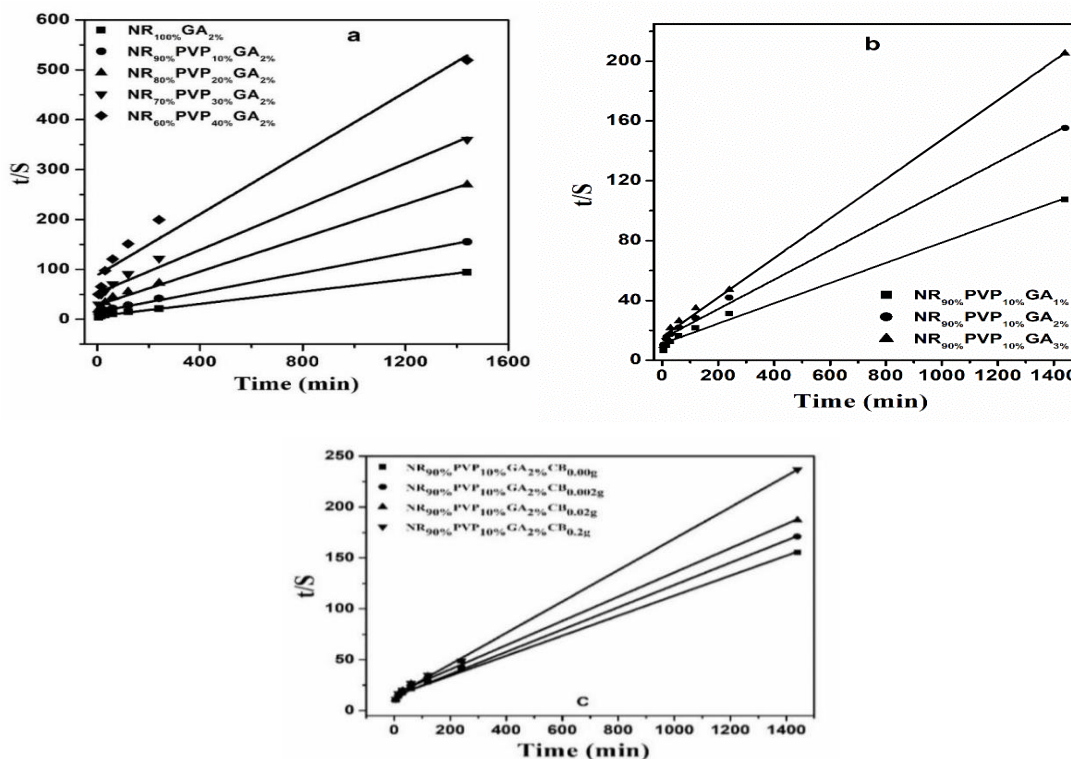


Figure 5 Swelling rate curves of NR/PVP semi IPN blends representing the effect of (a) polymer composition (b) GA

Mechanism of swelling

The figure 6 depicting ' $\ln F$ ' against ' $\ln t$ ' explores the mechanism of swelling. The swelling exponent (n) and swelling constant (K) are obtained from the slope and intercept of the plotted line. The K value decreases with rise in PVP, GA and CB content; showing the lessened solvent-polymer interaction blocking the easy transport of toluene molecules through the polymer matrix. ' n ' values of all system were found to be in between 0.47 and 0.58 signifies that diffusion mechanism follows a Fickian mode of transport [25].

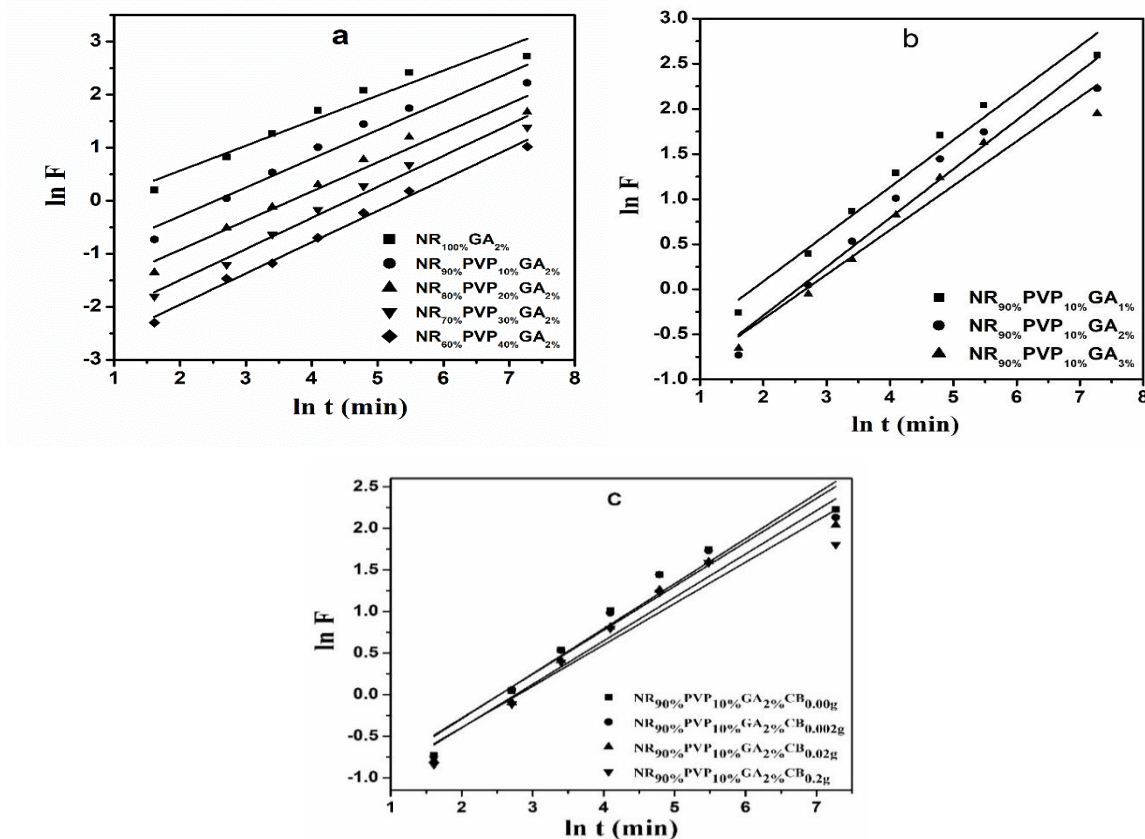
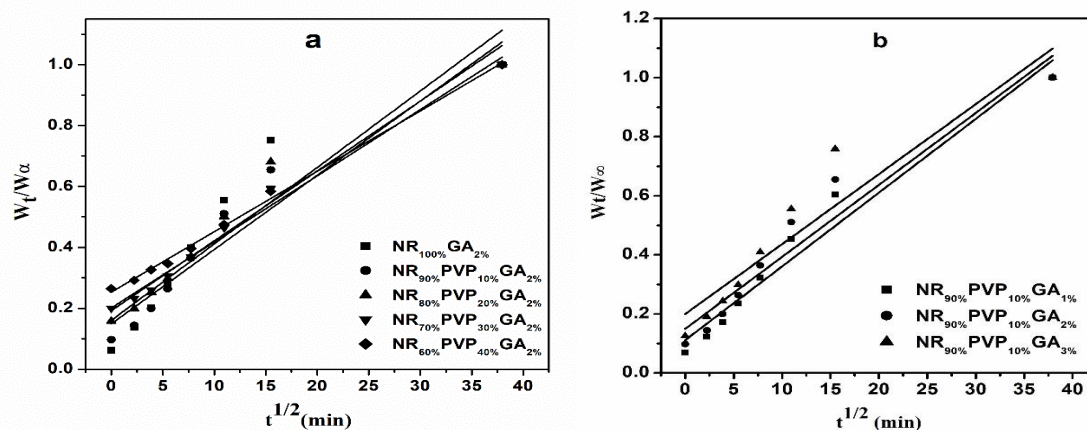


Figure 6 Swelling kinetic curves of NR/PVP semi-IPN films representing the effect of (a) polymer composition (b) GA concentration & (c) CB content

Diffusion studies

Plotting W_t/W_∞ against $t^{1/2}$ generates a line whose slope is employed to determine the diffusion coefficient (D). This is represented in figure 7. D value decreasing from $5.41 \times 10^{-9} \text{ cm}^2/\text{min}$ to $5.12 \times 10^{-9} \text{ cm}^2/\text{min}$ by incorporating 10% PVP into the NR matrix sounds that a well crosslinked network structure has developed. Further decrease in D value with increase in PVP content can be due to the healthy IPN formation. Similarly, decrease in D value is seen with increase in GA and CB content. Excessive crosslinking and continuous compatible semi-IPN composite formation will be the reason for this.



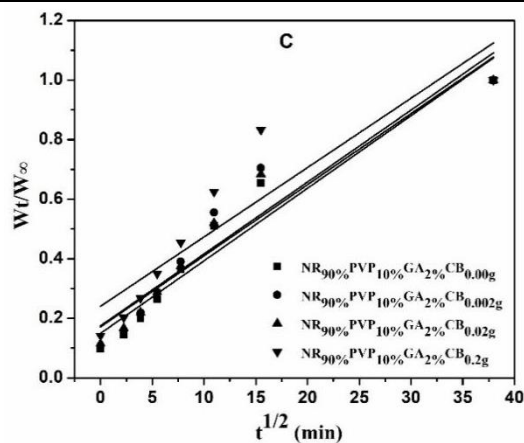


Figure 7 Diffusion curves of NR/PVP semi-IPN films representing the effect of (a) polymer composition (b) GA concentration & (c) CB content

Table 2. Swelling parameters of NR, NR/PVP semi-IPN and NR/PVP/CB composites

Particulars	NR _{100%} GA _{2%}	NR _{90%} P VP _{10%} G A _{2%}	NR _{80%} P VP _{20%} G A _{2%}	NR _{70%} P VP _{30%} G A _{2%}	NR _{60%} P VP _{40%} G A _{2%}	NR _{90%} P VP _{10%} G A _{1%}	NR _{90%} P VP _{10%} G A _{3%}	NR _{90%} P VP _{10%} G A _{2%} CB _{0.002g}	NR _{90%} P VP _{10%} G A _{2%} CB _{0.02g}	NR _{90%} P VP _{10%} G A _{2%} CB _{0.2g}
S%	1525	927	534	399	277	1339	702	842	769	608
ESC%	93.84	90.26	84.23	79.99	73.49	93.05	87.53	89.39	88.50	85.87
Ri	0.161	0.069	0.035	0.019	0.011	0.091	0.065	0.072	0.070	0.069
$k_s \times 10^{-3} \text{ min}^{-1}$	0.6	0.7	1.0	0.9	1.1	0.4	1.1	0.9	0.8	1.6
$S_{\max}$	1629	1016	593	462	327	1477	757	913	840	648
n	0.47	0.54	0.55	0.58	0.58	0.52	0.50	0.52	0.52	0.50
K	0.69	0.25	0.13	0.07	0.04	0.38	0.26	0.26	0.24	0.24
$D \times (10^{-9}) \text{ cm}^2 \text{ min}^{-1}$	5.41	5.12	4.51	4.05	3.40	5.37	4.83	5.03	4.91	4.66
Where S% = Swelling percentage, ESC% = percentage of equilibrium solvent content, Ri = Initial swelling rate, k_s = Swelling rate constant, $S_{\max}$ = Maximum equilibrium swelling, n = Swelling exponent, K = Swelling constant, D = Diffusion coefficient										

Removal efficiency of Bendiocarb.

The removal efficiency of bendiocarb insecticide from aqueous media were analyzed using NR/PVP system composed of 90% NR and 10% PVP with 2% GA; as this composition exhibited better property with well-developed semi-IPN structure. Removal potential is highly governed by the interaction of bendiocarb with the adsorbent material. It was observed from figure 8, that the %R for NR_{90%}PVP_{10%}GA_{2%} semi-IPN film was found to be 51% raised while comparing to pure NR films. One possible explanation for this could be the hydrogen bonds that form among bendiocarb molecule and semi-IPN polymer chains. Also, PVP being hydrophilic, causes the film to swell. When the film swells, it creates more space among the polymer chains, allowing easy pesticide diffusion into the polymer matrix. Improvement is observed in the insecticide removal capacity by the incorporation of CB. The addition of carbon black increases the film's pore capacity and surface area. This increased surface area offers additional sites for adsorption of the pesticide molecules, thereby enhancing the film's capacity to capture and retain them. Thus, from the analysis, it is revealed that the present material can be employed for removing bendiocarb insecticides from tainted water. Table 3 presents a summary of all the findings from the bendiocarb removal studies.

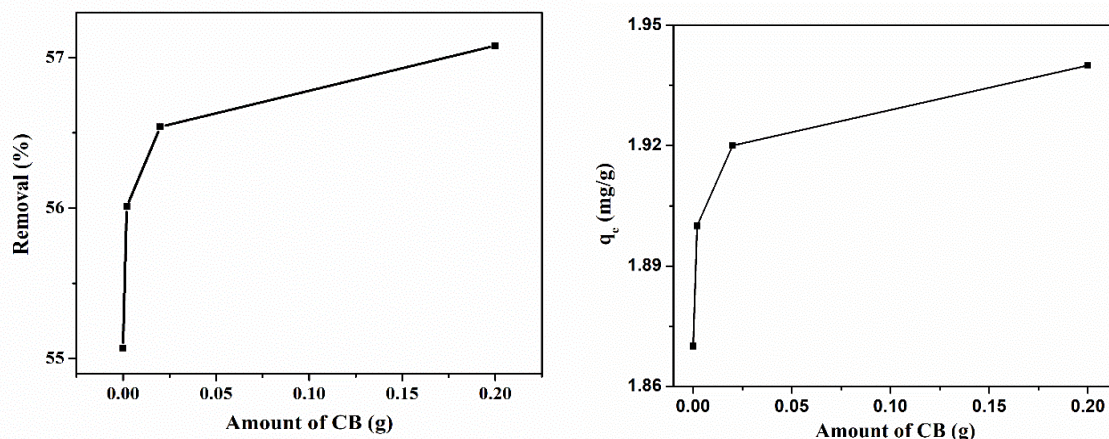


Figure 8 % Removal (a) and adsorption capacity (b) of bendiocarb from NR_{90%}PVP_{10%} semi IPN and composites

Table 3. Removal efficiency of bendiocarb pesticide using NR, NR/PVP semi IPN and its composites

Sample code	q _e	%R
NR _{100%} GA _{2%}	0.14	4.10
NR _{90%} PVP _{10%} GA _{2%}	1.87	55.07
NR _{90%} PVP _{10%} GA _{2%} CB _{0.002g}	1.90	56.01
NR _{90%} PVP _{10%} GA _{2%} CB _{0.02g}	1.92	56.54
NR _{90%} PVP _{10%} GA _{2%} CB _{0.2g}	1.94	57.08

III. Conclusions

A novel semi-IPN composites consisting of NR, PVP and CB were synthesized. Swelling studies of NR/PVP/CB film from non-polar solvent indicated that the diffusion mechanism as Fickian transport. This is a clear indication of uniform dispersion of CB into the polymer matrix developing a stronger crosslinked structure restricting the solvent molecule to diffuse into the interpenetrated network films. Moreover, composites exhibited better thermal stability. Compared to pure NR, NR/PVP semi-IPN can be employed as an adsorbent material for the removal of toxic chemical like bendiocarb present in the wet soil environment. The improved pesticide removal is likely due to hydrogen bonding between bendiocarb and the semi-IPN polymer blend, along with PVP-induced swelling that enhances pesticide diffusion. Incorporation of carbon black further boosts removal by increasing the film's porosity and surface area, providing more adsorption sites.

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