

Synergistic Impact of Sunlight Exposure and Graphene Compositization on The Performance of Conducting Polyaniline

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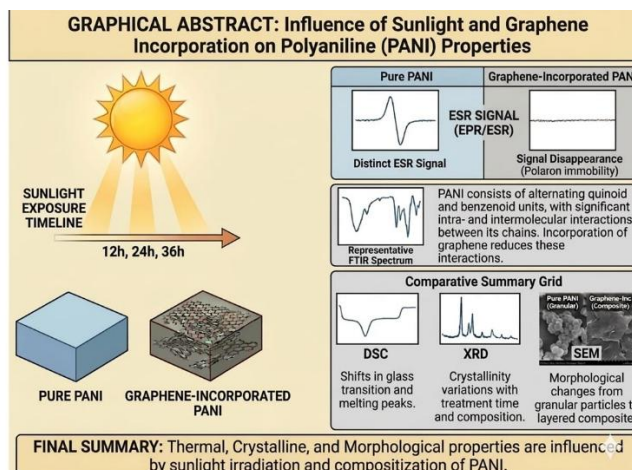
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ABSTRACT

Influence of sunlight exposure and compositization of graphene on conducting Polyaniline (PANI) has been investigated by spectroscopic Electron Spin Resonance (ESR), Fourier Transform Infrared (FTIR), Differential Scanning Calorimetry (DSC) and Scanning Electron Microscope (SEM) techniques. The inter and intra-molecular interactions/bonding of PANI are influenced by radiation treatment and compositization, affecting its chemical and physical properties. By monitoring variations in FTIR intensities of $3500\text{--}3000\text{ cm}^{-1}$ (corresponding to symmetric and asymmetric N–H groups, H–H, O–H vibrations) and $1600\text{--}1400\text{ cm}^{-1}$ absorption bands (corresponding to benzoic and quinoid groups) changes in chemical structure of PANI are ascertained. Such type of molecular interactions has been confirmed by ESR measurements. ESR spectrum of sun light exposed PANI does not contain hyperfine structure suggesting absence of hydrocarbon free radicals and chain cleavages have not occurred in polymer backbone. Charged species (polaronic) are thought to be responsible for the observed ESR signal. Due to compositization with GR the polaronic species become immobile and or react causing disappearance of ESR signal. DSC thermogram of PANI compose two endothermic peaks centred around 45°C and 240°C corresponding to release of moisture/water and glass transition temperature respectively. The peak positions were shifted on sunlight exposure and Graphene (GR) addition due to formation of networks in both the cases. Morphology of PANI is altered by sun light exposure as well as incorporation of GR. Formation of granular cracks and bores on PANI surface are evident due to sun light exposure; whereas cluster-like structures together with pores are observed on PANI@GR due to sun light exposure. The broad XRD peaks of PANI suggest absence of crystalline reflections and presence of amorphous domains of molecular networks.



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1. INTRODUCTION:

Conducting polymer composites have attracted significant attention and hold great promise due to their exceptional features and distinctive physicochemical properties, enabling a wide range of advanced applications¹. These unique properties facilitated interest especially in towards electronics and electrochemical activities. They have the electronic properties of semiconductors alongside of mechanical and processing advantageous parameters of polymers. One such potential material having conducting and electrochemical properties is Polyaniline (PANI). PANI based materials occupy an important place among the conducting polymers used in different applications. In general, PANI is fabricated by protonic acid doping performed via oxidation, either chemically or electrochemically. It can be readily synthesized under suitable conditions through both physical and chemical methods. These approaches are cost-effective, easily reproducible, and widely recognized for producing high-quality material. The main backbone features of PANI are benzoid and quinoid units with the delocalized conjugated structure that are response for the resonance hybridized having different redox states. Benzoin rings present in the structure are nonconducting in nature. Emeraldine salt having Π -conjugated system plays an important role in increasing its electrical properties. Protonation of emeraldine could be accomplished by chemical, photochemical or electrochemical methods²⁻³. PANI exists in multiple forms, namely leucoemeraldine base (LB), pernigraniline base (PB), and emeraldine base (EB). These forms differ in their physical and chemical characteristics, leading to distinct physicochemical properties that make them suitable for a wide range of potential applications. PANI is a promising material for many technological applications in various areas like sensors⁴, light-weight batteries⁵, supercapacitors⁶, corrosion inhibitors⁷⁻⁸ and EMI shielding⁹.

PANI is widely studied because its conductivity can be tuned by protonation, oxidation state, and morphology, but its practical use of outdoors depends strongly on photoaging and stability. For sunlight exposure, the main concern is that ultraviolet components of solar radiation, can

change both the doping state and the chemical structure of the polymer, which in turn alters optical absorption, color, and electrical conductivity. Existing sunlight- and UV-exposure studies suggest three recurring conclusions. First, direct ultraviolet irradiation can induce dedoping in protonated PANI. Kaitsuka and Goto reported that exposing PANI/camphorsulfonic acid films to 375 nm UV light produced a clear decrease in visible–near-infrared absorption associated with the conducting emeraldine salt state, and they interpreted the change as light-induced dedoping with a radical-mediated contribution¹⁰. Second, longer-term aging studies consistently show a decline in conductivity under ambient or oxidative conditions. Herrasti et al. found that aged PANI–HCl exhibited substantial macroscopic conductivity decay even when intrachain transport signatures changed less dramatically, indicating that environmental aging strongly affects inter-grain or mesoscale transport pathways¹¹. Third, recent degradation work emphasizes that formulation matters: additives, dopants, and composite architectures can either accelerate photochemical reactions or improve stability, depending on whether PANI is being used as the active photocatalyst or as the material to be protected¹²⁻¹⁵.

Recent studies suggested that graphene, graphene oxide (GO), and reduced graphene oxide (rGO) are introduced to modify the morphology, charge transport, wettability, and electrochemical stability of PANI. The main current themes are better interfacial charge transfer, improved cycling stability, and higher accessible surface area for sensing or energy storage applications. Recent primary studies show that the graphene component changes both structure and performance rather than acting as a passive filler. Yu et al. electrochemically synthesized a functionalized graphene/PANI composite on nickel foam and reported a large increase in specific capacitance relative to neat PANI, attributing the improvement to the high-surface-area graphene support and synergistic electronic interaction between the two phases¹⁶. Balboa-Palomino et al. then showed that increasing GO content in electrochemically synthesized PANI–GO films improved hydrophilicity and reduced capacitance loss during cycling, indicating that GO can improve the operational stability of PANI-based electrodes when incorporated at suitable loading¹⁷. In a complementary study, Fenniche et al. fabricated three-dimensional PANI nanofiber/GO and PANI nanofiber/rGO thin films and found that intertwined nanofiber–nanosheet architectures provide a flexible electrochemical platform for sensors, supercapacitors, and related devices¹⁸. The most mechanistically informative recent work is that graphene/PANI interface study by Bl'aha et al. which examined charge

redistribution at the molecular scale. They concluded that polyaniline behaves as a p-type dopant of graphene during Fermi-level alignment, while graphene electrons help stabilize polarons and bipolarons in the polymer¹⁹. This result is important because it moves the field beyond simple “graphene improves conductivity” statements and gives a more explicit interfacial explanation for why graphene/PANI hybrids often outperform pristine PANI. Madhabi et al reported the improved in thermal and electrical properties of PANI introducing insitu reduction of graphene oxide plane that further reduced to rGO/PANI nanocomposites exhibiting a excellent performance and observed the enhancing in physical properties by varying concentration of graphene oxide in the composites²⁰.

Taken together, the recent literature suggests that graphene incorporated polyaniline is most useful when it creates a well-coupled interface rather than merely increasing carbon content. Therefore, it is desirable to have knowledge on photo degradation effects in PANI and graphene doped PANI. Both PANI and GO- PANI are exposed to sun light to different times and resultant effects are investigated experimentally with various spectroscopic techniques.

Scope of Present Studies

1. UV light irradiation of PANI can be treated as equivalent to its doping. Because the UV light exposure creates reactive species that acts as dopants²¹. On a similar line the authors propose

the effect of sun light radiation changes in chemical and physical properties of PANI.

2. PANI and its graphene derivatives PANI (GR, GO, rGO, GCN etc) are well known for the photo catalytic applications. During the application they have been exposed to sun light along with the pollutants (methylene blue etc).
3. Though photo degradation of PANI is attempted, a comprehensive study involving techniques ESR, FTIR is reported. Therefore, authors recorded ESR spectra of PANI under different conditions and the data is compared with the PANI –graphene composite.
4. Thermal properties are compared by recording DSC thermograms under different conditions.

Experimental:

PANI and PANI@GR in the form of black powder are purchased from Sigma Adrich and used as received. FTIR spectra are recorded using a Perkin Elmer spectrometer over the range of 4000–450 cm^{-1} with a resolution of 4 cm^{-1} for pellets of samples mixed with KBr. ESR spectra are recorded on Bruker spectrometer operating at X-band frequencies and 100 KHz modulation at room temperature. DSC thermograms are recorded on MettlerToledo calorimeter with a heating rate of 10°C/minute under flushing Nitrogen. Morphology is investigated by SEM microscopy using VEGA3 TECAN microscope for the samples painted with conductive coating. X-ray diffractograms are recorded on Philipps spectrometer using $\text{K}\alpha$ radiation.

RESULT & DISCUSSIONS:

FTIR Studies:

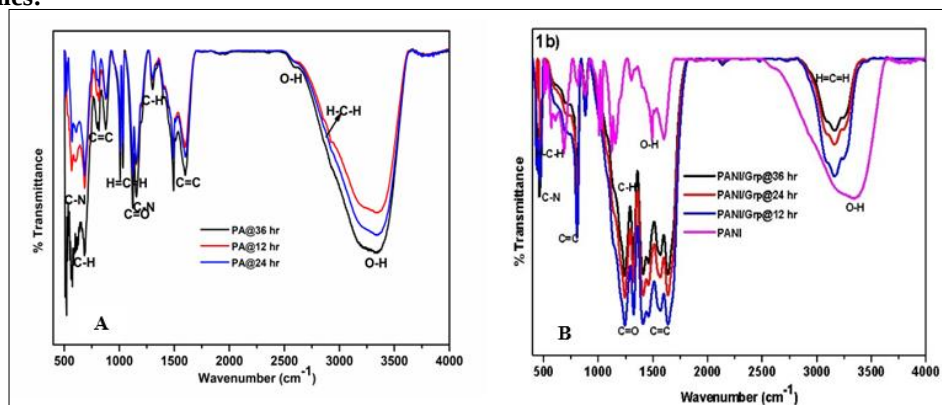


Fig.1: FTIR spectra of a) PANI b) PANI@GR exposed to sun light at 12 hrs, 24 hrs and 36 hrs.

FTIR spectra of PANI exposed to sun light for different times are as shown in Fig 1a. The spectra contain several absorption bands centred around 3337, 3050, 2930, 1600, 1493, 1370, 1240, 1155,

1123, 1033, 1008, 870, 800, 685, 572 and 519 cm^{-1} positions. The absorption bands represent various chemical groups and atoms of PANI as given in Table 1. On exposure to sunlight, the band positions and intensities varied with exposure time.

Table 1: FTIR absorption bands of PANI exposed to sunlight and their assignment²²

S.No	Band positions	Assignment
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	Exposure Time			
	12 hrs	24 hrs	36hrs	
1	3333	3337	3332	N-H symmetric stretch/OH
2	3050	3050	3000	N-H Asymmetric stretch N-H Symmetric stretch H-H
3	2920	2920	2920	C-H/CH ₂ (OR)CH ₃
4	1600	1599	1600	Quinoid vibration
5	1493	1493	493	Benzoid vibration
6	1370	1370	1370	C – H vibrations
7	1240	1240	1240	C-N stretch
8	1155	1155	1155	C-H/C-N
9	1123	1123	1123	Aromatic C–H vibrations
10	870	870	870	C-H out of plane vibration
11	800	800	800	C-H
12	685	685	685	C=C, C-H vibration
13	572	572	572	C-N-C vibration
14	519	519	519	C-N-C vibration

Sun light exposure of PANI or doping of GR results molecular interactions influence FTIR band positions and band intensities. By monitoring these changes, it is possible to estimate extent of interactions occurring in polymer²³⁻²⁵. PANI exhibit FTIR absorption bands in two important regions i) 3500– 3000 cm⁻¹ representing the N–H symmetric & asymmetric stretch/H– H bonding/OH/COOH vibrations and ii) 1600–1400 cm⁻¹ absorption band correspond to benzoid /quinoid group vibrations. The observed FTIR spectral shapes indicate intensity of 3500–3000 cm⁻¹ band is broad/intense/predominant when

compared to the remaining absorption bands which appeared like latent structures (as shown in Fig 1a). When PANI is exposed to sun light exposure or GR doped, intensity of 3500- 3000 cm⁻¹ band appeared to have decreased at the expense of increasing of intensity of remaining absorption bands. Similar results are observed in case of PANI@GR also. The intensity ratio of 3500-3000/1600–1400 cm⁻¹ band is taken as measure of extent of molecular interaction. The results are compared in terms of histograms depicted in Fig 2a and 2b (sun light exposed PANI). The decrease is more for PANI than PANI@GR.

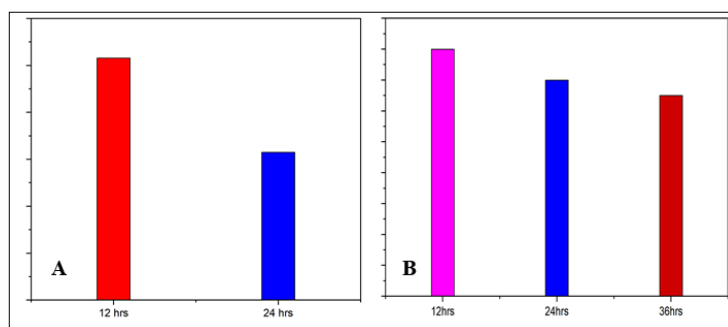


Fig 2: Histogram Depicting intensity ratio variation of 3600 – 3000 / 1600 – 1400 cm⁻¹ absorption bands a) PANI b) PANI@GR

ESR Studies

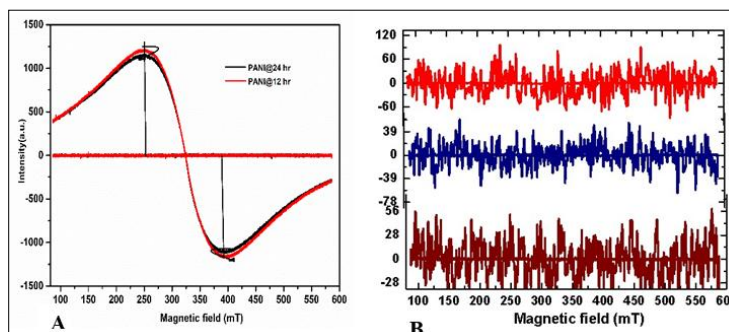


Fig 3: ESR spectra of PANI exposed to sunlight a) homo polymer 12 hrs and 24 hrs b) PANI@GR Red – 12 hours: Blue- 24 hours: Brown -36 hours

ESR spectra of sun light irradiated PANI are broad singlet as shown in Fig 3a. Lack of hyperfine structure of the spectrum indicate the absence of

hydro-carbon radicals (which normally cause hyperfine spectra in irradiated polymers). The result suggest that chain cleavages have not

occurred either in the backbone of PANI or on its constituent chemical groups. Due to the presence of benzoid and quinoid groups in its backbone, the PANI is considered to be radiation resistant and cleavage of chains is difficult²⁶. In the absence of hydrocarbon free radicals, the observed Singlet spectrum is assigned to be due to polarons, which are normally present in conducting polymers including PANI. Presence of ESR singlet and polarons conducting polymers and in PANI is also reported previously²⁷. Further, formation of free electrons in PANI is also possible due to protonic rearrangements which may also give ESR singlet spectra^{28, 29}. The broadness of ESR singlet in the present studies also suggest contribution from free electrons.

With the increase of exposure time the intensity ESR singlet slightly decreased and lobe ratio (a/b) was changed. Kalikov³⁰ have observed changes in ESR line shape/width in PANI and attributed the spectral changes to be due to distribution and mobility of polarons in the polymer matrix arising due to doping of the polymer³¹⁻³² have reported that conductivity of PANI is related to peak-peak width

(ΔH) of ESR signal. The conductivity increases with delta H pp value and it decreases with decrease of lobe ratio (a/b) and g-value. Therefore, the decrease in ESR intensity and g- value is in consequence with literature reports.

ESR spectra of PANI@GR exposed to sunlight at different times are as shown in Fig 2b. The absence of signal indicates presence of either the polarons or free electrons as observed for PANI. The disappearance or decrease of the ESR signal in PANI with microwave irradiation has been previously reported, where the authors observed that microwave exposure reduces its conducting state to an insulating state³³. The authors reported that the conducting state of PANI is reduced to an insulating state due to microwave irradiation³¹. Oshawa et al (R) have reported that suppression of inter chain transfer of polaronic species decreases due to coulombic interactions resulting in decrease / disappearance of ESR signals. Therefore, in the present studies compositization of PANI with GR may also expect a similar effect of suppression of polaronic species causing disappearance of ESR signal.

DSC studies

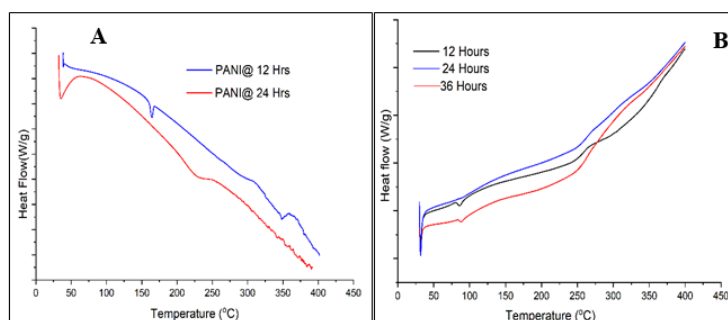


Fig. 4: DSC thermograms exposed to sunlight(a) PANI 12 hrs and 24 hrs(b) PANI@GR 12 hrs, 24 hrs and 36 hrs

DSC thermograms of PANI exposed to sunlight for 12 hrs and 24 hrs exhibited endothermic peaks centred around 44 °C (peak P1) and 240 °C (peak P2) as shown in Fig4a. Thermal parameters

associated with the two peaks [T onset (T_o), T peak (T_p), T end set (T_E) and peak enthalpy (ΔH)] are listed in Table2.

Table. 2: Thermal parameters associated with PANI exposed to sun light for 12 hrs and 24 hrs

Exposure Time	Peak P ₁				Peak P ₂			
	T_o	T_p	T_E	ΔH J/g	T_o	T_p	T_E	ΔH J/g
12hrs	34	44.4	68.4	-298	212	240	256	-117
24hrs	45.4	53.6	53	-73	213	240	256	-72

Endothermic peak at 45 °C (peak P₁) correspond to the removal/release of moisture/ water trapped in polymer matrix either during synthesis of polymer or due to chemical reactions undergone by the polymer during heating³⁴. Such types of endothermic transitions are also reported previously^{22,35}. With sunlight exposure time the value of T_1 increased, while T_2 value remains the same With regard to endothermic peak P2 at 240

°C, it is corresponded to glass transition temperature of PANI. The Molecular chains of PANI are considered to have high inter-chain rigidity and high inter / intra chain hydrogen bonding together with dipolar interactions. For such a complex polymer system Bhadra used simulation techniques³⁶ and reported that the T_g value in between 30–250 °C. The T_g value reported in the present studies is with in this range.

Normally the glass transition is considered to be a first order transition accompanied with base-line shift; instead, an endothermic peak is observed in the present studies. This type of endothermic peak at glass transition temperature indicative of time dependent molecular relaxations in the T_g region³⁷.

For PANI@GR, two endothermic peaks centred on 86°C (peak P₁) and 245°C (peak P₂) are observed.

Thermal properties associated with the two peaks T_{onset} (T_o), T_{Tmax} (T_p), T_{end set} (T_E), heat of enthalpy (ΔH) are summarized in Table 3. The data indicate that P₁ shifted from 45°C (for PANI) to 86°C while endothermic peak at 240°C (for PANI) shifted to 245°C (5 °C shift). The peaks are assigned to release/evolution of water /moisture and glass transition temperature respectively³⁶.

Table 3: Thermal parameters associated with PANI@GR exposed to sun light for 12 hrs, 24 hrs and 36 hrs

Exposure Time	Peak P1				Peak P2			
	T _O	T _P	T _E	ΔH J/g	T _O	T _P	T _E	ΔH J/g
12 hrs	81.1	86.29	93.43	4.48	208	205	267	22.26
24hrs	83.3	88.6	95.45	-2.04	179	246	286	86.22
36 hrs	61.9	89.3	114.4	-6.17	203	246	270	24.04

The differences in P1 and P2 values of sun light exposed PANI and PANI@GR are thought to be associated with the change in physicochemical and structural properties of PANI brought out by intense network formation due to sun light exposure and GR addition (R)PANI an inspiring and extreme conducting polymer³⁸. Due to sun light exposure of PANI@GR, the T₁ decreased by 10°C but T₂ or T_g value remained the same.

SEM studies

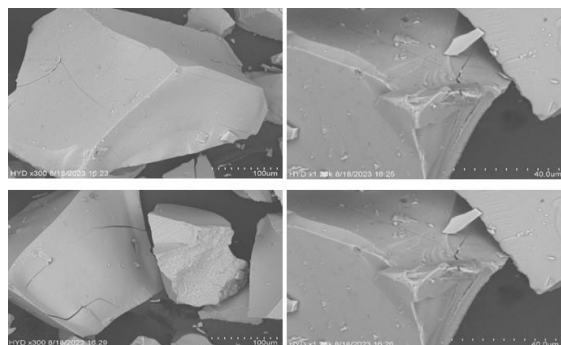
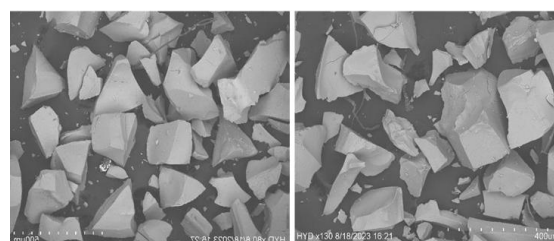


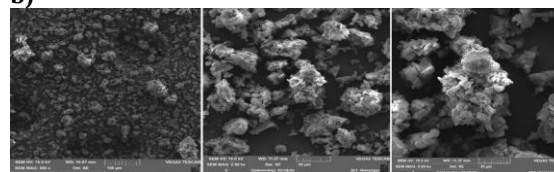
Fig. 5: SEM micrographs of PANI exposed to sunlight (a) 12 hours (b) 24 hours

Effect of sunlight exposure on surface morphology of PANI is investigated by recording SEM micrographs shown in Fig 5a. The smoother surface of PANI has become rough and formation of cracks and bores were observed due to sunlight exposure. The cracks size ranged from 20 - 100 μm and holes with a diameter of 1-5 μm determined using granulo metric methods and reduction in granular size and shape were noticed.³⁹

a)



b)



c)

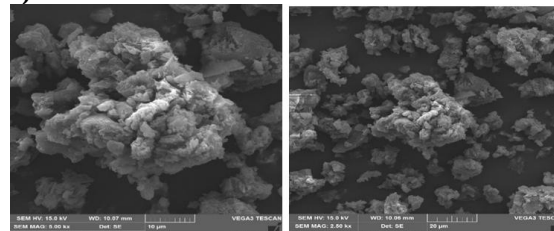


Fig.6) SEM micrographs of a) unexposed PANI b) Sunlight exposed PANI@GR 12 hrs c) Sunlight exposed PANI@GR 24hrs

The SEM micro graphs of PANI @GR (shown in Fig 6b) suggest granular structure of PANI is changed to aggregated particles structure due to addition of GR in polymer matrix. The particle/ granular shape of PANI is considerably altered to cluster shape structure due to compositization. The result suggest that severe interactions have occurred between PANI and GR. The irregular clusters have sizes ranged from few Nano meters to hundreds of micrometres indicating non-uniform interactions took place. When the nanostructures are exposed to sunlight, the cluster structure is almost found to be intact in case of PANI@GR 24hrs. Whereas for PANI@GR 48hrs the clusters are influenced by sunlight exposure. Small holes

together with minute cracks can be seen on the cluster surface. Due to severe interactions cluster structure is intact so that sunlight exposure does not have much influence on morphology of PANI@GR. The results are in agreement with the FTIR and ESR data.

XRD studies

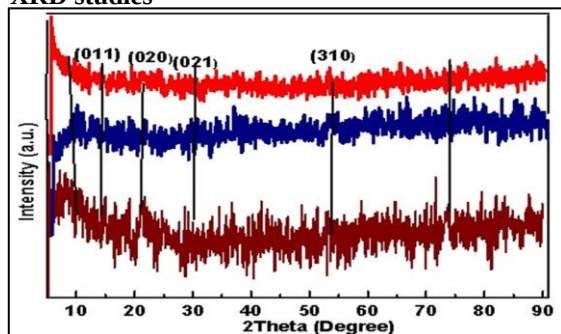


Fig 7: XRD patterns of sunlight exposed PANI at various times

XRD spectra of PANI exposed to sunlight for different times are as shown in Fig 7. The diffractograms exhibited diffused diffraction peaks centred around $10-50^\circ$ suggesting the amorphous nature of polymer. They correspond to (011), (020), (021) and (310) planes [34,40]. On sunlight exposure of PANI, the broadening of peaks further increased suggesting the increase of amorphous suggesting the crosslinking of molecular chains.

CONCLUSION:

Effect of sun light exposure and GR compositization of PANI has been successfully investigated. For PANI sun light exposure causes dissociation of chemical bonds of causing molecular interactions which influence FTIR spectra. Due to these molecular interactions either by irradiation or compositization the diminishing of polaron charge carriers occurs causing decrease of ESR intensities. The molecular interactions could be studied by monitoring intensity ratio of FTIR absorption bands. Thermal properties are influenced by irradiation as well as compositization. The water / vapour release temperature increased while the glass transition temperature increased due to irradiation and compositization. Morphology of PANI was remarkably changed in form of cracks, pores, but its granular structure was not effected seriously. But due to compositization with GR the granular structure of PANI transformed to cluster structure of different sizes ranging from micro meters to nano size. Intense network formation resulted in cluster structure, which played resistant role against sun light exposure resulting in less degradation. Only small cracks / hole / pores are resulted in nano composites. The XRD data suggest the polymer almost retained amorphous nature in all cases.

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