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Influence of Seashell Addition on Thermo-Mechanical Properties of Nylon 66 Polymer Matrix Composite

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Abstract: *In this work, influence of seashell particulate reinforcement on nylon 66 polymer matrix composite is investigated experimentally by determining the thermo-mechanical properties of the composite viz., Differential scanning calorimetry (DSC), Dynamic Mechanical Analysis (DMA) and Thermal Gravimetric Analysis (TGA). Seashell particulates of size 75 µm size is reinforced in the matrix of nylon 66, a thermoplastic polymer, to improve its properties by forming a polymer matrix composite. From the seashores of Marakkanam, Tamil Nadu, India, seashells were collected, which are dried in sunlight to remove the moisture and is cleaned properly to remove the sludge present. Seashell are grounded to powder through mechanical ball milling method and the required size is obtained from the sieve machine. Various proportions of seashells such as: 3, 6 and 9% by weight is added to the required amount of nylon 66, mixed and compounded in twin screw extruder and specimens of required dimension is obtained in injection moulding machine. As per ASTM standards, thermo-mechanical properties were studied and reported.*

Keywords: Sea Shell particulate, Reinforcement, Nylon 66, Differential scanning calorimetry (DSC), Dynamic Mechanical Analysis (DMA) and Thermal Gravimetric Analysis (TGA).

1. INTRODUCTION

In order to cope with the rapid development of modern science and technology, there are harsher special requirements for materials. The research of material is gradually breaking away from the track of researching by experiences and fumbling methods. It develops in the direction of material designing according to the designed properties [11]. The composite material which is made of metallic, non-metallic and polymeric material by certain processes, can retain the advantages of the original components, overcomes some shortcomings and show some new properties [2]. The emergence and development of such composite materials is a classic example of material designing. Composites are produced to optimize material properties, mechanical (mainly strength), and chemical and/or physical properties. In the later, optimization of thermal (thermal expansion/thermal conduction, specific heat, softening and melting points) as well as electrical (electrical conductivity/electrical permittivity, dielectric loss), as well as optical and acoustical properties can be performed [3]. Polymers are mostly organic compounds based on carbon, hydrogen and other non-metallic elements. Polymer Matrix Composites (PMC) are the most developed composite materials group and they have found widespread applications. PMC can be easily fabricated into any large complex shape, which is an advantage

Govindan and Srinivasan [4] investigated the thermal stability of hybrid natural fibre reinforced PLA polymer composite by crystallization and melting behaviour by means of Differential Scanning Calorimetry (DSC) and thermo-mechanical properties using Dynamic Mechanical Analysis (DMA) and found that composite having 20% reinforcement of sisal/basalt fibre exhibits more thermal stability than composites reinforced with 10 and 15% of natural fibres. Buccella et al. [5] investigated the mechanical and rheological properties of polyamide 6, a virgin polymer through viscosimetry measurements and end group analysis on solubilized chips. Through DSC, the thermal properties of chain extended materials were investigated for correlating the quasi-static tensile properties. Dong and Bhattacharyya [6] investigated the crystalline structure and thermo-mechanical behaviour of PP/clay nanocomposites processed by melt processing. DSC analysis shows that, very little effect of the additional clay in the PP matrix is observed on the melting behaviour of nanocomposites while PP/clay nanocomposites show the overall higher crystallisation temperatures and slightly better enhancement levels of crystallinity compared to neat PP. DMTA results shows that, enormous reinforcement effect takes place in PP/clay nanocomposites with the modulus improvement of over 40% at a high clay content (8–10 wt%) and temperature below the T_g and the glass transition temperatures (T_g) of most nanocomposites also increase compared to that of neat PP, resulting from the intercalated structures to restrict the mobility of PP molecular chains. Tabatabai et al. [7] assessed the properties of unsaturated polyester resin blended with Flu-Gas Desulfurization (FGD) gypsum through mechanical and thermo-gravimetric tests. Investigation shows that, mechanical properties are enhanced with addition of optimum amount of FGD hydrated gypsum particles due to a fiber-reinforcement effect. TGA results showed that the thermal decomposition of unmodified resin occurs at around 400

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°C. However, resins with FGD gypsum can retain a significant fraction of their mass up to 1100 °C.

Paz et al. [8] studied the thermal, mechanical and thermos-mechanical properties of Polyamide 6/Brazilian organoclay nanocomposites fabricated through melt intercalation. Observations from TGA shows that, nanocomposites show higher thermal stability in relation to pure polymer. The clay acted as reinforcing filler increasing the rigidity of the system. The mechanical properties (modulus and yield stress) increased with the presence of organoclay. Costa et al. [9] briefly reviewed the Dynamic Mechanical Thermal Analysis (DMTA) in the viscoelastic characterization of composites to study the structure/properties relationships of composites since a detailed understanding of the structure/properties relationships is crucial for achieving the specific goals and hence comprehensive characterization of the viscoelastic properties of composites is an essential step in development process. In this work, seashells present abundantly in the coastal seashores were used in particulate form of size 75 µm in the matrix of Nylon 66 to prepare the composite material. Compounding of the composite containing 3, 6 and 9% of seashell particulates in the nylon 66 matrix is carried out using twin screw extruder and preparation of specimens for further testing is done using injection moulding machine. Distribution of seashell particles inside the polymer matrix is studied by SEM analysis and Thermo-mechanical properties such as Differential scanning calorimetry (DSC), Thermo-gravimetric analysis (TGA) and Dynamic mechanical analysis (DMA) were determined as per ASTM standards to characterize the composite materials.

Figure 1. Equipment's used for composite preparation and fabrication

2. EXPERIMENTAL DETAILS

2.1. Material Selection

Nylon 66 is an aliphatic polyamide thermoplastic prepared by polycondensation of adipic acid with hexamethylenediamine. It exhibits high tensile strength, good sliding properties, high melting point and electrical insulation, elasticity, toughness, and abrasion resistance [10-11]. Nylon 66 has good solvent resistance but low weatherability and undergoes discoloration in air at elevated temperatures. Good mechanical properties are maintained up to 27°C. Moisture resistance of nylon 66 is fair, moisture acts as plasticizer, increasing flexibility and toughness of the polymer [12]. Nylon 66 is widely used as gear wheels, friction strips, piston guides, impact plates, cam disks, etc. [13]. Nylon 66 has a melting point of 265°C, which is high for a synthetic fiber, though not comparable to either polyesters or aramids such as Kevlar. Its long molecular chain results in more sites for hydrogen bonds, thereby creating chemical "springs", and making it very resilient [14]. The properties of nylon

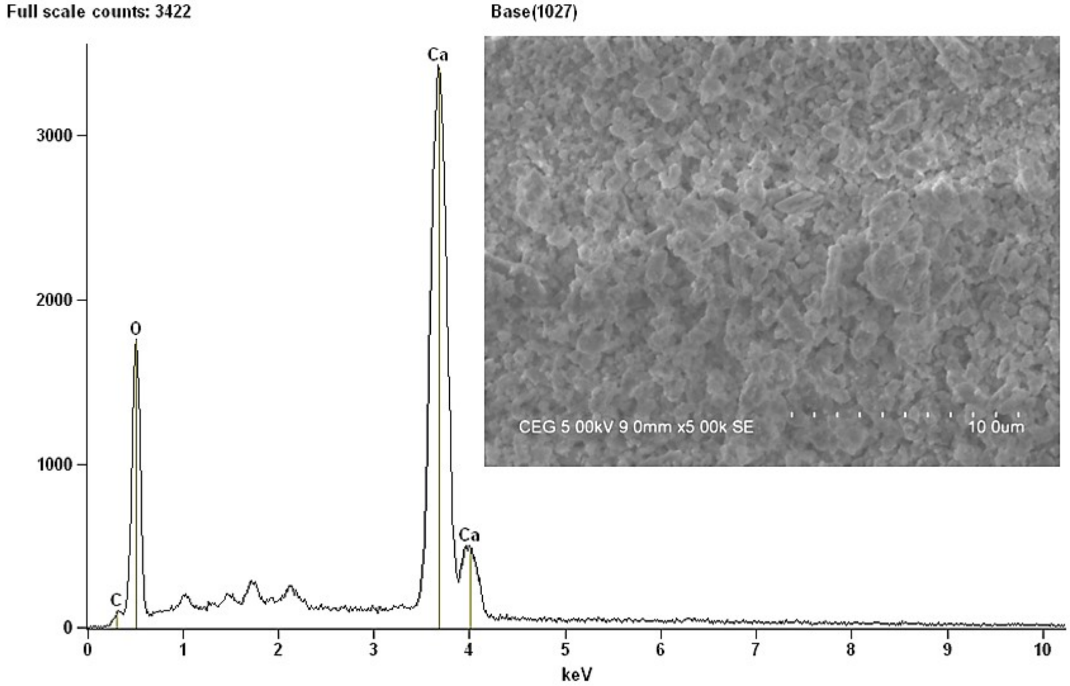

Figure 2. EDX spectrum and SEM micrograph of seashells

66 are: mould shrinkage of 1.5%, tensile strength of 83 MPa, flexural modulus of 2.81 GPa, flexural strength of 105 MPa, shear strength of 67.5 MPa, heat deflection temperature of 65.6°C, coefficient of thermal expansion of $8.1x10^{-5}$°C and of density 1.14 g/cm^3 [15].

Seashells of various molluscs such as oyster, clams, mussel and scallops, are available abundantly along coastal areas, which acts as a protective covering [16]. Shells are excreted from the outer surface of the animal called the mantle and are made up of mostly calcium carbonate. However, there are two distinct minerals (i.e. with different crystal structures) for calcium carbonate, which are calcite and aragonite [17]. Normally sea shells are mostly aragonite, which is slightly denser and harder than calcite (denser and harder than graphite but soft than diamond, but have same chemical formula 'C'). Shells have great mechanical properties, including high hardness and high toughness [18]. Shells' great mechanical properties are due to both their nanoscale structure and their combination of inorganic and organic materials. The sea shells were obtained from the seashores of Marakkanam, Tamil Nadu, India. The sea shells obtained should be cleaned properly and should be dried to remove moisture present in it. Nowadays, these seashells are mostly used in concrete aggregate for construction purposes [19-21].

2.2. Fabrication of Polymer matrix composite material

The cleaned and dried seashells were reduced to size by means of mechanical ball milling. The ball milling equipment shown in Figure 1, uses mild steel balls of diameter 48 mm to break the seashells into smaller pieces and then into powders. The powdered seashells are then segregated with respect to their micron size using a sieve equipment, and a particle size of 75 µm is chosen. After obtaining the seashell powder of desired particle size, compounding of nylon 66 and seashell is carried out in a twin screw extruder machine. Injection moulding machine is used to make the desired shape of the specimens for further processing and testing purposes. The twin screw extruder machine and injection moulding machine used is shown in Fig. 1.

Twin-screw extrusion is used extensively for mixing, compounding or reacting polymeric materials. The flexibility of twin-screw extrusion equipment allows this operation to be designed specifically for the formulation being processed. Injection moulding is a process in which a polymer is heated to a highly plastic state and forced to flow under high pressure into a mold cavity, where it solidifies. The moulded part, called a moulding, is then removed from the cavity. The process produces discrete components that are almost always net shape. Injection moulding is the most widely used moulding process for thermoplastics

Nylon 66 and powdered 75 µm sized seashells were mixed in three different combinations with the weight ratio of 97-3%, 94-6% and 91-9% respectively and the mixture is fed in to high speed co-rotating twin screw extruder for blending to make homogeneous mixing. Screw diameter of 28 mm, L/D ratio of 40, contains 5 different heat zones from feed point to exit point at various heat temperatures 125 ºC, 130 ºC, 140 ºC, 150 ºC, and 165 ºC, respectively with screw speed of at 150 rpm. Homogeneous mixing is carried out by twin screw extruder for 15 min and then extruded at the rate of 10 mm/ sec through a 1 mm gauge strands die. Strands were cooled in a water bath and then fed in to pelletizer to make compound pellets. Compounded pellets were dried at 60 °C in a vacuum for 12 hrs and stored in an air tight polyethylene bags. The extruded pellets were processed by injection moulding machine at a temperature of 170 ºC, back pressure of 7 bar, screw speed of 60 mm/sec and mould temperature of 30 ºC. Injection moulding machine has a screw diameter of 30 mm with L/D ratio of 20 to obtain hybrid composite specimens.

2.3. Thermo-Mechanical Analysis

2.3.1. Thermal gravimetric analysis

Thermogravimetric analysis (TA) or thermal gravimetric analysis (TGA) is a method of thermal analysis in which changes in physical and chemical properties of materials are measured as a function of increasing temperature (with constant heating rate), or as a function of time (with constant temperature and/or constant mass loss) [22]. TGA is commonly used to determine selected characteristics of materials that exhibit either mass loss or gain due to decomposition, oxidation, or loss of volatiles (such as moisture) [23]. The thermal stability of polymer matrix composites was investigated using Thermo Gravimetric Analyzer-TGA Q50 V20.10 Build 36, according to ASTM E1131 standard. Samples of about 40 mg were heated from 250°C to 800°C at a heating rate of 200°C /min under Nitrogen atmosphere by purging 50 ml /min.

2.3.2. Differential scanning calorimetry

Differential scanning calorimetry (DSC) and differential thermal analysis (DTA) are related techniques that measure the same thermal events with different methods [24]. DSC monitors the difference in heat flow between a sample and a reference as the material is heated or cooled while DTA measures a difference in temperature. For performing the DSC analysis, ASTM D3418 standard is followed. The thermal properties like glass transmission temperature T_g, crystallization temperature T_c, enthalpy of crystallization temperature H_c (J/g), melting temperature T_m, heat of melting H_m and degree of crystallinity X_c of seashell reinforced nylon 66 matrix composites were investigated using DSC in a Nitrogen atmosphere by purging gas 50 ml/min. Samples of about 2 mg of nylon+3%SS, nylon+6%SS and nylon+9% composites were scanned from 400 °C to 300 °C at the heating rate of 10 °C/min. The degree of crystallinity Xc (%) can be calculated using the following formula

$$X_c\% = \frac{\Delta H_m}{f\Delta H_m^\circ} \times 100 \quad (1)$$

Where ΔH_m is the heat of melting (J/g) of test specimen composites and ΔH_m° is the heat of melting for 100% crystalline.

2.3.3. Dynamic Mechanical Analysis

Polymers are viscoelastic materials, whose mechanical behaviour exhibits characteristics of both solids and liquids. Thermal analysts are frequently called on to measure the mechanical properties of polymers for a number of purposes [25]. Of the different methods for viscoelastic property characterization, dynamic mechanical techniques are the most popular, since they are readily adapted for studies of both polymeric solids and liquids. They are often referred to collectively as Dynamic Mechanical Analysis (DMA). Thermal analysts often refer to the DMA measurements on liquids as rheology measurements. Dynamic mechanical analysis involves imposing a small cyclic strain on a sample and measuring the resulting stress response, or equivalently, imposing a cyclic stress on a sample and measuring the resultant strain response [26]. In most commercial DMA instruments strain is the controlled input, while the resulting stress is measured. The storage modulus, loss modulus and loss factor (tan δ) of seashell reinforced nylon-66 composites were investigated as per ASTM D 4065 and measured as a function of temperature from 28°C to 200°C. Dynamic mechanical analyzer

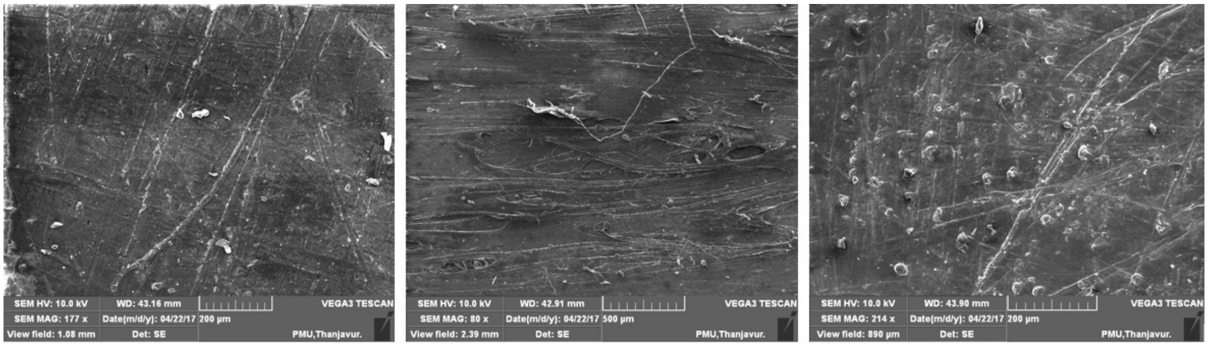

Figure 3. SEM images of (a) Nylon+3%SS (b) Nylon+6%SS (c) Nylon+9%SS

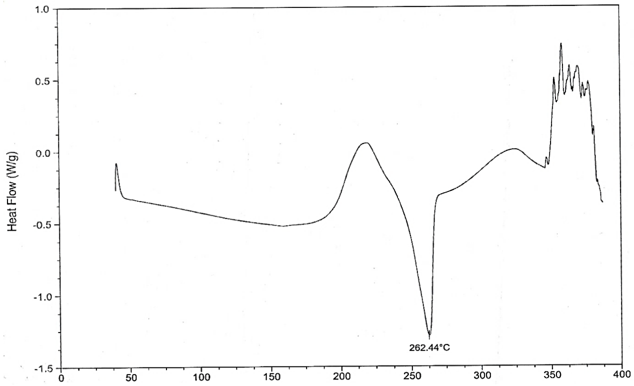

Figure 4. DSC analysis for Nylon+3%SS

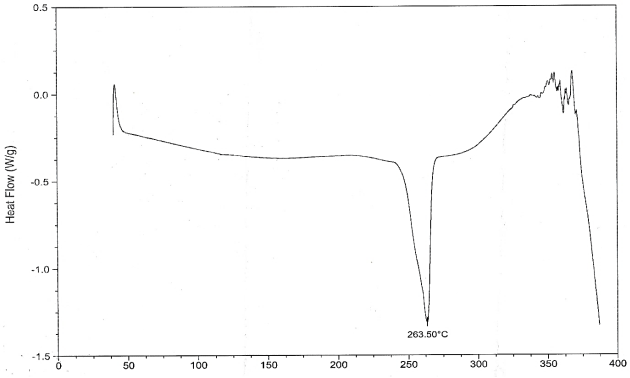

Figure 5. DSC analysis for Nylon+6%SS

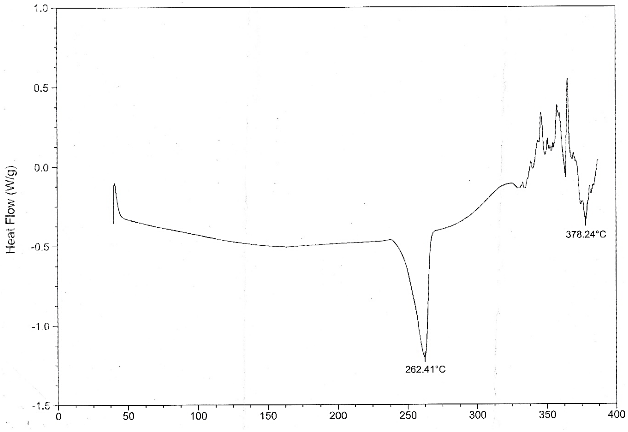

Figure 6. DSC analysis for Nylon+9%SS

DMA Q800 V20.6 Build 24 with a three-point bending fixture was used.

3. RESULTS AND DISCUSSIONS

The seashell particles of 75 μm size obtained after ball milling and sieve analysis is subjected to Energy-dispersive X-ray spectroscopy (EDX), an analytical approach for chemical characterization or elemental analysis of a given sample and SEM analysis to determine the particle size. EDS systems are relatively faster because the detector collects the signals of characteristic X-rays energies from a whole range of elements in a specimen at the same time rather than collecting signals from X-ray wavelength individually. An EDS spectrum is presented as the intensity of characteristic X-ray lines across the X-ray energy range. A spectrum in a range from 0.1 to about 10–20 keV can show both light and heavy elements because both *K* lines of light elements and *M* or *L* lines of heavy elements can be shown in this range [27].

From the EDX analysis shown in Fig. 2, it is observed that the seashell chosen for the reinforcement contains 51 wt.% of calcium, 48.53 wt.% of oxygen and 0.47% of carbon, which is shown as corresponding peaks in the graph. SEM micrograph shows mostly uniform sized seashells. After blending the polymer and seashell using twin screw extruder, the composite material is injected into the mould cavity as per the requirement using Injection moulding machine and the specimens were taken. The SEM micrograph is taken to visualize the distribution of seashell particles inside the Nylon 66 matrix, which is shown in Fig. 3. Uniform distribution of seashell particles is observed and with increase in percentage of seashell, higher particles are visible from the micrograph.

3.1. Differential scanning calorimetry

DSC scans of extruded and injection moulded Nylon+3%SS, Nylon+6%SS and Nylon+9%SS polymer matrix composites are illustrated in Fig. 4, Fig. 5 and Fig. 6 respectively. Observations from the graphs show their melting temperatures (T_m) are 361ºC, 373ºC and 378ºC respectively. However, the graphs showed the crystallization temperature (T_c) are 262 ºC, 263ºC and 262ºC along with the glass transition temperatures (T_g) of 220ºC, 240ºC and 240ºC respectively. Addition of seashell in Nylon 66 seem to in-

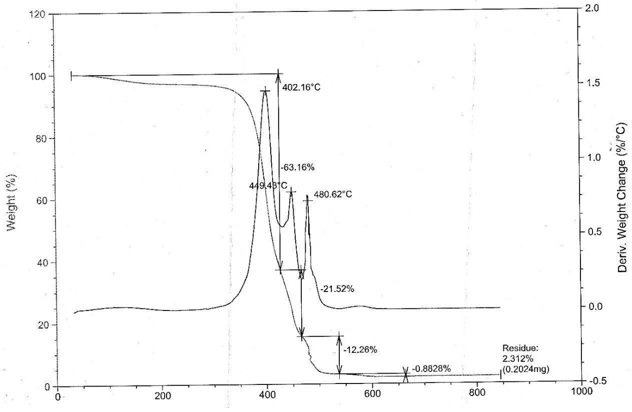

Figure 7. TGA analysis for Nylon+3%SS

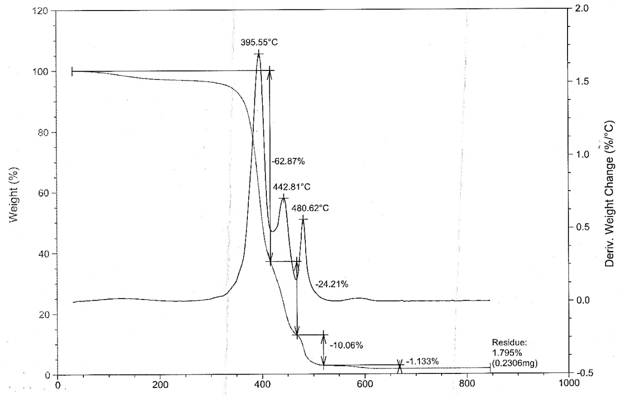

Figure 8. TGA analysis for Nylon+6%SS

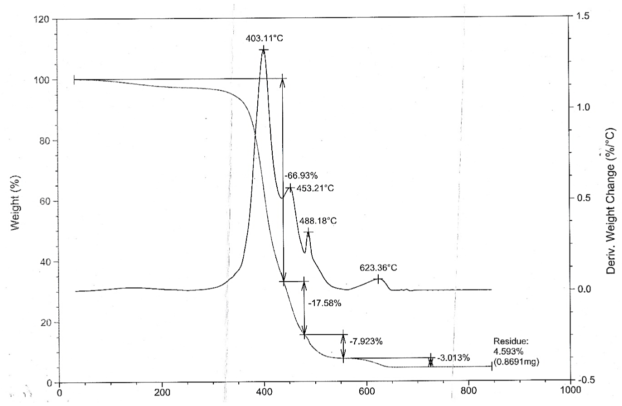

Figure 9. TGA analysis for Nylon+9%SS

crease T_g and T_m of Nylon+3%SS, Nylon+6%SS and Nylon+9%SS polymer matrix composites, however led to increase the percentage of crystallization. It was observed the percentage of crystallization Nylon+9%SS polymer matrix composite was 3% more than Nylon+3%SS PMC and 2% more than Nylon+6%SS PMC, which exhibited 48.26% crystallinity. Hence Nylon+9%SS PMC has improved thermal stability than other two PMC.

DSC scans of Nylon+3%SS, Nylon+6%SS and Nylon+9%SS polymer matrix composites are given in Table 1.

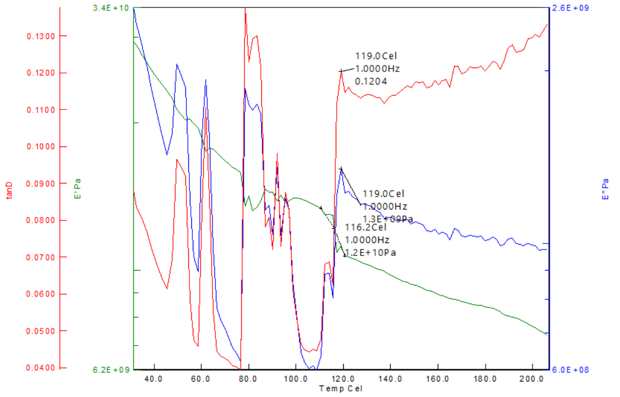

Figure 10. DMA analysis for Nylon+3%SS

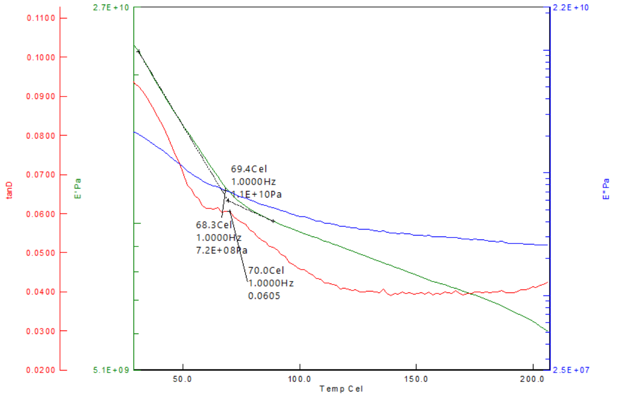

Figure 11. DMA analysis for Nylon+6%SS

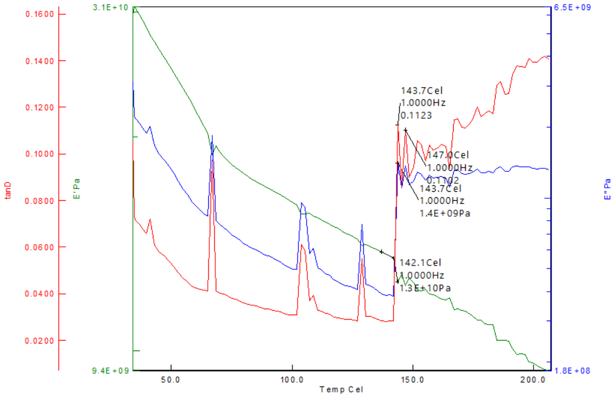

Figure 12. DMA analysis for Nylon+9%SS

3.2. Thermal Gravimetric Analysis

TGA performed on Nylon+3%SS, Nylon+6%SS and Nylon+9%SS polymer matrix composites are illustrated in Fig. 7, Fig. 8 and Fig. 9 respectively and which exhibited a stage T_{d1} degradation corresponding to weight loss of 63%, 62% and 67% at the temperatures of 325°C, 340°C and 370°C respectively. Whereas stage T_{d2} degradation corresponding to weight loss of 21%, 24% and 17% at the temperatures of 450°C, 442°C and 453°C respectively. Gradual weight losses in stage T_{d3} degradations are 12%, 10% and 8% at the temperatures of 480°C, 480°C and 488°C respectively. The degradation temperature for the fourth stage T_{d4} degradation corresponding to weight loss of 0.88%, 1% and 3% at the temperature of 540°C, 560°C and 610°C respectively. Those observation of final stage indicate that the influence of seashell in Nylon+3%SS PMC delays the degradation of Nylon+6%SS PMC is 20°C and Nylon+9%SS PMC is 50°C and weight loss in 0.25% and 2.13% respectively. Among three Nylon 66 based PMC studies Nylon+9%SS PMC higher weight loss in 95.44% from 100mg to 4.54mg and which had a greater thermal stability than other two PMC due to addition of Seashell into Nylon 66. Table 2 shows the TGA results of Nylon 66 based PMC. It is found that, performance of Nylon+9%SS PMC is more than other two PMC.

3.3. Dynamic Mechanical Analysis

Figures 10, Fig. 11 and Fig. 12 shows the storage modulus of the Nylon 66 based PM composites respectively as a function of temperature. Observations from the graph, that Nylon+3%SS composite has $12x10^9$ Pa, whereas its get increase to adding the percentage seashell into Nylon 66. The storage modulus of Nylon+9%SS increases when compared to Nylon+3%SS and Nylon+6%SS, which shows Nylon+9%SS having better mechanical properties with respect to rise in temperature. The gradual drop can be seen in the area of 80 to 150°C, which is associated with the glass transition temperature (T_g) of hybrid composites. The storage modulus continues to drop after glass transition region.

The loss modulus of the Nylon 66 based PM composites is shown in Fig. 10, Fig. 11 and Fig. 12 respectively as a function of temperature. Observations shows that, the loss modulus is more than Nylon+9%SS hybrid composites compared to and Nylon+3%SS and Nylon+6%SS. Which indicates less energy loss occurred on and Nylon+9%SS compared to other composites used in this study. The peak loss modulus value for the PM composite is around $1.3x10^9$ Pa for Nylon+3%SS and $0.72x10^9$ Pa Nylon+6%SS composite, whereas for Nylon+9%SS composite, it is around $1.4x10^9$ MPa. With increase in temperature further, the loss modulus tends to decrease for all PM composites.

The loss factor of tan δ variation of the various Nylon 66 based PM composites are shown in Figs. 10, 11 and 12 respectively as a function of temperature. The loss factor tan δ is expressed as a dimensionless number. A high tan δ value is indicative of a material that has a high, non-elastic strain component, while a low value indicates one that is more elastic. From the graphs, it is observed that tan δ value is higher for Nylon+6%SS composite has 0.06 and is lower for the other two Nylon 66 based PM composites are 0.12 and 0.11 respectively. Hence Nylon+6%SS composite exhibits high elastic strength but conclusion of overall DM analysis Nylon+9%SS PM composites better thermal properties. Table 3 shows the DMA results of Nylon+3%SS, Nylon+6%SS and Nylon+9%SS PM composites.

4. CONCLUSIONS

The conclusions derived from the Thermo-mechanical analysis of seashell reinforced nylon-66 polymer composites are:

1.EDX spectrum shows maximum peak values of calcium and oxygen and SEM micrograph showing mostly equalized seashell particles. Apart from this, uniform distribution of seashells is obtained during reinforcing them with the nylon 66 polymer matrix.

2.During DSC analysis the percentage of crystallization Nylon+9%SS polymer matrix composite was 3% more than Nylon+3%SS PMC and 2% more than Nylon+6%SS PMC, which exhibited 48.26% crystallinity and subsequently enhanced the T_g of 240ºC. Hence Nylon+9%SS PMC have improved thermal stability than other two PMC.

3.In analysis TGA, Nylon+3%SS, Nylon+6%SS and Nylon+9%SS polymer matrix composites exhibited four stage degradation weight losses. From observation of four stage degradation indicate that the influence of seashell in Nylon+3%SS PMC delays the degradation of Nylon+6%SS PMC is 20°C and Nylon+9%SS PMC is 50°C and weight loss in 0.25% and 2.13% respectively. Among three Nylon 66 based PMC studies Nylon+9%SS PMC higher weight loss in 95.44% from 100mg to 4.54mg and which had a greater thermal stability than other two PMC due to addition of Seashell into Nylon 66.

4.Observation of DM analysis on Nylon 66 based PM composites, that the storage modulus of Nylon+9%SS is $13X10^9$ Pa which is associated with the glass transition temperature (T_g) 150°C, which is higher than the other Nylon+3%SS and Nylon+6%SS PM composites, which shows Nylon+9%SS having better mechanical properties with respect to rise in temperature. The loss modulus is more

Table 1. DSC analysis of Nylon+SS composites

Sl. No	Sample	T_g °C	T_c °C	T_m °C	% Crystallinity
1	Nylon+3%SS	220	262.44	361.64	45.09%
2	Nylon+6%SS	240	263.5	373.48	45.82%
3	Nylon+9%SS	240	262.41	378.24	48.26%

Table 2. TGA analysis of Nylon+SS composites

Sl No	Sample	Td_1 °C	Td_2 °C	Td_3 °C	Td_4 °C	Weight Loss in %				Residual Weight in %	Max DTG °C
						Stage 1	Stage 2	Stage 3	Stage 4		
1	Nylon+3%SS	325	450	480	540	63.16	21.52	12.26	0.88	2.18	480.62
2	Nylon+6%SS	340	442	480	560	62.87	24.21	10.06	1.13	1.73	480.62
3	Nylon+9%SS	370	453	488	610	66.93	17.58	7.92	3.01	4.56	623.36

Table 3. DMA results of Nylon+SS composites

Sl. No	Material	Glass transition temperature T_g (ºC)	Storage Modulus ($1x\ 10^9$ Pa)	Loss Modulus ($1x\ 10^9$ Pa)	Tan Delta
1	Nylon+3%SS	119	12	1.3	0.12
2	Nylon+6%SS	69	11	0.72	0.06
2	Nylon+9%SS	143	13	1.4	0.11

in Nylon+3%SS and Nylon+9%SS compared to Nylon+6%SS indicates less energy loss occurred on Nylon+6%SS compared to other composites used in this study. Results of lass factor tan δ value is lower for Nylon+6%SS composite has 0.06 comparing with other two Nylon 66 based PM composites. Hence Nylon+6%SS composite exhibits high elastic strength but conclusion of overall DM analysis Nylon+9%SS PM composites better thermal properties comparing with other two Nylon 66 based PM composites.

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