Low-Density Polyethylene/Calcium Ferrite Nanocomposites Films Prepared for Structural, Morphological, Mechanical and DC Conductivity Characterization

Nayeemuddin, S A M N Quadri

Abstract: Low density polyethylene (LDPE)/Calcium ferrites (Ca:Fe2O4) hybrid nanocomposites (NCs) were synthesized by means of in-situ polymerization using Calcium nitrate as metal precursor, LDPE as polymerizing agent and polyethylene glycol (PEG) as a fuel. The prepared nano composites films were characterized by different characterization techniques such as Powder XRD, FTIR, SEM and TEM. The electrical properties of Low density polyethylene (LDPE)/Calcium ferrites (Ca:Fe2O4) hybrid nanocomposites (NCs) are very unique in comparison to the pure phases, LDPE and Ca:Fe2O4 nanoparticles (NPs), which were ascribed to the interaction between the LDPE and Ca:Fe2O4 nanoparticles. DC conductivity is observed to be enhanced as the Ca:Fe2O4 NPs concentration increased from 10 to 50 wt% in the hybrid NCs at the different temperature. Beyond the 14 Mpa Stress all the samples showing the sudden breakage stage. From the obtained measurements and data, the synthesized hybrid nano composites may be used in electronic device applications such as Parcel carriage Bags, designing of automobiles such as in aerospace parts due to its lights weight, as LED devices, solar cells and optical devices.

Keywords: LDPE, Calcium ferrite, XRD, TEM and Stress-Strain Graph and Conductivity.

I. INTRODUCTION

Hybrid composites of polymer and inorganic nano oxides have been broadly utilized as a part of the different fields, for example, military types of gear, safety, defensive suits of clothing, catalysis, car, aviation and optical gadgets as a result of their exceptional properties rising up out of the combination of natural and inorganic hybrid materials. For utilizing in various application zones consistently request extra properties and capacities, for example, high mechanical properties, fire hindrance, resistance to chemicals and various radiations, ecological steadiness, water repellency, electro-magnetic field resistance[1,2]. In addition, powerful features of the polymer and inorganic oxide nano-particles hybrid composites are dependent upon the constituents and their volume fraction, geometrical structure and incorporations of matrix and filler material, surface interactions among the matrix and inclusion. With the ongoing advancements in the materials science, the relationship of material properties with filler estimate has turned into a convergence of huge intrigue [3]-[5]. The natural leading polymers like polyethylene (LDPE) Poly-aniline (PANI) [2], Poly-pyrrole (PPy), Poly-thiophene (PTh) are used for the composites.

Among the mentioned polymers, polyethylene (LDPE) have pulled in extraordinary intrigue as a result of their various outstanding properties in comparison to others [5, 6]. Inorganic oxide materials at nano regime were effectively examined because of their potential applications and logical interests. Due to the size impact these materials exhibit good physical, mechanical and chemical properties [3],[5]. Low density polyethylene (LDPE)/Calcium ferrites (Ca:Fe2O4) hybrid nanocomposites (NCs),NCs comprising of Low density polyethylene (LDPE) (natural material) with Calcium ferrites (inorganic-oxide nano-material) researched increasingly on the grounds that their properties are very not quite the same as polyethylene (LDPE) and comparing Ca:Fe2O4 NPs attributable to interfacial interactions among the LDPE and Ca:Fe2O4 NPs [8], [9]. In order to enhance the electrochemical property of LDPE and its NCs, several reports are there. [5]-[7].The present work deals with the preparation of hybrid nanocomposites by dispersing the CaFe2O4 nanoparticles in the Low density polyethylene (LDPE)/ matrix. For this, inorganic oxide Ca:Fe2O4 NPs were synthesized by solution combustion green synthesis route and are then embedded in the Low density polyethylene (LDPE)/matrix and the obtained NCs were analyzed using various characterization techniques [9]-[10].
II. EXPERIMENTAL METHODS

All the chemicals were received from the commercial sources and used as received. The high purity Annular grade chemicals like calcium nitrate hexa-hydrate [Ca(NO$_3$)$_2$·6H$_2$O; purity 99.9%] ferric nitrate [Ca(NO$_3$)$_2$·6H$_2$O; purity 99.9%], low density polyethylene ( purity 98%), Ammonium per sulfate [(NH$_4$)$_2$S$_2$O$_8$; purity 99.9%], hydrochloric acid (HCl; purity 37%), Acetone [CH$_3$COCH$_3$; purity 99.9%] and polyethylene glycol (PEG) were taken from Sigma Aldrich as starting materials.

A. SYNTHESIS OF CALCIUM NANO PARTICLES

Low temperature combustion route is utilized to prepare CaFe$_2$O$_4$ NPs using polyethylene glycol (PEG) as organic fuel. Equal molar ratio of calcium nitrate hexa-hydrate and ferric nitrate hexahydrate was taken in a ceramic crucible then fixed with fuel, mixed homogeneously. Obtained reaction mixture was introduced into the pre heated muffle furnace whose temperature was maintained at 400°C. Here, combustion reaction takes place between metallic nitrates and fuel yielding nano powders of CaFe$_2$O$_4$.

B. SYNTHESIS OF LDPE/CALCIUM NANOPARTICLES

Synthesis of low density polyethylene (LDPE)/ Calcium ferrites (CaFe$_2$O$_4$) hybrid nanocomposites (NCS) Nano Films of LDPE/ CaFe$_2$O$_4$ was done using solution casting polymerization technique. LDPE/ CaFe$_2$O$_4$ NCS containing various percentages of Ca:Fe (i.e.10, 20, 30, 40, & 50 wt %) were synthesized by above said techniques.

III. RESULTS AND DISCUSSION

A. XRD analysis.

The XRD patterns of the synthesized samples were recorded in Shimadzu PXRD-7000 model for phase confirmation. The X-ray diffraction patterns of pure LDPE, and LDPE/CaFe$_2$O$_4$ (10-50 %) NCS films were presented in Figure 2. A wide diffraction peak was observed at nearly 28° Bragg’s angle (2θ) for LDPE nano particles [8]. The wide peak of LDPE gets weaker on doping with the filler CaFe$_2$O$_4$ NPs in the present LDPE polymer matrix. The XRD patterns of the LDPE/Ca:Fe$_2$O$_4$ are shown in Figure 2 and it shows a simple cubic structure of with lattice parameters a = b = c = 4.1678Å and is in accordance with the standard JCPDS card No.73-1519 [10]-[14].

Figure 2.XRD Pattern of LDPE/CaFe$_2$O$_4$ NCS

The lattice parameters for noticeable X-ray diffraction peak (200) plane of cubic structured CaFe$_2$O$_4$ in LDPE/ CaFe$_2$O$_4$ (10-50 mol%) polymer NCS were assessed utilizing standard relations and found to be, $a = b = c = 4.183$ Å. The crystallite size of LDPE/ CaFe$_2$O$_4$ in (200) plane is increased from 32 nm to 36 nm in LDPE/ CaFe$_2$O$_4$ NCS. The Increment in crystallite sizes of CaFe$_2$O$_4$ in LDPE/ CaFe$_2$O$_4$ NCS shows that the crystallinity of CaFe$_2$O$_4$ is exasperates by the absorption of sub atomic chains Low density polyethylene (LDPE) nanocomposites (NCS) on the surface of CaFe$_2$O$_4$. [10]-[12]

The typical crystallite sizes of LDPE/ CaFe$_2$O$_4$ (10– 50%) hybrid NCS for noticeable peaks (200) are calculated by Scherer’s formula.

\[ D = \frac{0.9 \lambda}{\beta \cos \theta} \quad \text{(1)} \]
Where; D common crystallite size and b full-width at half-maxima of the peaks.

Table.1 The estimated average crystallite size, LDPE/ CaFe$_2$O$_4$ (10-50 wt%) NCs.

<table>
<thead>
<tr>
<th>B. CaFe$_2$O$_4$ %</th>
<th>C. Crystallite size</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>D. Scherrer’s method</td>
</tr>
<tr>
<td></td>
<td>F. Plots</td>
</tr>
<tr>
<td>G. 10</td>
<td>H. 32</td>
</tr>
<tr>
<td>J. 20</td>
<td>K. 36</td>
</tr>
<tr>
<td>M. 30</td>
<td>N. 28</td>
</tr>
<tr>
<td>P. 40</td>
<td>Q. 34</td>
</tr>
<tr>
<td>S. 50</td>
<td>T. 28</td>
</tr>
</tbody>
</table>

B. Structural and morphological properties

The surface morphological studies of the present polymer nano compositions are shown Figure 3. Surface morphology of the pure LDPE indicates granular morphology with pores in the middle of spectrograms, Figure 3(b&f) indicates morphology of prepared nanocomposites, LDPE/ CaFe$_2$O$_4$ (10-50 wt%) NCs. For further increment of CaFe$_2$O$_4$ (10-50 wt%) NCs, LDPE/ CaFe$_2$O$_4$ (10-50 wt%) polymer matrix.

The TEM measurements can offer more information on the morphology and structure of the of LDPE/ CaFe$_2$O$_4$ (10-50 wt %) NPs shown in Figure 4(a-b), in LDPE/ CaFe$_2$O$_4$ (50 wt %) hybrid NCs, are presented in Figure. 4(a, b) and it shows that the synthesized LDPE/ CaFe$_2$O$_4$ (50 wt %) NCs size was in the range 30 to 34 nm in diameter. From TEM results LDPE/ CaFe$_2$O$_4$ 50 wt % NCs from these results are in good arrangement to the obtained results from the XRD pattern [10]-[14].Fig.4 give the detail information of structural parameter elsewhere.

Figure 4. TEM Images of (a-b) LDPE/ CaFe$_2$O$_4$ (50 wt %) nanocomposites

C. Fourier transforms infrared (FTIR) spectroscopy

The FTIR spectrum of the pure and doped Nano calcium ferrite composites is as shown in Fig.5. The spectra of the pure and doped Nano ferrite describe the vibrational nature of ions which are available. FTIR spectra give very useful in the investigation of the nature of the chemical structure (Bond) formed and their vibrations of various types of materials such as polymers, glasses and ferrites etc.

Figure 5. FTIR Spectra of LDPE/ CaFe$_2$O$_4$ (10-50) NCs.

The FTIR spectrum of the pure and doped Nano calcium ferrite composites is as shown in Fig.5. The spectra of the pure and doped Nano ferrite describe the vibrational nature of ions which are available. FTIR spectra give very useful in the investigation of the nature of the chemical structure (Bond) formed and their vibrations of various types of materials such as polymers, glasses and ferrites etc.
Low-Density Polyethylene/Calcium Ferrite Nanocomposites Films Prepared for Structural, Morphological, Mechanical and DC Conductivity Characterization

D. DC Conductivity

Figure 6. DC Conductivity studies of pure LDPE and LDPE/ CaFe$_2$O$_4$ (10-50) NCs.

Figure 6 shows the temperature dependent DC conductivity of pure LDPE and LDPE/ CaFe$_2$O$_4$ (10-50) NCs. Conductivity of LDPE/ CaFe$_2$O$_4$ NCs remains same up to the 80°then onwards there is the increase in the conductivity at higher temperatures [13]-[17]. Maximum conductivity showed for LDPE/ CaFe$_2$O$_4$ (30 wt %) samples may be due to the chain length that can be confirmed from IR graph. Increased conductivity may be due to increased charge polarization.

E. Mechanical Properties

LDPE/ CaFe$_2$O$_4$ polymer films is competent to raise the open volume among the polymeric chains. In doing so, the relieve of progress of polymeric chains with admiration to each other is spectacularly enhanced. In packaging, a plasticizer is a substance further to materials to impart flexibility, workability, and elongation. The plasticizer to the film is to overcome the film brittleness caused by extensive intermolecular force [15]-[19]. The prepared new ductile materials, The below graphs shows Strain –Strain relation of LDPE/ CaFe$_2$O$_4$ with different ratios (10-50) NCs.

Table 2 Various values of mechanical parameter of LDPE/ CaFe$_2$O$_4$ (10,30 & 50 wt%) NCs.

<table>
<thead>
<tr>
<th>Name of sample</th>
<th>Stress (MPa)</th>
<th>Strain</th>
<th>Y (MPa)</th>
<th>Poission ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>LDPE/ CaFe$_2$O$_4$ 10%</td>
<td>8.10</td>
<td>0.24</td>
<td>9.42</td>
<td>0.14</td>
</tr>
<tr>
<td>LDPE/ CaFe$_2$O$_4$ 30%</td>
<td>10.2</td>
<td>0.14</td>
<td>10.2</td>
<td>0.148</td>
</tr>
<tr>
<td>LDPE/ CaFe$_2$O$_4$ 50%</td>
<td>13.2</td>
<td>0.10</td>
<td>13.5</td>
<td>0.142</td>
</tr>
</tbody>
</table>

IV. CONCLUSION

In summary we have successfully prepared the LDPE/ CaFe2O4 NCs by solution casting polymerization route. The PXRD patterns confirm the amorphous nature of the pure LDPE and cubic phase of CaFe2O4 nanoparticles. Morphology of the pure LDPE and composites were studied in detail. To sum up all the results indicate that the present LDPE/ CaFe$_2$O$_4$ hybrids NCs are potential materials which can be used in the fabrication of optoelectronic devices. Surface morphology of the pure LDPE indicates granular morphology with pores in the middle of spectrograms LDPE/ CaFe$_2$O$_4$ (50 wt %) NCs size was in the range 30 to 34 nm in diameter. From TEM results LDPE/ CaFe$_2$O$_4$ 50 wt % NCs from these results are in good agreement to the obtained results from the XRD pattern. Conductivity of LDPE/ CaFe$_2$O$_4$NCs remains same up to the 80°then onwards there is the increase in the conductivity at higher temperatures.
Maximum conductivity showed for LDPE/ CaFe$_2$O$_4$ (30 wt %). Stress-Strain graphs show the strength and stability and smoothness/hardness of the synthesized nano films. Beyond the 14 Mpa Stress all the samples showing the sudden breakage stage. From the obtained measurements and data, the synthesized hybrid nano composites may be used in electronic device applications such Parcel carriage Bags, designing of automobiles such as in aerospace parts due to its lights weight, as LED devices, solar cells and optical devices.

REFERENCES


AUTHORS PROFILE

Nayeemuddin. Assistant Professor and Research Scholer, Department of Mechanical Engineering,Khaja Banda Nawaz College of Engineering Kalaburagi, Karnataka. India. He completed M-Tech in CAD/CAM and Pursuing PhD at Visvesvaraya Technological University, Belgaum, under the guidance of Dr. S A M N Quadri. His area of research is polymer Nano composite materials. He is having 6 years of experience in Automobile industries worked as Assistant Works Manager cum trainer and also having 8 years of teaching experience.