

Optimization of Nanoclay Loading on the Thermo-mechanical Behavior of Chemically Treated Jute Polyethylene Nanocomposites

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Authors' contributions

This work was carried out in collaboration among all the authors. Author MFH designed the study, managed the literature searches, performed the statistical analysis, wrote the protocol and draft of the manuscript. Authors MAA, MKEZ, MMH, RZ and CMZ managed the lab facilities and analyses of the study. All authors read and approved the final manuscript.

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ABSTRACT

In this study, the jute polyethylene nanocomposites were developed using hot-press technique. In order to enhance the compatibility between fiber, polymer and nanofillers, chemically treated jute (with 3-isocyanatopropyltriethoxy silane) and organically modified nanoclay were used for the manufacturing of nanocomposites in this study. The effect of different types of montmorillonite (MMT) nanoclay on the thermal behaviour of prepared nanocomposites have been investigated. Fourier transform infrared (FTIR) spectroscopy and scanning electron microscopy (SEM) were used to study fiber surface modification and surface morphology of nanocomposites, respectively. Tensile strength (TS) tensile modulus (TM) were considered to assess mechanical behavior. Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) were carried out to evaluate the thermal performances. It was found that the MMT nanoclay loaded nanocomposites shows higher tensile values and thermal stability than composite without nanoclay, and MMT-1.31PS loaded nanocomposite exhibited highest improvement among five types of MMT nanoclay used in this paper.

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1. INTRODUCTION

In recent years, the traditional jute products are becoming increasingly non-marketable due to the rise in the sale and use of the high intensity synthesis fibers. Therefore, it is urgent to develop value-added jute products for diversified use and it can be substituted for conventional fibers in many applications and has been applied as reinforcement to eco- and bio-composites [1-3]. The presence of surface impurities and hydroxyl groups (-OH) provide the fibers less attractive for the reinforcement of polymers [4]. In order to remove non-cellulosic constituents to obtain reactive -OH groups for interacting with the polymers, the surface characterization of natural fibers need to be modified by chemical treatments. In this study, silane chemical treatment can be used to overcome the stated incompatibility of surface polarities [5,6]. On the other hand, the matrix phase plays an important role in the performance of polymer composites. Thermoplastics offer many advantages over thermoset polymers, because of their low processing costs and design flexibility [7]. Most of the work has been reported so far deals with polymers such as polyethylene, polypropylene, polystyrene, and polyvinylchloride. Among the synthetic polymers, polyethylene (PE) has outstanding properties like low density, low cost, good flex life, excellent surface hardness, scratch resistance and admirable electrical insulating properties [8]. In the case of thermoplastic composites, the dispersion of the fibers in the composites is also an important parameter to achieve consistency in the product. Jute-polyethylene composite has showed remarkable increase in mechanical properties [9]. As jute is abundantly available in South Asia such as Bangladesh, India and China, it is worthwhile to analyze the thermal and mechanical performance of the composites produced with polyethylene (PE) matrix.

In addition, due to the availability, intercalation/exfoliation behaviors, and high surface area and surface reactivity, montmorillonite (MMT) can be used for the preparation of fiber polymer nanocomposites [10]. Generally, MMT nanoclay is hydrophilic in nature [11]. In order to achieve an enhanced compatibility with polymer matrices, organically modified clay is used for the manufacturing of nanocomposites. The MMT with greater carbon number of modifier that increases the hydrophobicity and finally

increased interfacial adhesion between polymer matrix and MMT [12]. In contrast, MMT with less carbon number of modifier are slightly dispersed in the composite system which cause air bubble and agglomerations [13,14]. At certain amount of nanoclay provides better dispersion in the composites system. It had been reported that the 2 wt% of nanoclay loaded fiber polymer nanocomposite exhibited an optimized mechanical improvement [15]. Thus, the 2 wt% of different types MMT nanoclay have been used to produce nanocomposites in this work. It is noted that the type of MMT nanoclay has not been optimized yet. Therefore, the aim of this study is to investigate the nanoclay effect on the thermal behavior of silane treated jute polyethylene/clay nanocomposites.

2. EXPERIMENTAL

2.1 Materials

Jute fibers and polyethylene (PE) granules used in this study were collected at Bangladesh Jute Research Institute (BJRI), Dhaka and supplied by Siam Polyethylene Company Limited, Bangkok, respectively. Methanol (CH₃OH), sodium hydroxide (NaOH), acetic acid (CH₃COOH), and silane(3-isocyanatopropyl triethoxysilane) were supplied by Merck, Germany. Montmorillonite (MMT-1.28E) clay surface modified with 25-30 wt% trimethylstearyl ammonium(C₂₁H₄₆N⁺), montmorillonite (MMT-1.30E) clay surface modified with 25-30 wt% octadecylamine (C₁₈H₃₉N), montmorillonite (MMT-1.31PS) clay surface modified with 15-35 wt% octadecylamine (C₁₈H₃₉N) and 0.5-5 wt% aminopropyltriethoxysilane (C₉H₂₃NO₃Si), montmorillonite (MMT-1.34TCN) clay surface modified with 25-30 wt% methyl dihydroxyethyl hydrogenated tallow ammonium (C₁₈H₃₇(CH₃)N⁺(CH₂-CH₂-OH)₂) and montmorillonite (MMT-1.44P) clay surface modified with 35-45 wt% dimethyl dialkyl (C14-C18) amine were supplied by Sigma-Aldrich (USA). Average particle size of MMT nanoclay mentioned above is ~ 20 micron.

2.2 Chemical Treatment of Jute Fiber

In this study, jute fibers were mercerized using 5% NaOH initially and then treated with silane coupling agent. The mercerized jute fibers were then immersed in methanol-water (90/10 w/w) containing 2% silane with constant stirring for 24 hours. After taken out from solution, the fibers

were washed several times with distilled water and dried in an oven at 80°C for 12 hours. Finally, for composite fabrication, the dried fibers were cut approximately into 3 mm in length.

2.3 Fabrication of Composites and Test Specimens

Jute fibers, PE granules, and different types of MMT nanoclay were mixed well using a Brabender mixer machine according to the Table 1 for the manufacturing of nanocomposites. It was then placed in a hot press machine and heated to 180°C for 1 hour under the pressure at 7 MPa. The composites were cooled in open air then collected for characterization.

2.4 Characterization

2.4.1 Fourier Transform Infrared Spectroscopy (FTIR)

The infrared spectra was recorded to determine the functional groups of jute fibers as a function of treatment using a Shimadzu FTIR 81001 Spectrophotometer taking scanned for 32 times from 4000 to 500 cm^{-1} .

2.4.2 Tensile test

To study the mechanical characterization of prepared composites, tensile tests were conducted according to ASTM D 638 using a Universal Testing Machine (Model: MSC-5/500, Shimadzu Company Limited, Japan) at a crosshead speed of 5 mm/min. The dimension of the specimen was 115 mm x 6 mm x 3.1 mm. In each case, five rectangular specimens were tested and the average value was reported.

2.4.3 Scanning Electron Microscopy (SEM)

A table top scanning electron microscope (TM3030, JEOL Company Limited, Japan) was used to examine the surface morphology. The interfacial adhesion between fiber, clay and polymer matrices were observed from the micrographs (fracture surface) taken at a magnification of 500x. The gold coated samples were observed under the SEM.

2.4.4 Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC)

To understand the thermal characterization of manufactured composites, TGA and DSC were

Table 1. Jute fibers, PE and different types of MMT nanoclay for composites fabrication

Composites	Composition by weight (%)			Specimen name
	Jute	PE	MMT	
Silane treated jute polyethylene composites	5	95		SJPEC
	10	90	--	
	15	85		
	20	80		
Silane treated jute polyethylene MMT-1.28E nanocomposites	5	93		SJPENC-1.28E
	10	88	2	
	15	83		
	20	78		
Silane treated jute polyethylene MMT-1.30E nanocomposites	5	93		SJPENC-1.30E
	10	88	2	
	15	83		
	20	78		
Silane treated jute polyethylene MMT-1.31PS nanocomposites	5	93		SJPENC-1.31PS
	10	88	2	
	15	83		
	20	78		
Silane treated jute polyethylene MMT-1.34TCN nanocomposites	5	93		SJPENC-1.34TCN
	10	88	2	
	15	83		
	20	78		
Silane treated jute polyethylene MMT-1.44P nanocomposites	5	93		SJPENC-1.44P
	10	88	2	
	15	83		
	20	78		

conducted using a TGA/DSC1 STAR System, Mettler Toledo thermal analyzer according to ASTM E1131 and ASTM D3418 respectively.

The thermal analysis was done in a nitrogen gas atmosphere with flow rate of 30 mL min^{-1} at the heating rate of $10^\circ\text{C min}^{-1}$ from 50°C to 800°C . The weight of the samples was maintained at 10 mg. The activation energy (E_a) was calculated from TGA graphs using Broido equation [16] as given below:

$$\ln\left(\ln\frac{1}{y}\right) = \frac{E_a}{RT} + \text{constant}$$

Where, y is the fraction of undecomposed non-volatilized material, T is the absolute temperature, and R is the gas constant ($8.314 \text{ J mol}^{-1} \text{ K}^{-1}$). The values of y have been taken from the TGA data. The values of $\ln(\ln 1/y)$ were plotted in Y axis, while the temperatures as $1/T$ (in Kelvin scale) was plotted in X axis. Finally from the slope of the trend line, the activation energy of the composite can be calculated.

3. RESULTS AND DISCUSSION

3.1 FTIR Study

Fig. 1 shows the IR bands of raw jute and silane treated jute fibers. The distinctive features of the

spectrum are due to the lignin, hemicelluloses and α -cellulose in jute [17]. The characteristic of hydrogen bonded O-H stretching vibration was observed in a broad region of $3600\text{-}3200 \text{ cm}^{-1}$ [18]. The O-H stretching vibration decreased from 3269 to 3242 due to the silane chemical treatment of fibers. The C-H stretching was observed near 2902 cm^{-1} which is for $-\text{CH}_2$ groups in cellulose and hemicelluloses. Due to the pre-treatment of fibers with NaOH, this band shifted to lower value, as a result in the partial removal of some waxy substances [19]. A significant peak at 1751 cm^{-1} is due to the C=O stretching for the $-\text{COO}-$ groups of hemicellulose in jute. After silane chemical treatment, this band shifted to lower value (1741 cm^{-1}) and became weak. This is due to the removal of acetyl group present in hemicellulose [20]. The band at 1641 cm^{-1} specified the absorbed water in crystalline cellulose [21]. This band disappeared due to the chemical treatment of fiber using silane. The peak at 1512 cm^{-1} indicates the aromatic rings in lignin, and another peak at 1359 cm^{-1} for the C-H bending in hemicellulose and lignin [22]. The water, lignin and hemicelluloses partially removed from jute by means of silane chemical treatment. As a result, the hydrophilic nature of jute fibers was significantly reduced [2]. Thus, the the fiber and matrix interfacial bonding was enhanced which is reflected in the thermal behaviors.

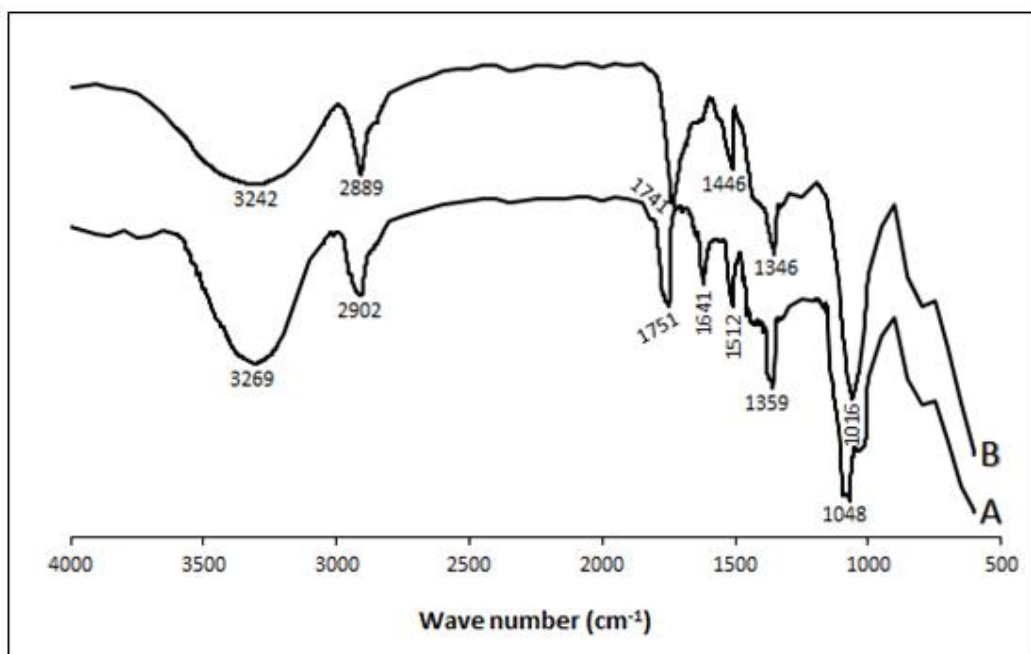


Fig. 1. FTIR curves for (A) raw jute and (B) silane treated jute

3.2 Mechanical Behavior

It had been reported that the treated jute composites exhibited greater mechanical improvement compared to raw one [2]. Thus, the mechanical behavior of silane treated jute composites without and with different types of MMT nanoclay have been examined in this study. The mechanical performance of the manufactured composites was carried out by tensile strength (TS) and tensile modulus (TM) behaviors. The tensile properties of prepared composites are found in Fig. 2 wherein C, D, E, F, G, and H belong to tensile strength and C', D', E', F', G', and H' belong to tensile modulus, respectively. It was observed that the tensile values for all the prepared composites increase continuously up to 15% fiber loading and then decrease after further addition of jute. Thus, the 15 wt% jute content was found as the optimum composition. This is explained by the homogeneity of fiber and wettability of polymer matrix. In lower fiber content, the composite shows poor tensile properties due to poor fiber population and low load transfer capacity to one another, whereas after 15 wt% fibers get agglomeration. At intermediate fiber loading (15 wt%), the population of the fibers is ideal for

maximum orientation and the fibers actively participate in stress transfer [9,23]. It was found that the nanoclay loaded composites (SJPEC) shown higher tensile value than without clay one. The highest tensile value were observed for MMT-1.31PS loaded nanocomposites (SJPEC-1.31PS) followed by SJPEC-1.34TCN, SJPEC-1.28E, SJPEC-1.30E and SJPEC-1.44P. The MMT-1.31PS incorporated silane treated jute polyethylene nanocomposites exhibited improvements in TS and TM by approximately 39% and 56% respectively over composite without nanoclay. Whereas, the MMT-1.30E loaded nanocomposites showed improvements by approximately 30% and 44% respectively. It had been reported that MMT-1.30E loaded chemically treated jute polymer nanocomposites exhibited improvements in TS and TM by approximately 8% and 15% respectively [2]. The hydrophobicity of chemically modified MMT increases as the carbon number of modifier [12]. It was stated in "materials" section that the MMT-1.31PS clay modified with highest carbon number (C₂₇) of modifier. Thus, the interfacial adhesion between nanoclay and polymer matrix is due to the chemical modification of nanoclay that increases the hydrophobicity.

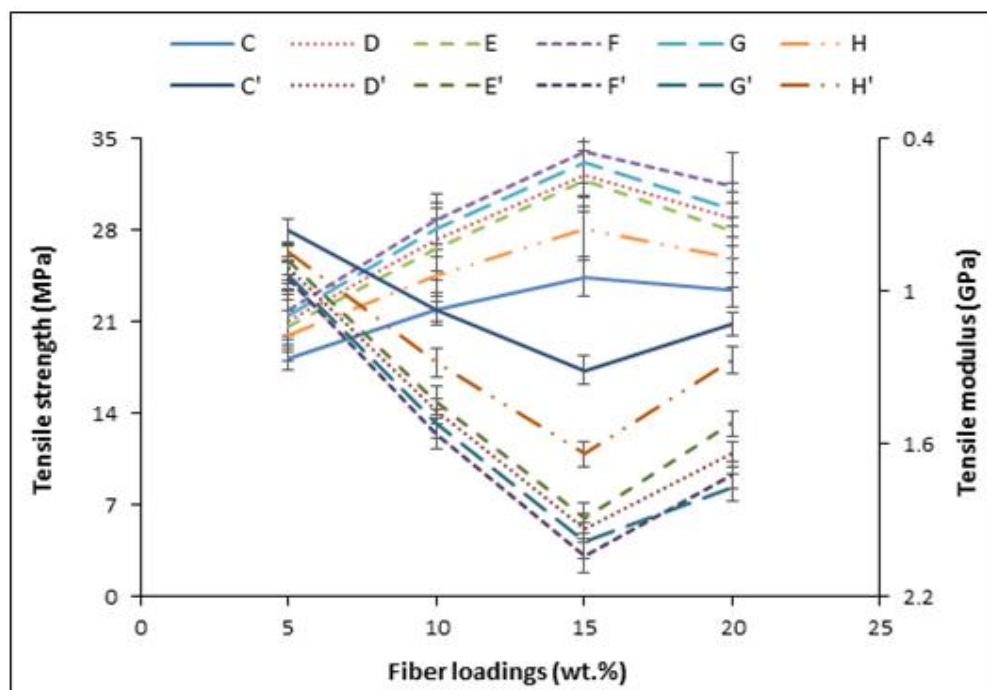


Fig. 2. Variation of TS and TM for C/C': SJPE and D/D': SJPEC-1.28E, E/E': SJPEC-1.30E, F/F': SJPEC-1.31PS, G/G': SJPEC-1.34TCN, and H/H': SJPEC-1.44P

3.3 SEM Study

Scanning electron microscopy (SEM) has been used to investigate the interfacial bonding among jute, PE and MMT nanoclay. Fig. 3 shows the SEM images of prepared silane treated jute composite and nanocomposites. It was seen that there are considerable differences in the interfacial adhesion among the components in composite system. The SEM image of silane treated composite without nanoclay (SJPEC) shows pullout traces of fiber with rough surfaces and micro-voids (Fig. 3I). Whereas, a remarkable improvement in surface morphology was found in the case of clay filled nanocomposite. The highest progress was observed for SJPECC-1.31PS (Fig. 3L) followed by SJPECC-1.34TCN (3M), SJPECC-1.28E (3J), SJPECC-1.30E (3K), and SJPECC-1.44P(3N). The results stated above may be the reason of the MMT nanofillers which increases the bonding with jute and PE. As a result, the less pull out of fibers was observed from the fractured surface [24]. Additionally, the MMT with greater carbon number of modifier that increases the hydrophobicity yield better interfacial adhesion between polymer matrix and MMT [12]. Conversely, MMT with less carbon number of modifier are poorly distributed in the composite system. This can cause of air bubble and agglomerations [13]. Therefore, the SEM micrographs of MMT-1.31PS loaded nanocomposite were exhibited highest improvement of surface morphology.

3.4 TGA Study

Thermogravimetric analysis of the 15 wt% silane treated jute composites with and without different types of nanoclay was used to evaluate thermal properties are shown in Fig. 4 and thermal data are summarized in Table 2. Three stages in maximum rate of decomposition had been found for raw jute composites [25]. Whereas, two stages were observed for prepared nanocomposites in this study. The first stage at 288-330°C attributed to the degradation for lignin and hemicellulose, and the second one appeared at 372-388°C for the degradation of cellulose and other cellulosic materials from the fiber which also leading the char formation [26]. The addition of nanoclay led to the higher thermal degradation as well as higher thermal stability of the nanocomposites. The thermal stability of materials is evaluated with the help of activation energy. The greater activation energy of sample recognizes greater thermal stability. The highest value of activation energy was found for

SJPENC-1.31PS followed by SJPENC-1.34TCN, SJPENC-1.28E, SJPENC-1.30E, and SJPENC-1.44P. This might be due to the addition of MMT with highest carbon number (C_{27}) of modifier among all types of MMT nanoclay that showed reduced hydrophilicity of nanoclay and thus increased interfacial bonding between fiber, polymer matrix and MMT [11,12]. In contrast, MMT with less carbon number of modifier are slightly dispersed in the composite system which cause air bubble and agglomerations [13,14].

It had also been reported earlier that the significant improvement in thermal stability for chemically treated fiber polymer nanocomposites [25]. Thus, the enhanced DSC values are due to the addition of MMT nanoclay as nanofiller with greater carbon number of modifiers, which is reflected on tensile behaviors and SEM images.

3.5 DSC Study

Differential scanning calorimetric (DSC) analysis is carried out to determine the thermal energy released or absorbed via chemical reactions of the fiber constituents during heating. This lead to the exothermic and endothermic reactions. Endothermic reactions provide information on sample melting, phase transitions, evaporation, dehydration and pyrolysis. Exothermic reactions provide information on crystallization, oxidation, combustion, decomposition and chemical reactions [27]. The DSC curves for silane treated jute composites without and with different types of MMT nanoclay were shown in Fig. 5 and the decomposition peaks were found in Table 2. The composites exhibited three decomposition patterns. Firstly, the broad endothermic peaks were observed within 92-104°C which corresponds to the evaporation of absorbed water by the fibers. Secondly, the sharp exothermic peaks were observed within 309-325°C which is related to the decomposition of lignin and hemicellulose from the fibers. Lastly, the endothermic peaks obtained within the range of 372-376°C which shows the degradation of cellulose, leading to char formation [28,29]. It was seen that the decomposition peaks of nanoclay incorporated composites (JPENC) exhibited higher value than raw composite (SJPEC).

It was also observed that the decomposition peaks shifted to higher values with greater carbon number of MMT modifier. The highest value was obtained for SJPENC-1.31PS followed by SJPENC-1.34TCN, SJPENC-1.28E,

SJPENC-1.30E, and SJPENC-1.44P. This is due to the hydrophobicity of MMT which increases as the carbon number of modifier increases and eventually reduces the voids and increases the interfacial adhesion between treated fiber and PE matrix [11,12]. It is stated that the MMT loaded

nanocomposites with greater carbon number of modifier shows better interfacial adhesion among jute, polyethylene and MMT lead to significant improvement in thermal properties which is also reflected on tensile behaviors and evident from SEM images.

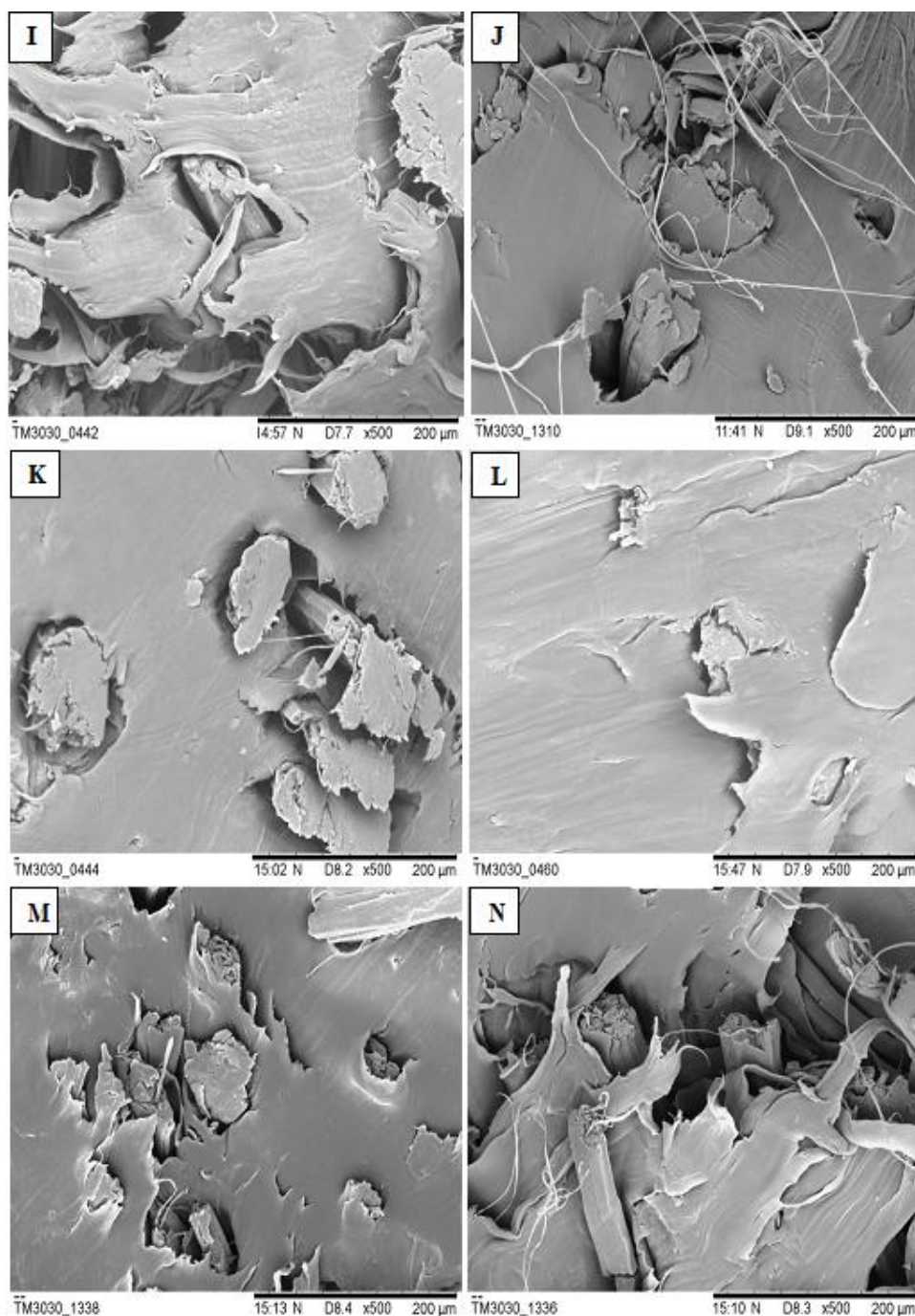


Fig. 3. SEM Images for 15 wt% fiber loaded composites: SJPEC (I), SJPENC-1.28E (J), SJPENC-1.30E (K), SJPENC-1.31PS (L), SJPENC-1.34TCN (M), and SJPENC-1.44P (N)

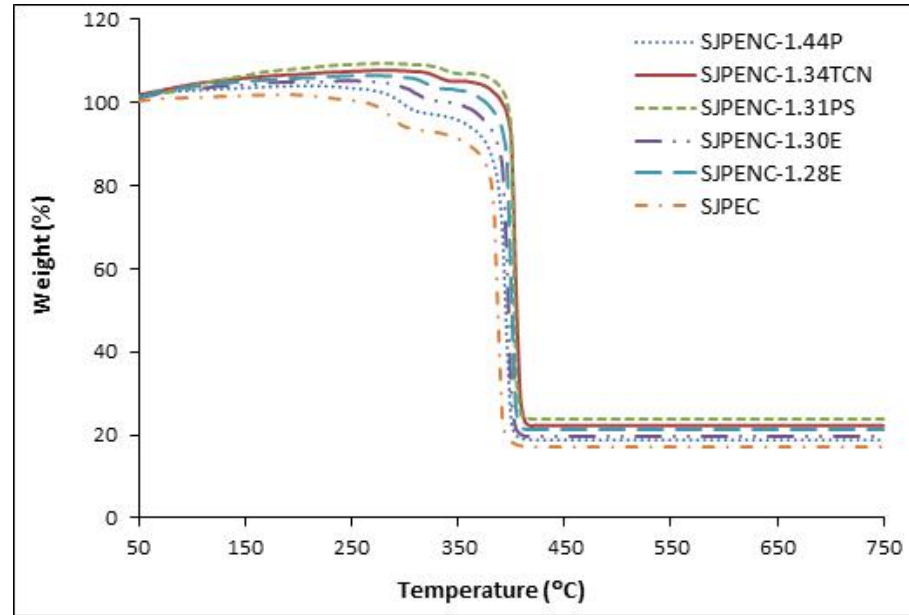


Fig. 4. TGA Curves for 15 wt% fiber loaded composites: SJPEC, SJPENC-1.28E, SJPENC-1.30E, SJPENC-1.31PS, SJPENC-1.34TCN and SJPENC-1.44P

Table 2. Thermal properties of 15 wt% silane treated jute composites without and with different types of MMT nanoclay

Sample	Stage	TGA analysis	DSC analysis		Activation energy E_a (kJ/mol)
		T_{max} (°C)	T_{max} (°C)	Nature of peak	
SJPEC	1 st	----	92.2	Endo	248.2
	2 nd	288.1	309.1	Exo	
	3 rd	371.7	371.7	Endo	
SJPENC-1.28E	1 st	----	100.4	Endo	273.5
	2 nd	321.6	321.6	Exo	
	3 rd	384.2	375.9	Endo	

Sample	Stage	TGA analysis	DSC analysis		Activation energy E _a (kJ/mol)
		T _{max} (°C)	T _{max} (°C)	Nature of peak	
SJPENC-1.30E	1 st	----	100.4	Endo	271.4
	2 nd	317.5	317.4	Exo	
	3 rd	380.1	375.9	Endo	
SJPENC-1.31PS	1 st	----	104.1	Endo	278.6
	2 nd	329.9	325.3	Exo	
	3 rd	388.4	375.9	Endo	
SJPENC-1.34TCN	1 st	----	104.1	Endo	255.3
	2 nd	325.8	321.6	Exo	
	3 rd	388.4	375.9	Endo	
SJPENC-1.44P	1 st	----	96.2	Endo	254.5
	2 nd	296.5	313.2	Exo	
	3 rd	375.9	375.9	Endo	

T_{max}= Temperature for maximum rate of decomposition occur

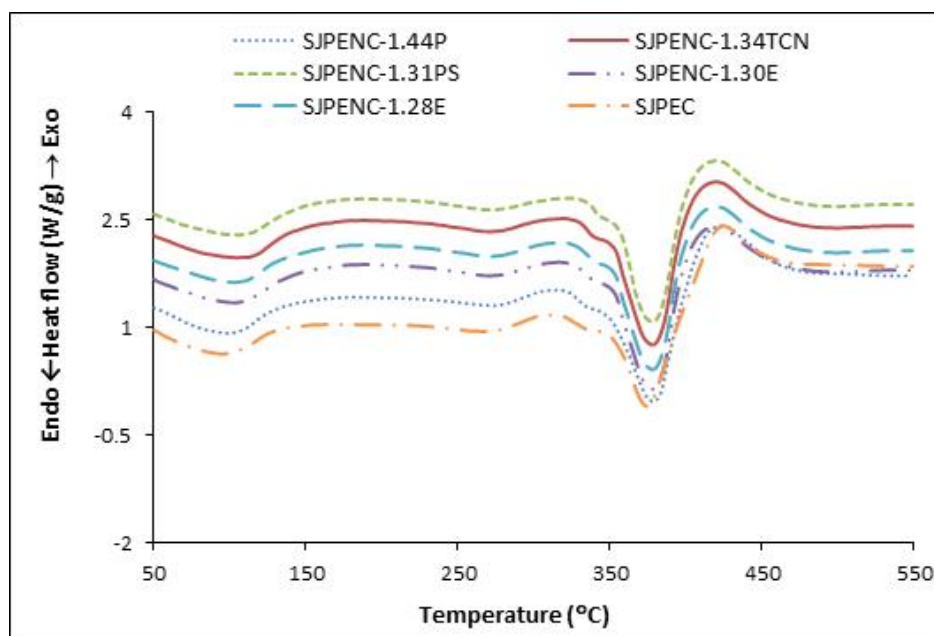


Fig. 5. DSC Curves for 15 wt% fiber loaded composites: SJPEC, SJPENC-1.28E, SJPENC-1.30E, SJPENC-1.31PS, SJPENC-1.34TCN, and SJPENC-1.44P

4. CONCLUSION

Hot press technique was applied for the preparation of silane treated jute composites without and with various MMT nanoclay. Fourier transform infrared (FTIR) spectroscopy and scanning electron microscopy (SEM) were used to study surface characterization. The mechanical and thermal properties were evaluated from TS, TM, TGA and DSC analysis, respectively. The fiber loading have been optimized from tensile values by 15 wt%. From the above analysis, it was found that the MMT nanoclay loaded nanocomposites shows higher tensile values and thermal stability than composite without nanoclay. The higher improvements were observed for SJPENC-1.31PS followed by SJPENC-1.34TCN, SJPENC-1.28E, SJPENC-1.30E, and SJPENC-1.44P. This might be due to the MMT with greater carbon number of modifier that increases the hydrophobicity and eventually increased interfacial adhesion between polymer matrix and MMT. The above findings suggested that the interfacial interaction among fiber, polymer and nanofiller into the composite system has become more satisfactory due to the carbon number of clay modifier. This research may help to prepare nanocomposites using jute with nanoclay and develop the performance of nanocomposites in the interior and exterior structural uses.

DISCLAIMER

The products used for this research are commonly and predominantly use products in our area of research and country. There is absolutely no conflict of interest between the authors and producers of the products because we do not intend to use these products as an avenue for any litigation but for the advancement of knowledge. Also, the research was not funded by the producing company rather it was funded by personal efforts of the authors.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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