

Effect of Nanoclay on Jute Fiber Reinforced Polyester Composites

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This research focus on the development of nano-structured composites and to investigate the effect of nanoclay on the mechanical and thermal properties of jute fiber reinforced polyester composite. 1wt.% nanoclay was reinforced in polyester with 5 vol.% jute fibre of size 5 mm. For comparison 5 vol.% jute fiber composite without nanoclay was also prepared. Tensile and flexural strength of the produced samples were measured by using an Instron Universal Testing Machine. Hardness of the developed nanocomposites was measured by a Shore A Hardness Tester. The fracture surfaces were investigated by Scanning Electron Microscope (SEM). Thermal properties were evaluated using Thermo-gravimetric Analysis (TGA). It is found that modulus and strength of nanoclay reinforced polyester nanocomposite is higher than that of pure polyester, jute fiber reinforced polyester composite and nanoclay reinforced jute fiber composites. However, thermal stabilities of nanoclay reinforced jute fiber composites are found to be higher than that of pure polyester, nanoclay reinforced nanocomposite and jute fiber reinforced composite. In fractographic analyses, it is found that pure polyester undergoes ductile fracture, nanoclay reinforced nanocomposite undergoes brittle fracture and jute reinforced composite undergoes mixed fracture.

Keywords: Nanocomposite, Nanoclay, Tensile property, Flexural strength, Fracture morphology.

I. INTRODUCTION

Composites are combinations of materials differing in composition, where the individual constituents retain their separate identities. These separate constituents act together to give the necessary mechanical strength or stiffness to the composite part. Composite material is a material composed of two or more distinct phases (matrix phase and dispersed phase) and having bulk properties significantly different from those of any of the constituents. Matrix phase is the primary phase having a continuous character. Matrix is usually more ductile and less hard phase. It holds the dispersed phase and shares a load with it. Dispersed (reinforcing) phase is embedded in the matrix in a discontinuous form. This secondary phase is called the dispersed phase. Dispersed phase is usually stronger than the matrix, therefore, it is sometimes called reinforcing phase. They are extensively used as materials in making aircraft structures, electronic packaging to medical equipment, and space vehicle to home building [1].

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In the simplest case, appropriately adding nanoparticulates to a polymer matrix can enhance its performance, often dramatically, by simply capitalizing on the nature and properties of the nanoscale filler [2] (these materials are better described by the term nanofilled polymer composites) [3]. This strategy is particularly effective in yielding high performance composites, when good dispersion of the filler is achieved and the properties of the nanoscale filler are substantially different or better than those of the matrix. An example of this would be reinforcing a polymer matrix by much stiffer nanoparticles [4,5] of ceramics, clays, or carbon nanotubes. It should be noted that the improvement in mechanical properties may not be limited to stiffness or strength. Time dependent properties could be improved by addition of the nanofillers [6]. Alternatively, the enhanced crystallization behavior under flow conditions [7] and other physical properties of high performance nanocomposites may be mainly due to the high aspect ratio and/or the high surface area of the fillers [9], since nanoparticulates have extremely high surface area to volume ratio when good dispersion is achieved. Nanoparticle dispersion in the polymer matrix is a key issue, which limits the applicable particle volume fraction and there for also the multi-functionality of the composite material [9]. Recent research on thin films (thickness <50 micrometer) made of polymer nanocomposites has resulted in a new and scalable synthesis technique, which allows the facile incorporation of greater nano-material quantities. Such advances will enable the future development of multi-functional small scale devices (i.e. sensor, actuator, medical equipment), which rely on polymer nanocomposites. Nanoscale dispersion of filler or controlled nanostructures in the composite can introduce new physical properties and novel behaviors that are absent in the unfilled matrices. This effectively changes the nature of the original matrix [3] (such composite materials can be better described by the term genuine nanocomposites or hybrids). Some examples of such new properties are fire resistance or flame retardancy [10], and accelerated biodegradability.

II. LITERATURE REVIEW

Submit Composites can be defined as materials that consist of two or more chemically and physically different phases separated by a distinct interface. The different systems are combined judiciously to achieve a system with more useful structural or functional properties non attainable by any of the constituent alone. Composites, the wonder materials are becoming an essential part of today's materials due to the advantage such as low weight, corrosion resistance, high fatigue strength, and faster assembly. They are extensively used as materials in making aircraft structures, electronic packaging to medical equipment, and space vehicle to home building [1]. The basic difference between blends and

composites is that the two main constituents in the composites remain recognizable while these may not be recognizable in blends. The predominant useful materials used in our day-to-day life are wood, concrete, ceramics, and so on. Surprisingly, the most important polymeric composites are found in nature and these are known as natural composites. Natural Fibre Composites Glass, carbon, Kevlar, and boron fibres are being used as reinforcing materials in fibre-reinforced plastics, which have been widely accepted as materials for structural and nonstructural applications [11]. However, these materials are resistant to biodegradation and can pose environmental problems. Natural fibres from plants such as jute, bamboo, coir, sisal, and pineapple are known to have very high strength and hence can be utilized for many load-bearing applications. These fibres have special advantage in comparison to synthetic fibres in that they are abundantly available, from a renewable resource and are biodegradable. But all natural fibres are hydrophilic in nature and have high moisture content, which leads to poor interface between fibre and hydrophobic matrix. Several treatment methods are employed to improve the interface in natural fibre composite [12-14], the mystery of the 'nano-world' has been progressively exposed in recently years. The nanometer scale is simply a range of 1-100 nm. The real interest in nanotechnology is to create revolutionary properties and functions by tailoring materials and designing devices on the nanometer scale. Polymer/layered silicates nanocomposites (PLSN) are a new class of composites. The filler is present in the form of sheets of one to a few nanometer thick and hundreds to thousands nanometers long, and their study in different polymer matrices will constitute the main object of this contribution. PLSN have been more widely investigated probably because the starting clay materials are easily available, filler loading required are 3-6 %w/w and because their intercalation and exfoliation chemistry has been studied for a long time [15]. Compared to conventional polymer composites and the pristine polymer matrix, PLSN can exhibit many advantages:

- a. They are lighter in weight than conventionally filled polymers because high degree of stiffness and strength are realized with far less high density inorganic material.
- b. They exhibit outstanding diffusional barrier properties without requiring a multipolymer-layered design.
- c. Their mechanical properties are potentially superior to unidirectional fibre reinforced polymers, because reinforcement from the inorganic layers will occur in two dimensions rather than in one.
- d. Improve solvent resistance, greater dimensional stability and superior flame retardancy.

These potential properties enhancements have led to increased applications of PLSN in various fields, such as the automotive industry (exterior and interior body parts and fuel tanks), packaging industry (bottles, containers, and plastic films), electronic industry (packaging materials and exterior parts of electronic devices), coating industry (paints, wire enamel coatings, etc.) and aerospace industry (body parts of airplanes and exterior surface coatings) [16].

III. METHODOLOGY

The raw materials for performing the experiment were:

1. Pure Polyester as matrix: 2 litres
2. Jute Fibres: 5 mm
3. Graded Nanoclay:
Nanoclay Nanomer 1.31 PS.
Montmorillonite clay surface modified with 15-35 wt% octadecylamine and 5 wt% aminopropyltriethoxysilane.
4. Hardener:
Commercial Name : Methyl ethyl ketone peroxide hardener
Molecular formula : $C_8H_{18}O_6$
Molar mass : $210.22 \text{ g mol}^{-1}$
Appearance : Colorless Liquid
Density : 1.170 g cm^{-3}

Preparation of Jute fibres:

The Jute fibres were prepared as follows:

1. The Jute fibres were first cut keeping an average length of 5 mm.
2. Initially the jute fibres, after cutting in 5 mm remain entangled together. Therefore, to eliminate the agglomeration, the fibres are soaked or immersed in a bucket filled with cold water and the jute fibres were separated from each other using free hand.
3. After that, the fibres were then filtered using sieves and thin cloth and dried in air.
4. After drying, the fibres are collected and kept in a clean air-tight packet so that no dust or moisture is attracted by them.

Preparation of composites sample:

Liquid thermoset type polyester was selected as matrix material for this study. Matrix material, i.e., polyester was purchased from local market. The dimension of the casting dies were 100mm in diameter and 6mm in thickness and it was calculated that each casting is filled up by 50 gm of the liquid polyester and four castings were produced at a time, i.e., for each experiment, 200gm of liquid polyester was required. First of all, pure polyester samples were produced for standard comparison. Usually air/gas remains dissolved into the liquid polyester and when solidified, it makes various types of defects such as gas pockets, bubbles, microcracks etc. These defects deteriorate the properties of the cast samples. So, before casting the liquid polyester and the nanoclay into the dies, it was made gas/air free. For each time of vacuum process, it took about 10 minutes. Then hardener (methyl ethyl ketone peroxide) of required proportion was also added inside the gas free liquid polyester and stirred properly for obtaining a uniform mixture. The mixture was again evacuated putting the beaker inside the vacuum chamber for removing air. Due to addition of hardener, the mixture formed glutinous mass within 2-3 minutes. At this moment, the beaker was taken from the chamber and the pasty mass was cast into die of required shape.

Nanoclay reinforced composites:

In the case of nanoclay reinforced composite casting, 50 gm for each casting, i.e., $50 \times 4 = 200$ gm of pure polyester was taken in a container and then 1 wt% of graded nanoclay was added to the polyester and the mixture was stirred properly to

get uniform distribution of the reinforcing particles. The well-mixed polyester with nanoclay was taken into the vacuum chamber and made gas/air free following the procedure mentioned earlier. After that, the beaker was taken out of vacuum chamber, followed by addition of required proportion of hardener into the mixture. This mixture was again evacuated following the same procedure. After full vacuum process, the glutinous mass was cast into the die to obtain specimens for various tests. Nanoclay Jute fibre reinforced composites. The same procedure as described above was carried out to produce jute fibre reinforced polyester composites, and jute fibre with graded nanoclay reinforced polyester composites. Amount of jute fibres added were 5 vol.% and the amount of nanoclay was kept fixed, i.e., 1 wt.%. So, basically four categories of samples were produced. They are listed below for simplicity:

1. Pure polyester samples abbreviated as P.
2. Polyester reinforced with graded nanoclay abbreviated as (P + GNC).
3. Polyester reinforced with jute fibres abbreviated as (P + J).
4. Polyester reinforced with jute fibre and graded nanoclay abbreviated as (P + J + GNC).

After solidification of the castings, curing was performed for each sample. The samples were removed from the dies carefully and were transferred to drying oven and kept there at 110°C for 24 hours. Sample preparation for tensile test:

More than eight samples of pure polyester and each group of composites were made through die cast method. For preparing Tensile test samples, properly prepared liquid polyester, mixture of liquid polyester and nanoclay and mixture of liquid polyester, jute fibres and nanoclay were machined with a hacksaw to form rectangular samples as shown in Fig.1 (a). The rectangular samples were then machined to get standard ASTM tensile test samples as shown in Fig.1 (b). The final dimensions of the tensile test samples were 100 mm x 12 mm x 6 mm with gauge length 30 mm.



(a)



(b)

Fig. 1(a): Rectangular sample before machining to Tensile sample and also used for bend test and 1(b): Tensile test specimen for tensile test.

Sample Preparation for Bend Test:

For performing bend test, casting samples were machined to produce a rectangular shape of dimensions 5 mm thickness,

13 mm width with span length of 80 mm. Figure 2 shows the specimen produced for bend test.



Fig. 2: Bend test specimen for measuring flexural strength

Fractography

After tensile tests, fracture surfaces were cut for fractographic analysis under the SEM. The electrical conductivities of all test samples (polyester, nanoclay reinforced composites and jute fibres plus nanoclay reinforced composites) used in this research project were very poor. To avoid this, the fracture surfaces of all samples were coated by gold sputtering technique. The samples were bonded with conductive carbon tape on an aluminium stub. Then the stub and fracture surface was further connected with highly conductive copper foil. For fractographic analysis, 15 kV accelerating voltage was used. Under SEM, various fracture features were observed and they were photographed.

Thermo-Gravimetric Analysis (TGA)

Small solid particle was collected from the pure polyester and composites. These samples were measured by digital weighing machine. For thermal stability analysis, the decomposition rates were measured by a SETARAM TGT DTA 92 thermo-gravimetric apparatus. A sample of initial mass of 15 to 20 mg was introduced into a platinum crucible. The mass variation of the sample (TG) was then measured as a function of temperature (or time), and the rates of mass loss (DTG) were determined by using a thermo-balance under an inert gas nitrogen atmosphere up to 750°C, at a heating rate of 20°C min⁻¹. Finally, the data were saved. All samples of the polyester and nanoclay reinforced polyester composites were tested in the same way.

IV. RESULTS AND DISCUSSIONS

Effect of Jute on polyester and graded nanoclay reinforced polyester

Figures 3(a) and 3(b) show, in the form of bar charts, the comparison of tensile properties and Young's Modulus respectively between polyester, polyester reinforced with graded nanoclay, polyester reinforced with jute and graded nanoclay and polyester reinforced with jute only. It has already been told that a substantial increase in strength and modulus were obtained, when graded nanoclay was added to pure polyester as reinforcement, due to equal homogenous distribution of nano-scale particles in polyester matrix. But when jute fibres and graded nanoclay both were added as reinforcement, both strength and modulus has decreased compared to that of graded nanoclay reinforced composite. The lowest strength belongs to polyester reinforced with jute only. The major reasons of lower strength and modulus may be due to inhomogeneous distribution of the jute fibres over the polyester matrix which degraded the composite as a

whole, dirt and other impurities brought about by the jute fibres which acted as defects in the composites, some blowholes were formed due to trapped air and moisture among the jute fibres, and moreover, there is a minimum critical fibre length below which the fibres do not act as reinforcing agents [17,18].

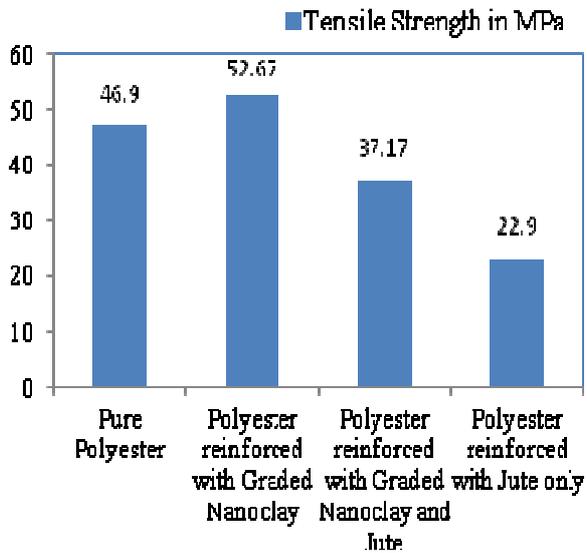


Fig. 3(a): Bar Chart showing Tensile properties of the composites

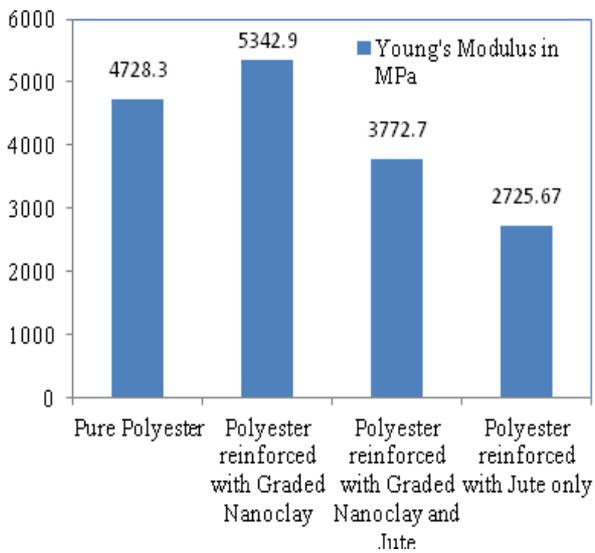


Fig. 3(b): Bar Chart showing Young's Modulus of the composites

Figures 3(c) illustrates bar chart showing the flexural strengths of unreinforced polyester, polyester reinforced with graded nanoclay, polyester reinforced with jute and graded nanoclay and polyester reinforced with jute only. From the figure, it can be observed that flexural strength of polyester reinforced with graded nanoclay is about 15% more than that compared to unreinforced polyester. Upon addition of jute fibres as additional reinforcing fillers the flexural strength of the composites degrades from 82.12 Mpa to 56.10 Mpa, i.e., by about 30%. The lowest flexural strength belongs to polyester reinforced with jute only.

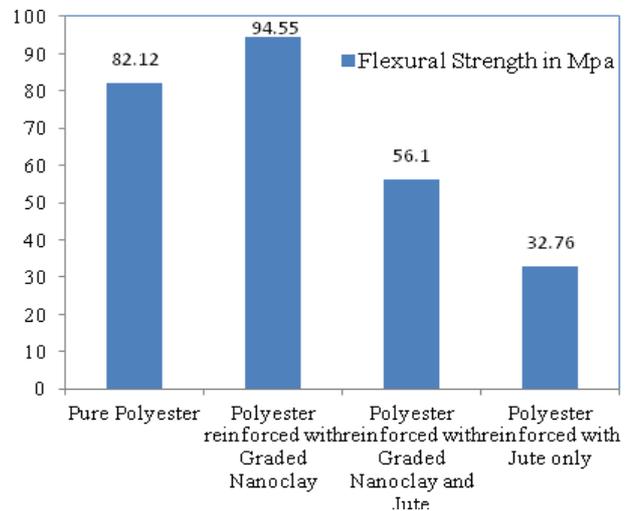
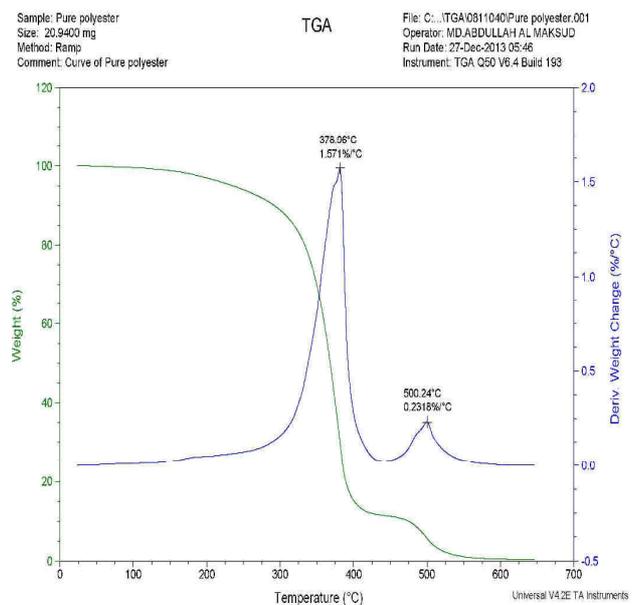


Fig. 3(c): Bar Chart showing Flexural properties of the composites

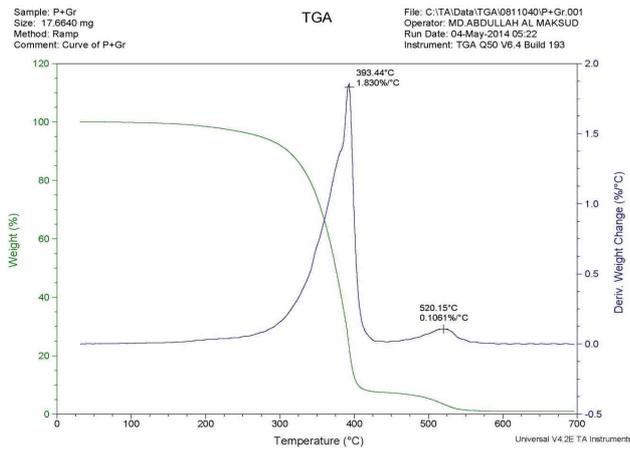
TGA Analysis

Figure 4a, 4b, 4c and 4d show TGA curves for all the four types of samples. Table 1 shows the maximum derivative weight change of each sample and their corresponding temperatures. From the table, we can clearly observe and judge the thermal stabilities of the composites and thus know which composite has the highest thermal stability and which has the lowest one. From the table, we observe that the maximum derivative weight change occurs at 396.97 degree Celsius which is greater than any of the composites mentioned earlier. This means that, when both jute fibres and graded nanoclay are used as reinforcement, **thermal stability is the highest**. This is because the heat is distributed to both fibres and nanoclay equally therefore enabling more uniform temperature distribution as a result of which more thermal energy is required to decompose the composite.



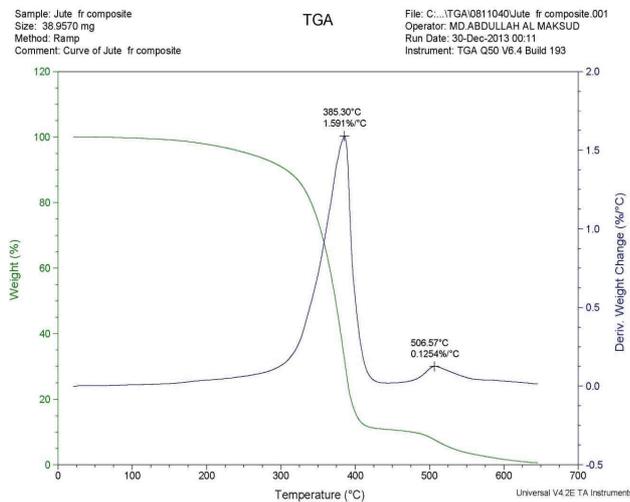
4(a)

Fig. 4(a): TGA curve for pure polyester



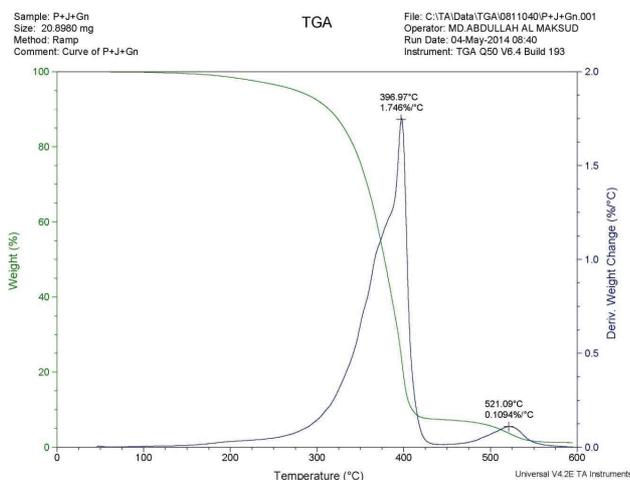
4(b)

Fig. 4(b): TGA curve for polyester reinforced with graded nanoclay only.



4(c)

Fig. 4(c): TGA curve for polyester reinforced with jute only.



4(d)

Fig. 4(d): TGA curve for polyester reinforced with jute fibre and graded nanoclay.

Table 1: Maximum derivative weight change of each sample and their corresponding temperatures

Sample	Temperature at maximum derivative weight change (°C)	Deriv. Weight change (%/°C)
P	378.96	1.571
P + GNC	393.44	1.830
P + J	385.30	1.591
P + J + GNC	396.97	1.746

SEM Fractography

Figure 5(a) shows SEM photograph of the fracture surface of pure polyester. Here, we can see that, there are no particles or impurities present which indicates the presence of virgin polyester. The photograph also shows the initial fracture zone of the material and the direction of fracture which is along the tensile loading. From the figure, it can be observed that the fracture is a ductile type fracture. Figure 5(b) shows the SEM photograph of fracture surface of graded nanoclay reinforced polyester composite. Here, it clearly indicates is that, the fracture is brittle, there is no debonding or cup and cone observed, and fracture occurs at a single leap, viz., it is a brittle fracture. The fractography analysis is supportive with the mechanical properties where tensile and flexural strengths have increased upon addition of graded nanoclay. Figure 5(c) shows the SEM photograph of jute reinforced polyester. The figure clearly indicates that there is considerable fibre-matrix debonding existing in the fracture surface which lead to lowering the strength of the composite. Some fibre pull-out phenomena is also observed. Figures 5(d) shows SEM photograph of polyester reinforced with jute and graded nanoclay. It shows both misaligned and aligned fibres along with some agglomeration of graded nanoclay.

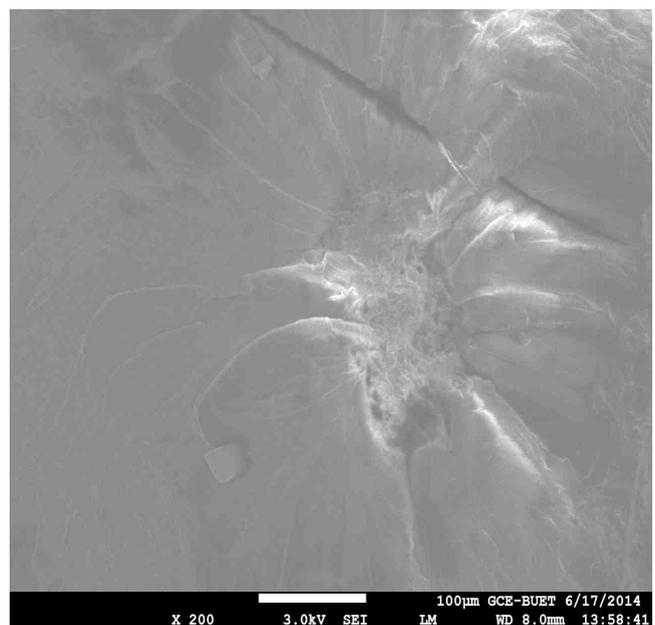


Fig. 5(a): SEM photograph of fracture surface of pure polyester

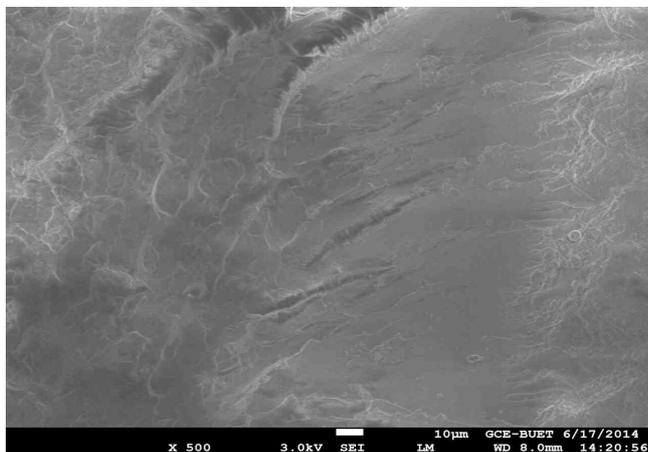


Fig. 5(b): SEM photograph of fracture surface of nanoclay reinforced polyester



Fig. 5(c): SEM photograph of fracture surface of jute reinforced polyester composite

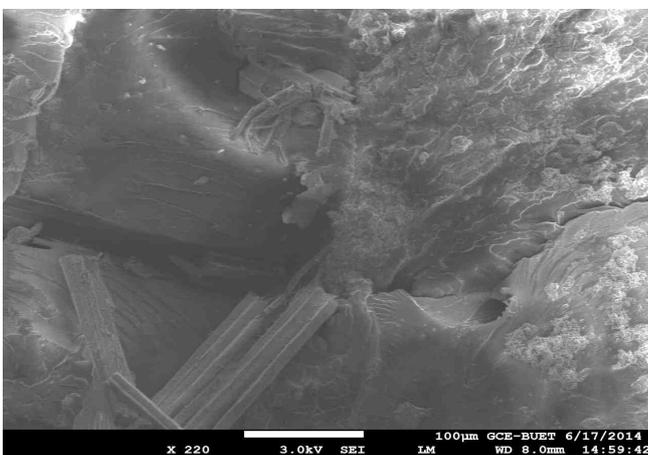


Fig. 5(d): SEM photograph of fracture surface of polyester reinforced with jute and graded nanoclay

V. CONCLUSIONS

The research was conducted to explore the effects of graded nanoclay and jute fibres on polyester and to modify their properties by a viable combinations of the reinforcing fillers. The findings are noted below:

1. When graded nanoclay was added to pure polyester as reinforcement. The strength and modulus has increased by about 12% compared to that of pure polyester and flexural strength has increased by about 15%.

2. When jute fibres and graded nanoclay both were added as reinforcement, both strength and modulus has decreased compared to that of graded nanoclay reinforced composite.
3. Thermal stability increases when graded nanoclay is added as reinforcement.
4. When both jute fibres and graded nanoclay are added as reinforcement in polyester, thermal stability is the maximum.
5. According to fractographic analysis, fracture of pure polyester is ductile type and fracture of graded nanoclay reinforced polyester composite is brittle type, and the fractures of other composites are a combination of the two, that is, mixed type.

VI. RECOMMENDATIONS

1. To increase the tensile strength and flexural strength of polyester composite, sizes of reinforcing particles should be as small as possible (to a nano-level). But extra cautiousness must be taken into consideration, since small particles tend to agglomerate to form larger particles and hence can degrade the quality of a composite.
2. For achieving positive feedback in different properties such as tensile and flexural strengths, critical length of jute fibre, the fibres' lengths compared to their diameters, and how the fibres are arranged in the matrix and its volume percent must be taken into consideration. Otherwise, they will only lower the strengths of polymer composites.
3. In case of modification of thermal stability, jute fibres are useful materials. So by selecting appropriate fibre length, fibre alignment and volume percent of fibres, both strength and thermal stability can be optimized.

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