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TECHNOLOGY****EFFECT OF NANOIRON ON MECHANICAL AND THERMAL PROPERTIES OF
BLENDED NANOCOMPOSITES****G.Naveen Kumar^{*1}, Dr.Y.V.Mohana Reddy² & Dr.K.Hemachandra Reddy³**^{*1}Department of Mechanical Engineering, Institute of Aeronautical Engineering, Dindigal, Hyderabad
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Anantapuramu-515002, Andhra Pradesh, India.**ABSTRACT**

We reports on blend nanocomposites, which are two highly dissolve nanoparticles are using as chemical reduction method. Blend is prepared using epoxy and polyester in which filler as nanoiron was dispersed to evaluate mechanical and thermal properties. Response was initiated mixing of solution-1 (FeCl₃6H₂O) into solution-2 (FeCl₂4H₂O) as one, under the occurrence of ammonium to increase nanoparticles. Different weight ratios (viz.1%, 2%, 3%, 4%, 5% & 7%) of nanoiron filler particles were dispersed into the matrix to prepare different systems to assess the optimization of performance. Thermal analytical properties of Differential Scanning Calorimetry (DSC) and Thermo Gravimetric Analysis (TGA) were performed for the particular samples. The mechanical properties of tensile strength and flexural strengths were investigated on epoxy+polyester nanocomposites to assess the influence of nanoiron. SEM analysis was conducted on different fractured surfaces to study the mechanical behavior. Mechanical and Thermal analytical properties were improved due to their uniform particle dispersal and chemical bonding between nanoparticles, epoxy and polyester matrix. After incorporation these nanoparticles turn into magnetically harder into the polyester resin matrix.

KEYWORDS: Nanocomposite, Epoxy/Polyester blend, mechanical properties and thermal properties.**INTRODUCTION**

Nanocomposite materials together with conduct ferromagnetic properties were received tremendous interest due to their probable applications in electrical-magnetic shields, sensors, display devices, molecular electronics, batteries, microwave-absorbants and electrochemical non-linear optics. Moreover blend method is adding of nanoparticles physically inorganic ferromagnetic conduct with polymer, some approach such as chemical polymerization and electrochemicals have been reported to co-ordinate conducting polymer with nanomaterials and ferromagnetic properties with core shell structure. Dramatically nanocomposite dispartate to their bulk or atomic counterparts were fascinated more interest due to their exclusive physicochemical properties for a broad range of possible device applications such as high-sensitivity chemical gas or UV lasers, DNA sequence sensors and solar cells. Nano structural materials such as nanoiron had been used as fillers together in the polymeric nanocomposites to increase the thermal and mechanical properties and their metallic nanocomposite to manage the electro evidence. Inorganic nanoparticles were reinforced by polymer nanocomposites which were attracted by more interest due to their homogeneity, cost-effective, processability, weightlessness and their changeable physical properties. As a structural polymer polyester resin, have preferred as a polymer matrix in existing study due to the confirmation that the cured resins are thermosetting with a net structure possessing good resistance to the chemicals, moisture and good mechanical and thermal properties. So, their resultant composites had the sufficient applications in invention of structure materials such as electro affidavit tanks, automotive parts and marine vessels which require higher thermal properties and/or high resistance to unsympathetic environments such as strong acids or bases. In addition, the functional groups of the polymer surrounding the nanoparticles facilitate these nanocomposites as good candidates for various applications such as site-specific molecule targeting in biomedical specializations. The composite fabrication exist challenges are to attain reliable filler dispersion and to setup strong chemical bonding between the nanoparticles to the polymer matrix, which are fundamental to afford a high tensile strength due to restricted stress within the nanocomposite. The interfacial interactions between polymer matrix and fillers play a significant role in identifying quality and their properties of the nanocomposites. A weak bonding connection between the polymer matrix and fillers such as the

composites made by bare mixing will carry in artificial defects, which consequently results a poor effect on the thermal properties of the nanocomposites. Polymer matrix and fillers were introducing good reactions between them and is still a challenge for definite composite fabrication. Even though, suitable chemical treatment of the nanofiller surface by introducing proper practical groups may improve both the strength and toughness of the following composites with superior compatibility between the nanofillers and the polymer matrix and makes the nanocomposites steady in unkind environments as well. Hence, the surface functionalization of nanoparticles with blend agent or a surfactant is important not only to study the nanoparticles throughout processing but also to provide them familiar with the polymer matrix [1-3]. In this paper, the effect of element functionalization by a practical methacryloxypropyl in the polyester resin curing method and the optimal circumstances were investigated for high-quality nanocomposite fabrication. The nanocomposites reinforced polyester showed enhanced mechanical properties under scanning electron microscope study. Polyester nanocomposites reinforced by nanoiron composites which show improved thermal properties under DSC and TGA analysis. The nanocomposites containing nanoiron shows superior thermal stability [4]. After the nanoiron particles were dispersed in the polyester resin matrix the nanoparticles have found to be magnetically harder. The present paper nanoparticles are synthesised by chemical reduction process. The main aim of this paper is to explore original advance on mechanical and thermal properties of nanoiron dispersed into the polyester nanocomposites, i.e. Different weight ratios (viz.1%, 2%, 3%, 4%, 5% & 7%) of polyester nanocomposites have developed [5-9]. Therefore, superior mechanical and thermal properties were expected.

MATERIALS

Unsaturated polyester (UP) with 2% cobalt accelerator/catalyst 50% methyl-ethyl-ketone-peroxide (MEKP) and 10% DMA solution was employed in the present study. Ratio of UP/accelerator/catalyst/promoter is 100/2/2/2. The fluid resin has a density of 1.231 g/cm³ and a viscosity of 370 centipoises (cps) at room temperature. Nanoiron particles with a standard of diameter of 10-15 nm and a specific surface area of 45m²/g was functionalized and used as nanofillers for the nanocomposite fabrication. Cobalt naphthenate (CoNap) have used as a catalyst promoter (accelerator) to decompose the catalyst at room temperature. Methacryloxypropyl-trimethoxysilane(MPS) and tetrahydrofuran(THF). All the chemicals are used as-received without further treatment.

CHARACTERIZATION

Characteristics of epoxy+polyester filled nanoiron blend were studied with DSC. The solution of 5mg is added in an alumina crucible and heat is applied at the rate of 10°C/min [10-11]. The thermal characteristics of the nanoiron filled with epoxy+polyester blend have measured using both differential scanning calorimetry and thermo gravimetric analysis at a rate of 10°C/min under nitrogen flow.

PREPARATION OF NANOIRON PARTICLES

Response was initiated mixing of solution-1 (FeCl₃6H₂O) into solution-2 (FeCl₂4H₂O) to form nanoparticles. 0.1 Mole of 27.030gms of solution-1 were dissolved 1000ml de-ionised water, whereas 19.881gms of solution-2 were dissolved in 1000ml de-ionised water were used to prepare these solutions. Then solution-1 has added then to solution-2 go down by 2:1 stoichiometric ratio under vigorous stirrer. Ammonium (45ml) was added into the solution; forth with nanoiron was produced in less than one second. After reaction, the product particles were divides from the solution by a strong magnet and washed with de-ionised water. To dry the particles freeze-drying overnight was utilized [12-16].

SURFACE FUNCTIONALIZATION IRON OXIDE NANOPARTICLES

Nanoiron particles functionalization follows the procedures related to our previously reported nanoparticle method and is mentioned as follows. Fe₂O₃ nanoparticles (13.38g, 83.8mol) were added into a mixture of 4g Methacryloxypropyl and 30 ml THF. The resulting colloidal suspension was ultrasonically stirred for 1hr and precipitated by a permanent magnet at room temperature. The precipitated nanoparticles were rinsed with THF to remove unnecessary Methacryloxypropyl for successive nanocomposite fabrication and the dried nanoparticles was used for further particle characterization [17-18]. The Fe₂O₃ as-received nanoparticles or MPS functionalized Fe₂O₃ nanoparticles were spread into 30ml resin on a specific weight percentage ratio. The diffusion was carried out with water in ultrasonic bath for about 1hr. The above particle-suspended solution was then ultrasonically stirred in water ultrasonic bath. Catalyst (initiator) 2.0 wt% was added into the nanoiron particles which was stirred and degassed for 2 min. Then, 0.3 wt% promoter was added and mixed without delay.

The mixed viscous solution was poured into silicone rubber molds. The curing via free-radical bulk copolymerization or homopolymerization initiated by the catalyst was done at 85°C for 1hr under normal atmospheric circumstances and cooled down to room temperature naturally in the oven following procedures similar to those that used for our reported alumina nanoparticles filled polyester resin nanocomposites fabrication [19-22]. After this curing method a viscous liquid solution was still observed in the mold, signifying that nano filling materials have a considerable effect on the quality of fabricated nanocomposites. However, high-quality nanocomposites have formed by room temperature curing for 24hr followed by post curing at 100°C for 2hr.

FABRICATION OF NANOIRON COMPOSITES

Nanoiron was dried in the hot air oven for about 30min at 50°C and then predetermined ratio of nanoiron and matrix were taken by stoichiometric ratio in the beaker. Modified solution was stirred for about 45min manually to see that particles are to be mixed well with the matrix. Then the same mixture is set up under the presence of Mechanical stirrer and followed by ultra-sonicator for another 45min subsequently. Once the modified solution is ready then the promoter and accelerator were mixed on to the 2:2 ratio for every 100grams of polyester. Solution was again stirred for another 10min before they pour into the mould. Mould was impregnated with OHP sheets carefully to put the casting under compression. Mould allowed curing for 24hours. Then the moulds were kept in the oven for about 50min at 80°C, then the casting were removed from the mould. Samples were cut from the casting on par with ASTM standards.

TENSILE LOAD MEASUREMENTS

Tensile strength was studied using an Instron Universal Testing Machine supplied by Instron Corporation; a series-9 automated testing machine was used with a crosshead speed of 5mm/min. Testing samples were prepared in dumb-bell shapes and these dimensions are 100x20x3mm³ based on the ASTM D 638 standards. In each case, six samples were tested.

FLEXURAL LOAD MEASUREMENTS

Flexural strength and modulus were tested using an Instron Universal Testing Machine with a crosshead speed of 2mm/min. The three-point bending test system was used for all samples. In each case, six samples were tested. Authors used 50KN load cell for testing further the sample sizes and 100x20x3mm³ was cut in accordance with ASTM D 618.

SCANNING ELECTRON MICROSCOPY ANALYSIS

A JEOL JSM 840A JAPAN scanning electron microscope (SEM) was used to study the morphology of fractured surfaces of nanocomposite samples at uniform magnifications. The fractured surfaces were gold-coated initially subjecting it to SEM analysis. The scanning electron microscope of different cross-sections with different magnifications of the nanocomposites samples with reinforcement is studied.

RESULTS AND DISCUSSIONS

Measurements of variation of tensile strength as a function of temperature for epoxy and polyester blended nanoiron composites are shown in Fig.1. It was observed that tensile strength was gradually increases from 1wt.% to 4 wt.% after that it decreases. 23% of tensile strength was increased at 4wt.% when compared with 1wt.%. At 4wt.% to 7wt.% gradually decreasing the tensile strength. Authors notice two reasons for decrease in tensile strength at 4 wt.% to 7wt.%. Firstly as the nanoiron increases from certain values to a limited value it will optimized its magnitude of the performance as the nanoiron goes on increases its viscosity also increases as a result of that flowability of the modified solution, it will be very difficult as a result of that stiffness will be decreases due to addition of nanoiron composites transforms from ductile to brittle natures there by strength will be decreases. Secondly due to increased viscosity and agglomeration in this turn, strength will be reducing. Increased tensile strength is due to the addition of filler material then it will consequently increase the stiffening effect of the composites. Similar observations were noticed from the old literatures.

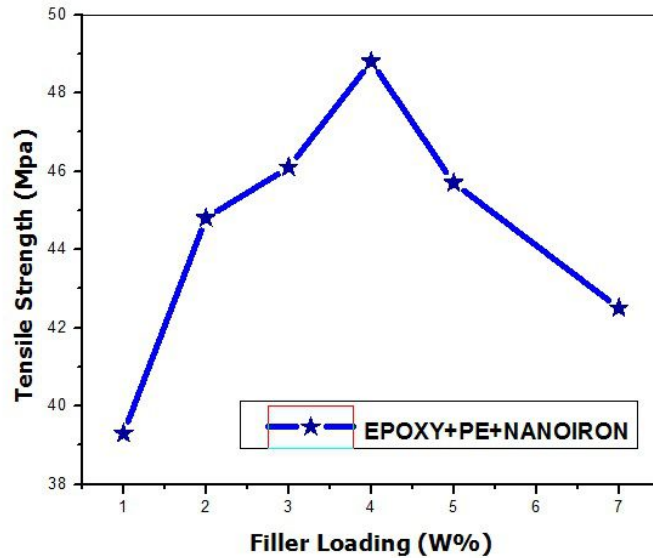


Fig 1: Variation of tensile strength as a function of filler nanoiron loading of epoxy+polyester blended nanocomposites.

Measurements of variation of flexural strength as a function of temperature for epoxy and polyester blended nanoiron composites as shown in Fig.2. It was observed that flexural strength was gradually increases from 1wt.% to 4wt.% after that it decreases. 28.9% of flexural strength was increased at 4wt.% when compared with 1wt.%. Reasons were attributed that when the filler nanoiron is going more than limited as a result of that viscosity increases consequently matrix will not flow as it offers more resistance to flow and with this property matrix cannot flow in all directions as a result of this voids will be formed.

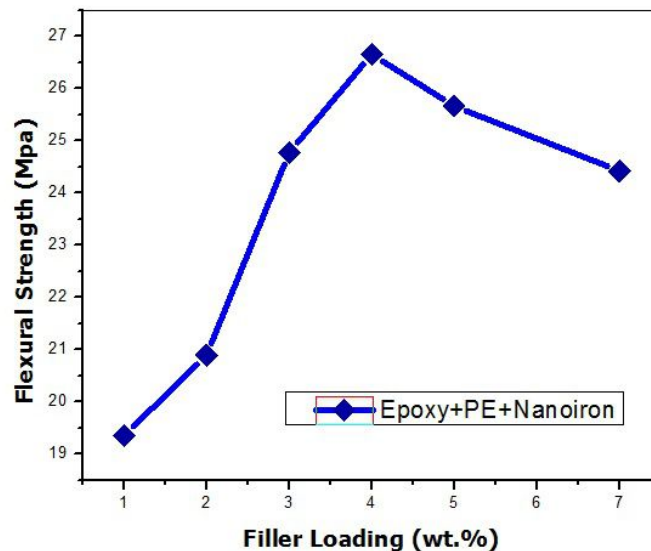


Fig 2: Variation of flexural strength as a function of filler nanoiron loading of epoxy+polyester blended nanocomposites.

The sample surfaces of epoxy and polyester filled nanoiron are shown through SEM images in Fig 3. It is learnt from the cross section of fractured surface of minimum nanoiron filler with epoxy and polyester is smooth due to brittle failure. However, on addition of nanoiron particles, crack surface becomes rough. The roughness increases as nanoiron content increases in the matrix. The fracture roughness indicates that the resistance of

propagation of crack is small and the crack has not propagated as easily as seen in epoxy and polyester. The fracture surface roughness indicates that crack propagation is large and increased the torturous path of propagating crack. This effect results in higher stress to failure and caused improved strength of nanoiron composites. Though the fracture roughness is predominant at 7 wt.% as in fig.3(f), the existence of unexfoliated aggregates, voids, etc. could have decreased the strength of nanoiron. The fracture surface of polyester with 1wt.% of nanoiron is seen at Fig 3(a). Fracture surface of polyester with 2wt.% is rougher than 1wt.% is seen at fig 3(b). As it increases as the filler loading wt.% increases as shown in fig.3. At 5wt.%, the presence of voids is noted in fig 3(e). This indicates that particles have peeled off from material as crack propagates and create void at the positions where nanoiron particles were present. From the Figs 3(a-f), it is learnt that, at 4wt. % the roughness was more predominant from the Fig 3(d) than the Fig 3(c). In the Fig 3(e) at 5wt.%, though the fracture surface is rough though, due to the existence of voids is partially visible and has decreased the strength of the material. The poor bonding strength, smooth fracture surface, voids, etc could decrease the tensile strength of the nanoiron filled epoxy and polyester nanocomposites. In polyester though the tensile strength increases up to 4%, for higher nanoparticles content, it decreases due to unexfoliated aggregates, voids, etc.. It requires further investigation of the synthetic procedure to understand the methods of improving tensile strength for higher nanoiron contents.

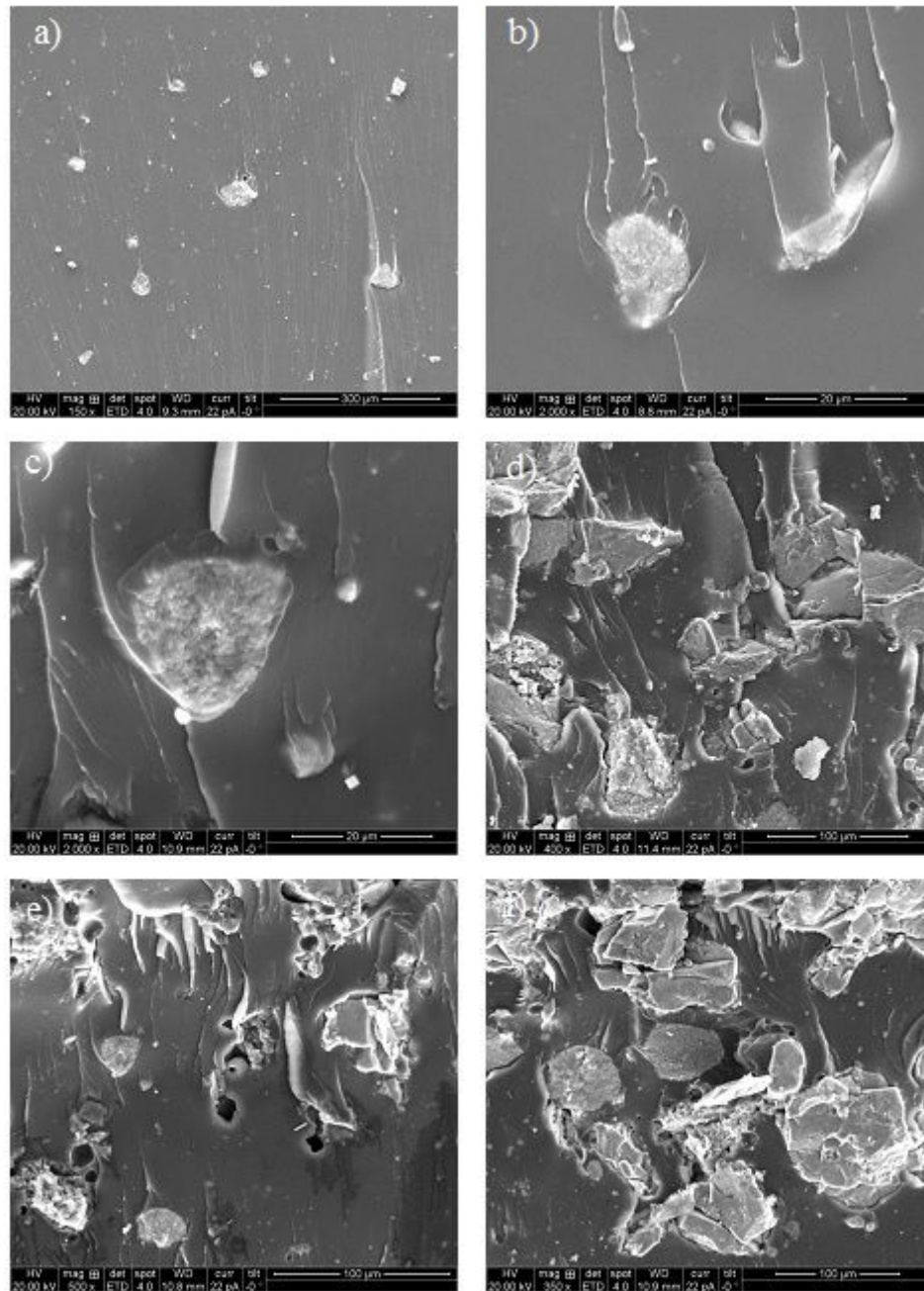


Fig.3 SEM analysis of different weight ratios a)1%, b)2%, c)3%, d)4%, e)5% & e)7%) of nanoiron composites.

Differential scanning calorimetry is used to study the behaviour of materials as a function of temperature. Fig.4 illustrates the heat flow in a sample as a function of temperature. Glass transition temperature at the first broad peak is 188°C. The second peak was observed at the 350°C is the degree of crystallinity and crystallinity was further increased to 400°C and the decomposition was completed at around 480°C. Differential scanning calorimetry used to study the behaviour of materials as a function of temperature.

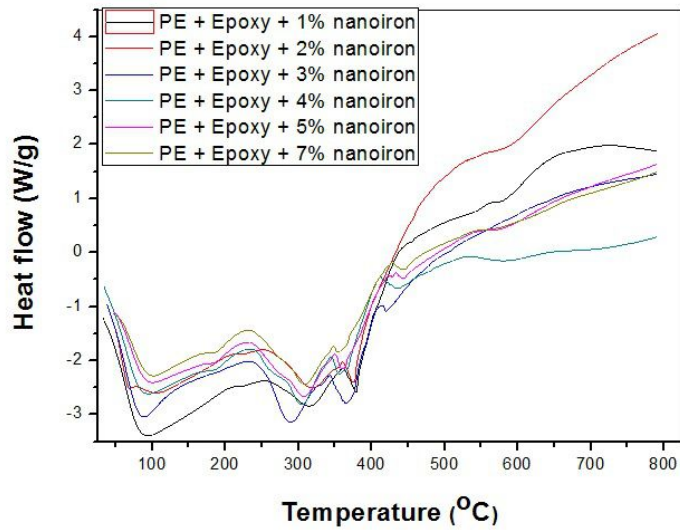


Fig 4: variation of heat flow curves as a function of temperature of blended nanoiron composites.

Fig.4 illustrates that at 1wt.% of nanoiron the glass transition temperature at the first broad peak is 188°C. The second peak was observed at the 350°C is the degree of crystallinity and crystallinity was further increased to 400°C and the decomposition was completed at around 480°C. At 2wt.% of nanoiron the glass transition temperature at the first broad peak is 194°C. The second peak was observed at the 345°C is the degree of crystallinity and crystallinity was further increased to 360°C and the decomposition was completed at around 500°C. At 3wt.% of nanoiron glass transition temperature at the first broad peak is 200°C. The second peak was observed at the 335°C is the degree of crystallinity and crystallinity was further increased to 350°C and the decomposition was completed at around 500°C. At 4wt.% nanoiron the glass transition temperature at the first broad peak is 202°C. The second peak was observed at the 333°C is the degree of crystallinity and crystallinity was further increased to 352°C and the decomposition was completed at around 500°C. At 5wt.% nanoiron glass transition temperature at the first broad peak is 175°C. The second peak was observed at the 348°C is the degree of crystallinity and crystallinity was further increased to 360°C and the decomposition was completed at around 500°C. At 7wt.% nanoiron glass transition temperature at the first broad peak is 170°C. The second peak was observed at the 344°C is the degree of crystallinity and crystallinity was further increased to 364°C and the decomposition was completed at around 490°C. Differential scanning calorimetry used to study the behaviour of materials as a function of temperature.

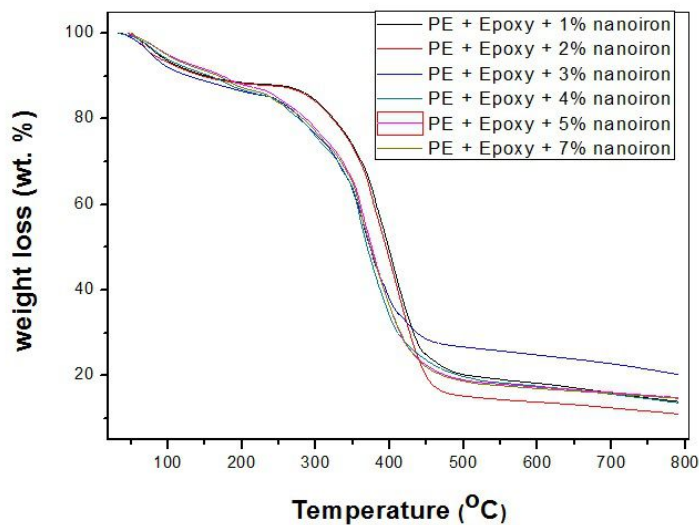


Fig 5: TGA thermographs variation of weight loss% curves as a function of temperature of blended nanoiron composites.

Fig 2 shows TGA curves of polyester filled with different weight ratios of nanoiron particles (viz. 1%, 2%, 3%, 4%, 5% & 7%). It shows the thermal stability of 1wt.% and 3wt.% filler of nanocomposites which does not show much difference between the both wt.% ratios as both the curves merged up to 320°C and it is due to dissociation of iron oxide ions. It is also learnt that due to the moisture content in the nanoiron the decomposition was started at 130°C. On addition of 4wt.% nanoiron in epoxy and polyester, the thermal stability was increased up to 325°C. The hard iron-oxide layers act as barrier for volatile degradation of polyester matrix. This effect causes such enhanced thermal properties of nanocomposites. The enhanced thermal stability of nanoiron fillers was observed even at higher temperature (>400 °C) was due to the existence of inorganic phases (SiO₂, Al₂O₃, MgO, etc) dominates the iron-nanocomposites. At 2wt.% nanoiron shows weight loss was remain constant up to 320°C. This is the clear indication, through functionalization of nanoiron particles moisture content is reduced some extent. And also thermal stability was increased to 330°C for 4wt.% whereas for 5 and 7wt.% up to 330°C and 335°C respectively. Another significant point come to light is decomposition temperature for nanoiron particles were changed dramatically. There is a marginal shift in decomposition temperature when nanoiron is added into the epoxy and polyester. The improved thermal stability is noticed for epoxy and polyester filled nanoiron variants are higher temperatures i.e 345°C than nanoiron particles.

CONCLUSIONS

The results of the study showed nanoiron particles are highly disperses were synthesized through chemical reduction method and then dispersed into epoxy and polyester. Epoxy and polyester blends were prepared by dispersing into nanoiron with different weight ratios such as 1%, 2%, 3%, 4%, 5% and 7%. Nanoparticles of iron-oxides nanocomposites can be used as ferrofluids, high-density information storage, magnetic resonance imaging, tissue-specific releasing of therapeutic agents and biomechanical applications.

Tensile strength and flexural strength were studied, nanoiron as filler for epoxy and polyester nanocomposites. It is concluded in mechanical properties that nanocomposites can be used for high strength, stiffness, and bending applications in aerospace, automobile, and marine and lightweight article applications. Overall studies indicate that the reinforced nanocomposites at 4wt% nanoiron loading are promising candidates for structural applications where high strength and stiffness is indispensable. From the SEM analysis it is learnt that addition of nanoiron impart the changes in the phase transformation from brittle to ductile nature that ultimately rule the improved performance up to optimum weight %, on other hand, beyond optimum level performance is reduced due to the formation of agglomerations and voids due to the increased viscosity of the modified polymer solution. Mechanical properties like tensile strength, flexural strength were optimized due to the same reason as mentioned above.

Thermo gravimetric laboratory analysis showed an increased thermo-stability of nanoiron particles filled with polyester nanocomposites as study of nanoparticles filled counterparts. 3°C thermal stability was increased for 4wt.% nanoiron filled composites when compared with other nanoiron wt.% filled composites. In DSC and TGA analysis it is learnt that addition of inorganic nano suspension into the polymer were played their major role in improvement of thermal stability and curing characteristics.

The present study thus bears testimony to all of these findings. Hence the present study not only discloses that nanoiron overseen through the polymer with different surface treatment promotes the performance of composites, but that unique tailored properties are improved by changing the orientation in the matrix. This present work however needs further study to develop a comprehensive evaluation of mechanical and thermal properties from exposure to nanoirons.

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