

Study of Thermal Parameter of Polyaniline-Pbs Nanocomposite Using DTA Technique

JB Bhaishwar^{1*}, DP Meghe¹ and SP Dongre³

¹Department of Physics, Nagpur Institute of Technology, Nagpur, India

²Department of Physics, Bhalerao Science College Saoner, Saoner, India

Abstract

DTA thermal analysis indicated that the polyaniline powder had discernible moisture content. This phenomenon was in agreement with the TGA results. Moreover, in the first run of DTA thermal analysis, an exothermic peak at 150°C-310°C was found. This peak was due to the chain cross linking, resulting from a coupling of two neighboring -N=Q=N-groups to give two -NH-B-NH groups through a link of the N with its neighboring Quindío ring. Thus, on the basis of thermal profile of these materials, we can say that among all composite material, the PANI/PbS composite materials, cross-linking or oxidative reaction starts at higher temperature, which indicates that the thermal stability of PANI/PbS nanocomposites is higher.

Keywords: DTA • Thermal analysis • Polyaniline • PBS • Nano-composite

Introduction

Polymer composites are broadly utilized in car, aviation, development, and electronic applications [1]. They work on mechanical, electrical, warm and other actual properties. The Nano-composites are another class of materials that gives preferable properties over their micro-composite partners [2]. A little expansion of nanoparticles fundamentally works on various properties without forfeiting the light weight of polymer grids. The nano-composites overall allude to composites in which no less than one stage has aspect on the request for a couple of nanometers. They can be created with the utilized of three distinct sorts of nanoparticles. The principal kind of nanoparticles just has one aspect in the nanometre scale. They have a platelet-like construction [3]. Dirt is a genuine illustration of this kind of nanoparticles. The second kind of nanoparticles has two components of the nanoparticles in the nanometre scale. Nanotubes and nanofibers have a place with this gathering [4-6]. The third kind of nanoparticles has each of the three aspects in the nanometre scale, for instance, round silica particles. An assortment of this kind of particles is exceptionally retentive particles. While the element of the molecule might be in the request for microns, the pore sizes are in the request for nanometers [7,8].

The filling of nanoscopic metals into polymer grids addresses a choice to course and adjustment issues. For reasonable uses of nanoparticles, polymers are fundamentally involved as filling stage since they might have various attributes: They can be an electrical

and warm encasing or conveyor [9]. Polymers could have a hydrophobic or hydrophilic nature and can be precisely hard plastic or versatile elastic, etc. Eventually, polymer filling is the simplest and most reasonable way for nanostructures metal adjustment, treatment, and application [10]. This has fuelled examination concerning the planning of metal-polymer nanocomposites. These composites most usually appear as meager polymer movies or powders, as this is typically the easiest construction to plan, and furthermore great for taking advantage of the ideal properties. The Preparation strategies of polymer metal nanocomposite can be named *in situ* and *ex situ* techniques. In the *in situ* strategies the monomer is polymerized, with metal particles presented previously or after polymerization [11,12]. Then, at that point, the metal particles in the polymer framework are diminished artificially, thermally or by UV illumination, to shape nanoparticles [13]. In the *ex situ* process, the metal nanoparticles are combined first, and their surface is naturally passivized. In the present paper Polyaniline/pbs nanocomposite prepared and it get analysis with DTA for thermal parameter and their Stability.

Materials and Methods

Chemical synthesis of polyaniline Chemical synthesis of pure aniline using APS as oxidant and H₂SO₄. Pure aniline dissolved in 100 ml distilled water with H₂SO₄ added under magnetic stirring for 2 hours. The solution of ammonium per sulphate in sulphuric acid was then added drop-wise in the solution of aniline. The precipitate of polyaniline obtained with dark green clouded [14]. The precipitate

*Address for Correspondence: JB Bhaishwar, Department of Physics, Nagpur Institute of Technology, Nagpur, India, Tel: 8007673735; E-mail: jitendrabhaishwar@gmail.com

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Received: 12 October, 2022, Manuscript No. JME-22-77049; Editor assigned: 17 October, 2022, PreQC No. JME-22-77049 (PQ); Reviewed: 01 November, 2022, QC No. JME-22-77049; Revised: 10 January, 2023, Manuscript No. JME-22-77049 (R); Published: 17 January, 2023, DOI: 10.37421/2169-0022.2023.12.624

washed with water. A PANI precipitate was dried under at 50°C-100°C for more than 8 hours. Synthesis of PANI/PbS nanocomposites The same synthesis process was adapted for preparation of PANI/PbS Nano-composite at different weight ratio of PbS nanoparticle. The PANI nanocomposite was chemically characterized by infrared spectroscopy, XRD and TGA.

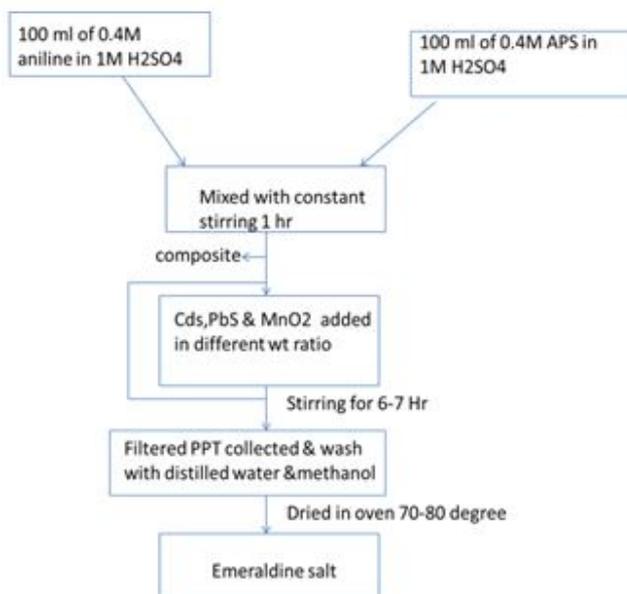


Figure 1. Chemical synthesis of polyaniline.

SR. NO	Material	Crystalline size particle(nm)	d'spacing (Å)	2θ (°C)
1	PURE PANI	0.71	3.52	25.27
2	PURE PbS	0.95	3.21	27.77
3	5% PANI/PbS	1.048	3.23	27.56
4	10% PANI/PbS	1.42	3.24	27.47
5	15% PANI/PbS	1.23	3.012	29.65
6	20% PANI/PbS	1.437	3.009	29.68
7	25% PANI/PbS	1.438	3.0096	29.65

Table 1. Materials and crystalline size particle.

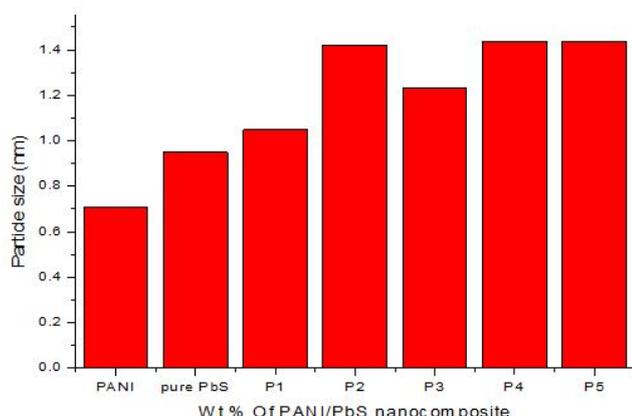


Figure 2. Crystalline size of crystalline particle for pure PANI, Pure PbS and different wt % of PANI/PbS nanocomposite.

Characterizations of nano-composites

XRD with Philips PW-3071, using Cu-K α radiation of wavelength 1.544 Å, with scanning rate of 20/min. at 45 KV and 40 mA. Fourier Transform Infra-Red (FTIR) spectroscopy (Model: Perkin Elmer 200) with frequency of 400 cm⁻¹-4000 cm⁻¹. Thermal analysis of sample recorded by Perkin-Elmer Diamond TGA/DTA in argon atmosphere at a heating rate of 10°C/min.

XRD characterization of pure PANI and PANI/PbS nanocomposite:

The XRD pattern of pure PANI, Pure PbS and different wt % of PANI/PbS nanocomposite are shown in the Figures 1 and 2. The particle size of crystalline particle of pure PANI and the nanocomposites are calculated by using Debye-Scherrer formula. $D=0.94\lambda/(\beta \cos\theta)$, where D is the average crystallite size (in nm), k is the shape factor which is often assigned a value of 0.94, λ is the wavelength of Cu K α radiation (1.5418 Å), β is the full width at half maximum of the diffraction peak taking into consideration the correction due to instrumental broadening (0.09°).

Crystalline size of crystalline particle for pure PANI, Pure PbS and different wt % of PANI/PbS nano-composite are given in the Table 1 and bar graph 3.3. From the bar graph of XRD peak it is clear that as the wt % (5-20) PbS nanoparticle increases in the PANI matrix the degree of crystallites of nanocomposite also increases. Pure PANI shows crystalline reflection at specific angle in XRD pattern and amorphous at diffused background, as a result it reveals the polycrystalline structure.

Ultraviolet and Visible (UV-Vis) spectroscopy of PANI/PbS nanocomposite:

The maximum absorption wavelength of pure PbS and PANI/PbS Nano composite are shown in the pie graph (Figures 3 and 4). In pure PbS the absorption wavelength is obtaining at 263, 277 and 768 nm. The interesting feature observed in PANI/PbS Nano composite is the presents of absorption bands at 400 to 500 nm. Because this band is not present in pure PANI as well as pure PbS this indicated that when PbS nanoparticle interacts with PANI some structural change is occurred. The presents of this band in the nano composite give the photoluminous characteristics. The polaron- π^* transition band at 320 to 385 nm becomes broader and shows the red shift. This implies that the doping state of the nanocomposites has been improved. Such phenomena can be attributed to the existence of greater number of charges on the polymer backbone by introducing nanocrystalline PbS into the polymer matrix [16].

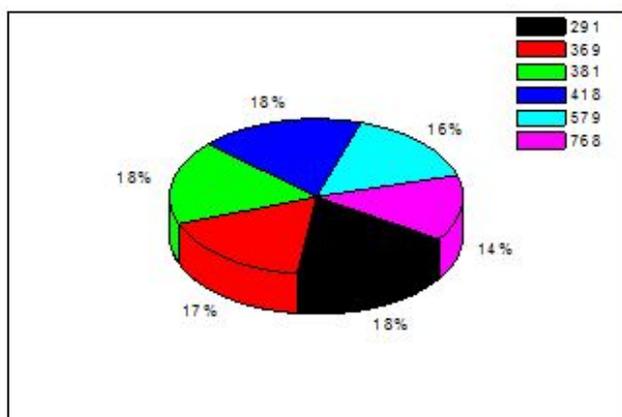


Figure 3. The maximum absorption wavelength of pure PbS and PANI/PbS nanocomposite.

Transmission electron microscopy of PANI/PbS nanocomposite

The morphology of PANI-PbS nano composites is found out based on the TEM images. The TEM image of H₂SO₄ doped PANI is shown in Figure 4. Figures 4 and 5 depicts the TEM images of pure PbS and doped H₂SO₄ PANI/PbS.

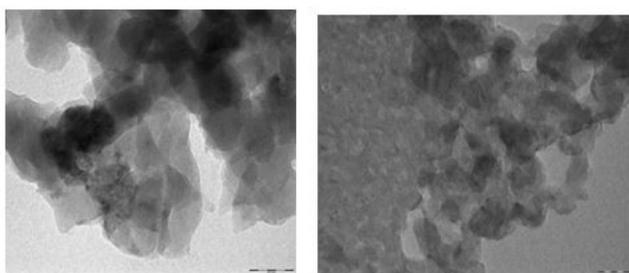


Figure 4. The TEM image of H₂SO₄ doped PANI.

TEM micrograph (10%) PANI/PbS nanocomposite shows that particles were aggregated into a big structure, although the particles were in contact with each other. Most of the particles were similar in size and have irregular rounded shapes. It is in the 100 nm scale. As the weight % PbS nanoparticle increased in the polymer than particle size decreased. The nanocomposite becomes more ordered structure, hence electrical conductivity is also increased. This is also clear by XRD and UV spectra. The average diameter of nanoparticle is 12 nm ranges.

Results and Discussion

DTA analysis of pure PANI and PANI/PbS nano composite

Figure 5 shows DTA thermogram of PANI/PbS (5% to 25%) nanocomposite which indicated only endothermic peaks at about

230°C to 245°C due to the evaporation of water molecules trapped inside the composite or bound to the polymer backbone. whereas the change above 350°C may be assigned due to the degradation of composite. The decreased onset value of temperatures from 284°C (pure PANI) to 242.59, 244.15, 233.06, 227.55 and 237.25°C for different wt ratio of (5%-25%) PANI/PbS nanocomposite indicated that the thermal stability of nanocomposite is good than that of pure PANI which could be attributed to the retardation effect of nanostructures PbS as barriers for the degradation of PANI [15-17]. In DTA of PANI the glass transition was not observable, because the glass transition is buried in the peak due to the removal of water and it does not exhibit hysteresis. The exothermic transition observed at 99°-160°C is believed not to be T_g. Instead it would be attributed to a series of chemical reactions. The decreased peak temperatures of PANI/PbS Nano composite, further demonstrate the ordered polymer structure as well as good interfacial interactions between the metal oxide and the polymer matrix. The DSC results of composite materials are also found in good agreement with TGA results which indicates that all the nanocomposites shows minimum value of onset temperature as compare to the pure PANI. Table 2 given below gives the information of thermal parameter of Polyaniline nanocomposite with different wt percentages of PbS. Some change in the melting temperature and enthalpy in PANI/PbS (5%-25%) nanocomposite indicated miscibility with PANI matrix.

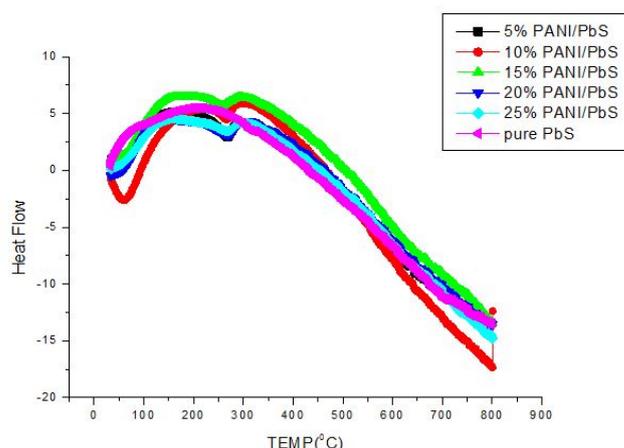


Figure 5. DTA analysis of pure PbS and PANI/PbS (5%-25%) nano composite.

Sr. No	Material	Melting temp (°C)	Onset temp (°C)	Enthalpy change (J/g)	Specific heat (Δ Cp (J/g°C))	Peak area
1	Pure PANI	-	284	-	12.27	-
2	5% PANI/PbS	268.31	242.59	47.37	9.98	264.377
3	10% PANI/PbS	267.88	244.15	34.46	6.52	167.613

4	15% PANI/PbS	256.81	233.06	30.4788	6.479	123.622
5	20% PANI/PbS	267.13	227.55	60.699	16.192	308.7
6	25% PANI/PbS	266.42	237.25	36.8611	9.405	152.234

Table 2. Thermal parameter of pure PANI and PANI/PbS Nano composite.

Conclusion

DTA thermal analysis indicated that the polyaniline powder had discernible moisture content. This phenomenon was in agreement with the TGA results. Moreover, in the first run of DTA thermal analysis, an exothermic peak at 150°C-310°C was found. This peak was due to the chain cross linking, resulting from a coupling of two neighboring -N=Q=N- groups to give two -NH-B-NH groups through a link of the N with its neighboring Quindío ring. Thus, on the basis of thermal profile of these materials, we can say that among all composite material, the Polyaniline/CdS and PANI/PbS composite materials, cross-linking or oxidative reaction starts at higher temperature than PANI/MnO₂ composites, which indicates that the thermal stability of PANI/CdS and PANI/PbS nanocomposites is higher than oxides Nano composites. These DTA results of composite materials are also found in good agreement with TGA results.

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How to cite this article: Bhaiswar, JB, DP Meghe and SP Dongre. "Study of Thermal Parameter of Polyaniline-Pbs Nanocomposite Using DTA Technique." *J Material Sci Eng* 12 (2023):624.