IJCRT.ORG ISSN : 2320-2882

INTERNATIONAL JOURNAL OF CREATIVE RESEARCH THOUGHTS (IJCRT)

An International Open Access, Peer-reviewed, Refereed Journal

Synthesis and Multitechnique Characterization of Amino-Functionalized PMMA/$BaCO_3$ Nanocomposites

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Abstract

In this study, amino-functionalized polymethyl methacrylate (PMMA) and its Barium carbonate ($BaCO_3$) nanoparticle-reinforced nanocomposites were synthesized and characterized. PMMA was chemically modified using hydrazinium sulfate followed by incorporation of $BaCO_3$ nanoparticles at different loadings (1, 2, and 4 wt%). The prepared materials were processed into polymeric films and investigated using FT-IR, UV–Visible spectroscopy, and scanning electron microscopy (SEM). The study demonstrates that amino functionalization combined with controlled $BaCO_3$ loading provides an effective approach for modifying the structural, optical, and morphological characteristics of PMMA-based nanocomposites.

Keywords: PMMA; $BaCO_3$ nanoparticles; Amino-functionalization; Polymer nanocomposite; FT-IR; UV–Visible spectroscopy; SEM.

Introduction

Polymer nanocomposites have gained considerable attention because the incorporation of inorganic nanoparticles can effectively modify the structural, optical, and morphological properties of polymer matrices [1]. Polymethyl methacrylate (PMMA) is widely used as a polymer matrix due to its good optical transparency, chemical stability, and ease of processing [2]. However, chemical functionalization of PMMA provides an additional approach for tailoring its interaction with inorganic nanoparticles and improving the functionality of the resulting composite materials [3].

Barium carbonate ($BaCO_3$) is an inorganic material with useful chemical and physical characteristics and has attracted interest for applications in functional composite systems [4,5]. Incorporation of $BaCO_3$

nanoparticles into a polymer matrix can influence the molecular interactions, optical response, and surface morphology of the composite, with the extent of these changes depending strongly on nanoparticle loading and dispersion [6].

In the present work, PMMA was chemically functionalized using hydrazinium sulfate and subsequently incorporated with different concentrations of $BaCO_3$ nanoparticles (1, 2, and 4 wt%). The prepared nanocomposite films were characterized using FT-IR, UV–Visible spectroscopy, and scanning electron microscopy (SEM) to investigate their structural, optical, and morphological characteristics. The study provides insight into the effect of controlled $BaCO_3$ nanoparticle incorporation on functionalized PMMA and demonstrates a simple approach for developing modified PMMA-based nanocomposite materials.

MATERIALS AND METHODS

Chemicals

Polymer Poly (Methyl Methacrylate) (PMMA) was purchased from Alfa Aesar, U.S. $BaCl_2$, Na_2HPO_4 were obtained from Merck, India. NaOH and Toluene were obtained from Fisher Scientific, India. Hydrazinium sulphate, Isopropanol, Distilled. Water were obtained from BDH Chemicals Division - Glaxo Laboratories, Alpha Chemicals, Nigra Lab Chemicals India respectively. All Chemicals are kept in cool and dry place and were used as such received.

SYNTHESIS

Preparation of nanoparticles of $BaCO_3$

The Nano-size $BaCO_3$ was synthesized by *in situ* deposition technique which was reported in previous study [7]. 24.4 gm (0.1mol) $BaCl_2$ was dissolved in 100 ml of double distilled water. PEG, 24.8 gm (0.0062 M) was also dissolved into 100 ml hot distilled water then complex of $BaCl_2$ and PEG was prepared by proper mixing on mild heating (Step-1 Solution) [8]. Another solution of NH_4HCO_3 (7.9 gm, 0.1 mol) was prepared by dissolving in 100 ml of double distilled water (Step-2) [9]. The Step 2 solution was added in (Step-1 solution) by stirring gently for 12 hours as shown in Figure 1. These solutions were kept overnight and finally the nanoparticles were filtered, washed thoroughly, with double distilled water, till freed from PEG traces, and dried at 100^0C for 2 hours [10].

Figure 1: Images synthesis of nanoparticles.

Preparation of buffer Solution

Buffer solution of pH 11.5 was prepared by adding 22.2 ml of 0.1 M Sodium hydroxide (NaOH) in 100 ml of 0.05M Di-Sodium Hydrogen Phosphate (Na_2HPO_4) solution. The pH of prepared buffer solution was tested by pH- meter and kept in glass bottle at cool place [11].

Preparation of Amino Group Containing Compound Solution

1gm of hydrazinium sulphate was dissolved in 100ml of buffer solution and stored for half hour at 50^{o}C. After that solution was filtered with whatman filter paper and stored in cool place and marked as Solution A [12]

Functionalization of Poly (Methyl Methacrylate) in solution form

In this method 3gm Poly (Methyl Methacrylate) was dissolved in 10 ml toluene at 50^{o}C. 50gm of solution A was added drop wise to dissolved PMMA followed by continuous stirring on magnetic stirrer [13]. The mixture of Solution A and PMMA was stirred for 1 hour at 70^{o}C. After one hour the PMMA was washed 4-5 times by distilled water to remove excess of solution A and dried at 110^{o}C with named as PMFN1 [14].

Synthesis of Functionalized-PMMA/Nanoparticles of $BaCO_3$

The polymer nanocomposites were synthesized in the variation of 1%, 2% and 4% by wt. addition of nanoparticles of $BaCO_3$ for each functionalized polymer. For this, 0.300 gm PMFN1 was dissolve in 3ml of toluene in a 25ml round bottom flask followed by stirring for 30 min on magnetic stirrer. For 1%wt. nanoparticles of $BaCO_3$ of functionalized polymer, 0.005gm nanoparticles of $BaCO_3$ was dissolved in 3ml toluene in another 25ml round bottom flask and stirred for 30 min to prepare suspension solution of nanoparticles of $BaCO_3$. After 30 min, the suspension solution of nanoparticles of $BaCO_3$ was added dropwise in functionalized polymer solution followed by continuous stirring. The mixture of nanoparticles of $BaCO_3$ and functionalized polymer stirred till all the toluene get evaporate and the viscous solution were formed. This viscous solution was cast on glass plate, care must be taken to taken to obtain uniform thickness and dried at 30^{o}C-35^{o}C to evaporate excess solution in the film. The prepared nanocomposites removed by paper cutter from a glass plate and stored for characterization.

For 2% and 4% loaded nanoparticles of $BaCO_3$ of functionalized polymer, the 0.010gm and 0.020gm nanoparticles of $BaCO_3$ was taken respectively and followed the same process for synthesis of polymer nanocomposites as shown in Table 1.

Table 1: The Data for the synthesis for all functionalized polymer nanocomposites.

Sr.No.	Sample code	Fun-PMMA Code	Fun-PMMA (gm)	NP_s of BaCO3 (gm)	NP_s of BaCO3 (wt.%)
1	TS1A	PMFN1	0.300	0.005	1%
2	TS1B	PMFN1	0.300	0.010	2%
3	TS1C	PMFN1	0.300	0.020	4%

RESULTS AND DISCUSSION

Physical appearance

Physical appearance of functionalized polymers and functionalized polymer nanocomposites slightly varies due to different and added loadings of nanoparticles of $BaCO_3$. All functionalized polymers and nanocomposites are very hard in nature, transparent in colour and light in weight [15,16].

Solubility

The solubility of thermoplastic polymers depends on the chemical nature of both the polymer and solvent, particularly polarity, molecular weight, branching, crystallinity, and cross-linking. The principle of "like dissolves like" generally applies, PMMA is soluble in several organic solvents, including aromatic solvents such as toluene [17,18,19].

Observation

FT-IR SPECTRA

The FTIR spectra of the 1%, 2%, and 4% $BaCO_3$ nanoparticle–hydrazinium sulphate/PMMA composites display absorption bands at 3565, 2998–2855, 1723, 1651, 1480–1437, 1388, 1269, 1239, 1188, 1141, 912, 843, and 750 cm^{-1} [20,21]. The bands at 3565 and 1651 cm^{-1} are associated with N–H vibrations, while 2998–2855 cm^{-1} corresponds to C–H stretching [22,23]. The strong 1723 cm^{-1} band represents C=O stretching, and the 1480–1388 cm^{-1} region is assigned to aliphatic CH_2/CH_3 bending. Bands at 1269–1188 cm^{-1} arise from ester and C–N vibrations, whereas 1141, 912, 843, and 750 cm^{-1} are related

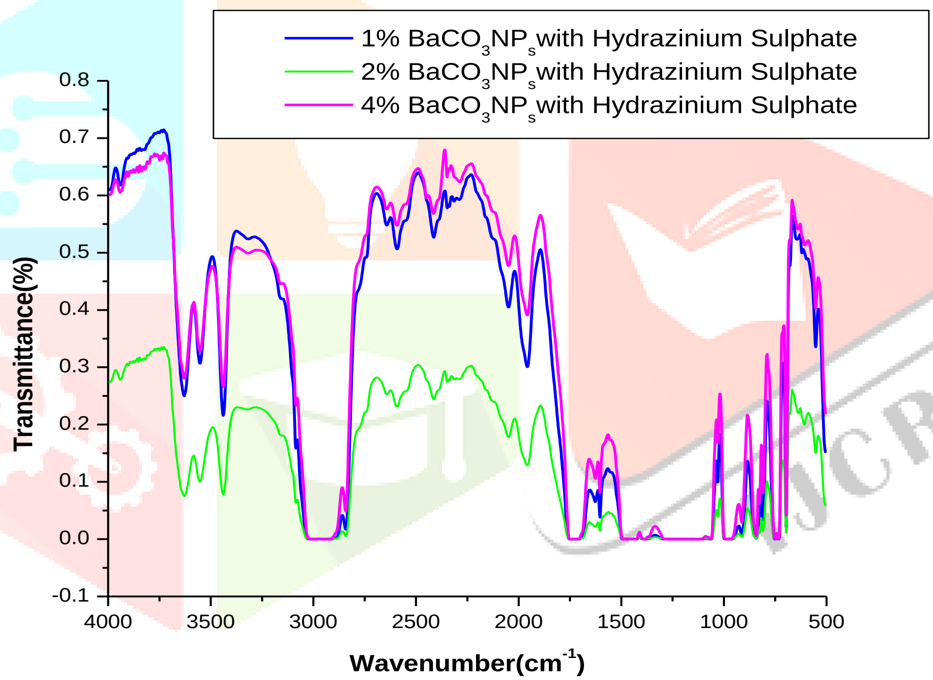

Figure 2: FT-IR of sample (TS1-A), (TS1-B) and (TS1-C)

to S=O, N–H wagging, and S–O vibrations. The characteristic CO_3^{2-} and SO_4^{2-} bands confirm the presence of $BaCO_3$ and hydrazinium sulphate [24,25,26]. Across the three compositions, the principal bands remain evident with intensity variations, indicating successful incorporation of $BaCO_3$ and interfacial interaction without significant alteration of the PMMA backbone [27].

OPTICAL STUDY OF SAMPLE (TS1A, TS1B, TS1C)

The UV–Visible analysis shows that increasing $BaCO_3$ concentration significantly affects the optical absorption of the PMMA nanocomposites [28]. The 1% sample shows an absorbance of about 0.25 near 340 nm, while the 2% sample exhibits higher absorption with a slight shift to around 350 nm [29]. The 4% sample shows a pronounced absorption peak above 3.0, followed by a decrease to about 0.75 near 350 nm as shown in Figure 3 [30]. Overall, the results indicate enhanced optical absorption with increasing $BaCO_3$

content, confirming its influence on the optical behavior of the PMMA matrix [31].

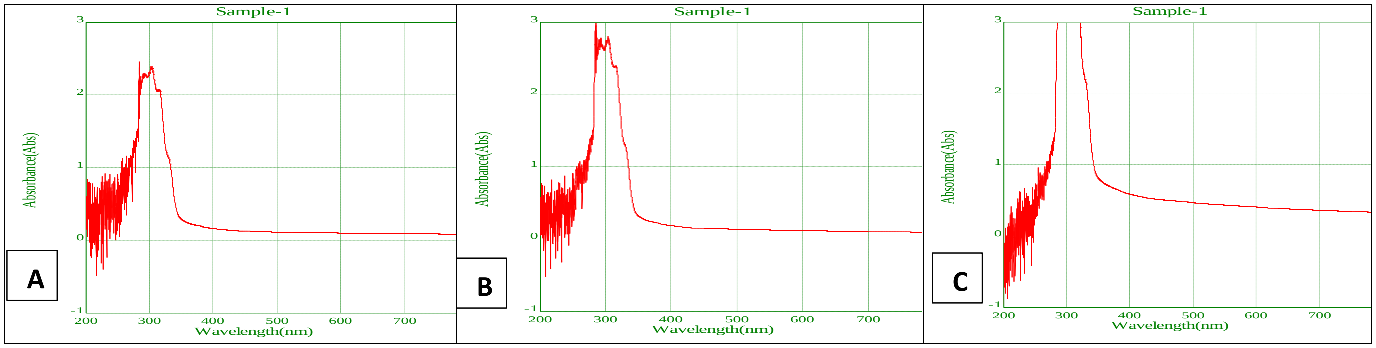

Figure 3: UV of sample (TS1-A), (TS1-B) and (TS1-C)

MORPHOLOGY

SEM Analysis

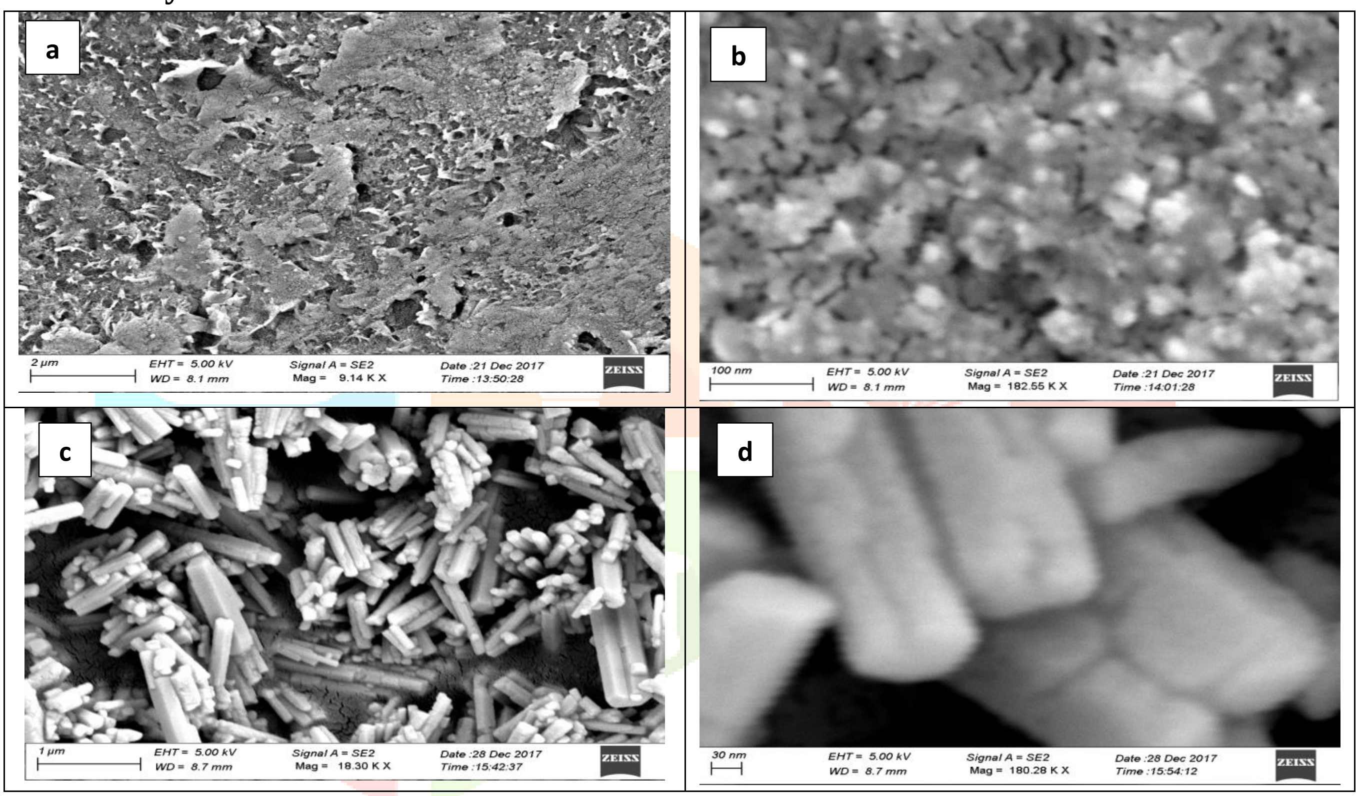

Figure 4. (a), (b), (c)and (d) SEM images of (TS1-A), (TS1-C), and NP$_s$ of $BaCO_3$

In Figure 4(a), the composite exhibits a relatively uniform and continuous surface with a thin-layer morphology at 9.14 kX magnification, indicating proper formation of the polymer matrix [32]. The higher-magnification image in Figure 4(b) (182.55 kX) clearly reveals the incorporation of $BaCO_3$ nanoparticles into the polymer matrix [33]. The nanoparticles are uniformly distributed and well dispersed, suggesting good interaction and compatibility with the polymer. The SEM micrographs of the $BaCO_3$ nanoparticles in Figure 4(c) and Figure 4(d) show clearly distinguishable particles with a predominantly rod-like morphology. The higher-resolution image provides a clearer view of the particle shape and distribution, with relatively limited agglomeration [34]. Overall, the SEM results confirm the successful formation of the composite and the effective incorporation of rod-shaped $BaCO_3$ nanoparticles into the polymer matrix [35]. Their good dispersion indicates strong matrix–particle interaction, which may contribute to the improved structural and functional properties of the nanocomposite [36].

Conclusion

Functionalized PMMA/$BaCO_3$ nanocomposites were successfully prepared with varying nanoparticle loadings. FTIR-ATR confirmed polymer functionalization, while UV–Visible analysis demonstrated concentration-dependent optical absorption and improved UV attenuation [37]. SEM observations indicated good nanoparticle dispersion and limited aggregation [38]. These combined properties suggest potential applications in UV-shielding films, protective coatings, optical materials, and packaging, although further mechanical and thermal studies are required to validate their practical performance [39,40].

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