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Electron Beam Irradiated Polyaniline/$LiClO_4$ Composite: Structure and Morphological Studies

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Abstract: Polyaniline and PANI-Lithium perchlorate composites were prepared by chemical reaction method. The composites are characterized with the help of FT-IR, SEM and XRD to confirm the chemical interactions and crystalinity, change in surface morphology and optical changes respectively. The obtained results suggesting these polymer composites are suitable candidates for optoelectronics display, electrode material and electrochromic devices.

Keywords: Polyaniline, DSC, SEM, XRD

Introduction

Recently there is great interest in nano sized materials to be involved in many areas of applications such as electromagnetic shielding [1], anti corrosion [2],electronics, biosensors [3, 4] and biochemical/biomedical engineering [5]. Conducting polymers have attracted considerable interest because of their excellent physical and chemical properties originating from their conjugated systemThe widely exploited properties of conducting polymers in technological and commercial applications are their thermally withstanding capacity, bio-compatibility, electrical conductivity, switching capability between conducting-oxidized and insulating-reduced state Polyaniline is one of the most investigated intrinsically conductive polymers (ICPs), due to its interesting physicochemical properties such as electrical conductivity, deep black color, ion-exchange capacity, and hydrophobic nature, strong adsorption capacity towards molecular and macromolecular species. PANI can be prepared in various forms including thin films, powders, colloidal particles, hollow particles, nanotubes, micrometer-sized composites and nano composites [6-9].

Materials and Methods:

Aniline (99.0%), Lithium perchlorate ($LiClO_4$) (mol.wt.106.4 g/mol), ammonium persulfate (APS, 98.0%) and hydrochloric acid (mol wt, 36.46 g/mol)were purchased from Sigma-Aldrich, USA. Polyaniline (PANI) and PANI/ Lithium Perchlorate ($LiClO_4$) composite (PALi)was synthesized in 100 ml 1M HCl using ammonium dipersulphate$(NH_4)_2S_2O_8$ (APS)as an oxidizing reagent by chemical reaction method.The PALi composite was irradiated using 8.1 MeV electron beam (EB) energy in the LINAC, RRCAT-Indore at 40, 80 and 120 kGy doses.The chemical change of the PALi composite before and after EB irradiation was analyzed by Fourier transform infrared spectrometer (FT-IR, ALPHA BRUKER) in the spectral range of 2500-500 cm^{-1} and surface morphology has been observed using a Sigma Zeiss scanning electron microscope (SEM).XRD characterization done by using X-ray Diffractometer wavelength λ=1.5406Å and samples were scanned from 10 to 60^o at the scanning rate of 10^o per minute. Thermo gravimetric analysis (TGA) and Differential Thermal Analysis (DTA) are studied using Q-600 TA instruments heating from 30 to 800 ^oC and from 30 to 300 ^oC with heating rate of 10 ^oC/min under nitrogen flow.

Results and Discussions

SEM

Figure shows SEM image of Unirradiated, EB irradiated pure PANI and polyaniline composites The clear observation of SEM image of plain PANI shows some pores, which are usually observed due to some deformation of polymer film during casting. However in some streaks with disjoints are also observed in the image. Composite images show that, the fine dispersion of salt particles in the PANI matrix is observed, irregular shaped particles with compacting nature of the particles are also observed. These films look more homogeneous however, particle agglomeration can't be ruled out.

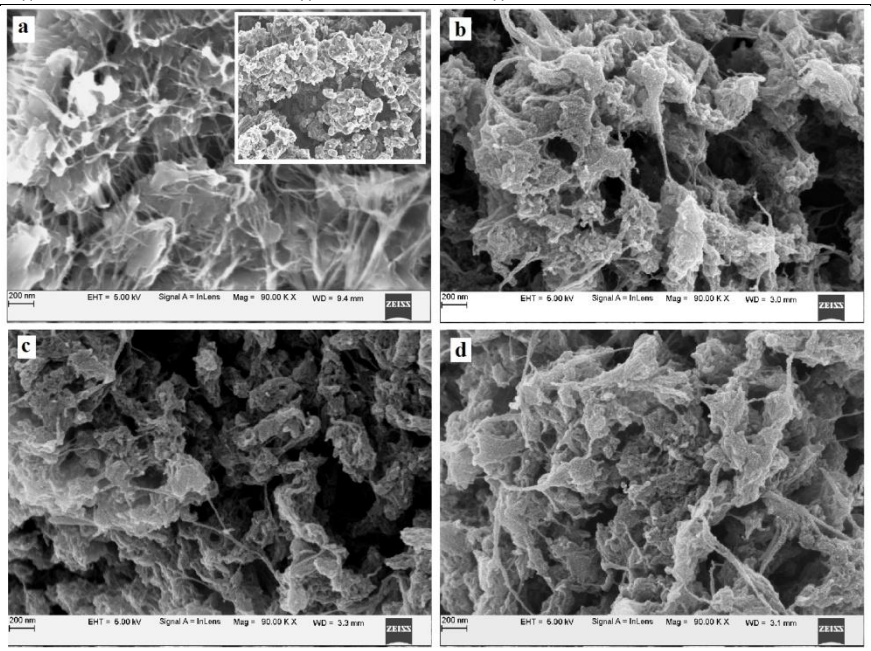

Figure1: SEM images of Unirradiated a) PALi (insert-pure PANI), EB irradiated PALi at b) 40 kGy, c) 80 kGy and d) 120 kGy.

FT-IR Analysis

The FT-IR spectra of non-irradiated, EB and GR irradiated polyaniline compositesare shown in fig. It reveals that some peaks intensity changed in the electron beam and gamma ray irradiated films in comparison with non-irradiated film. The bands at 824 and 1184 cm^{-1} is due to the CH_2 bending vibration, which correspond to the crystalline phase seem to decrease with irradiation dose. This may be attributed to the destruction of crystalline lamellar structure of polymer. The intensity of the bands at 1621 and 1192 cm^{-1} respectively represents the ketonic carbonyl groups C=C and C–O–C group.The change in intensity and transmittance of these peaks indicates the occurrence of chain scission and cross-linking. The peak intensity is increased and transmittance is decreased in EB irradiated films with respect to that of gamma irradiated films. Also, these results suggest that the affects are more pronounced in electron beam compared to that of gamma ray irradiation.

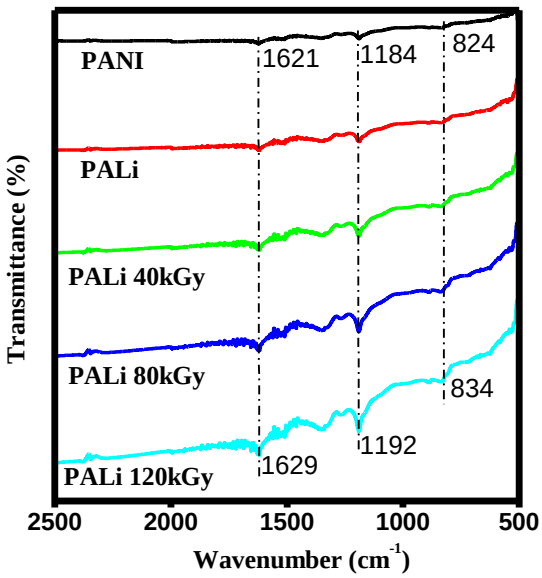

Figure2: FTIR spectra of unirradiated and EB irradiated PALi composite

XRD Analysis

XRD analysis gives the effect of the radiation dose on the structural phase of PANI composites. The X-ray diffraction spectra of non-irradiated and irradiated are shown in Fig. The XRD spectrum of a non-irradiated film shows partly crystallinity and amorphous phase. The changes in the structural parameters after irradiation affect the crystalline phase of the polymer; The increase in FWHM (β) and peak intensity (I) is clearly represent the decrease in crystallinity nonirradiated compared to EB-irradiated samples. These results suggest that EB irradiation is more effective on conducting polymer samples.

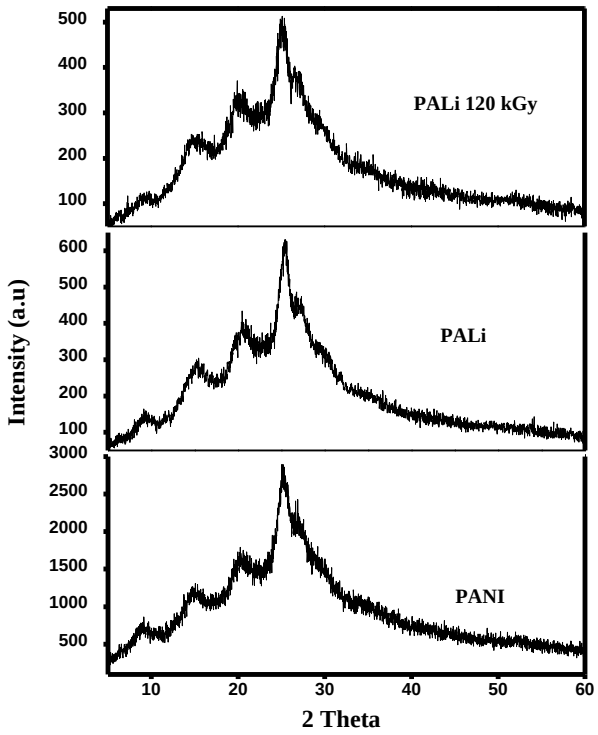

Figure 3: XRD spectra of PANI, unirradiated PALi and EB 120 kGy dose irradiated PALi composite.

Conclusion

We have investigated the EB irradiation induced modifications in polyaniline composite using FT-IR, XRD, SEM, and TGA ,DTA techniques. The results show the change in the crystallinity and chemical structures polymers after the irradiation by EB radiation. The modification in the irradiated samples was confirmed from FT-IR that the chain scissoning/branching occurs for both process, as the intensity of the wave number enhances with incremental irradiation. Another remarkable peak observed at 1621 cm^{-1} corresponds to the presence of C=C group. An increase in the intensity of this peak suggests the occurrence cross-linking.. In addition, the effects of EB irradiation on structural morphology was investigated by SEM, and the images reveal that after irradiation there is improvement in the surface morphology of the samples.

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