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ORIGINAL RESEARCH

Effect of fiber hybridization and montmorillonite clay on properties of treated kenaf/aloe vera fiber reinforced PLA hybrid nanobiocomposite

P. Ramesh · B. Durga Prasad · K. L. Narayana

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Abstract Hybrid fiber reinforced polymer nanobiocomposites were prepared from kenaf fiber, aloe vera fiber, polylactic acid (PLA), and montmorillonite (MMT) clay through the compression molding method. The effects of fiber hybridization and MMT clay on their mechanical, water absorption, thermal and biodegradability properties were studied. Before fabrication, kenaf and aloe vera fibers were treated with the 6% sodium hydroxide solution to improve the bonding nature and compatibility between fibers and PLA matrix. Results indicated that the biocomposites thermal, tensile, flexural, impact, abrasion resistance, and water resistance properties were increased by adding of MMT clay. The mechanical properties were found to be increased upon 15 wt% kenaf fiber, 15 wt% aloe vera fiber hybridization and 1 wt% MMT clay incorporated. In addition, the 1 wt% MMT clay included hybrid nanobiocomposite exhibited increased tensile strength, flexural strength, impact strength, and abrasion resistance by 5.24, 2.46, 37.10, and 23.91%, respectively compared to virgin PLA. Additionally, the tensile and flexural moduli of these nanobiocomposite are improved by 24.61 and 108.09%, respectively, than neat PLA. With the addition of 3 wt% MMT clay resulted in the biocomposite decomposition temperature from 280 to 307 °C at T_{10} likewise 337 to 361 °C at T_{75} SEM analysis disclosed that MMT clay strongly enhances the bonding and compatibility among fibers and PLA. TEM result reveals that the quality of MMT dispersion decreases with increase in MMT content. The fiber hybridization improved the biodegradability and water resistance properties of biocomposites, however, the addition of MMT clay improved water resistance but decreased biodegradability.

Keywords Polylactic acid · Kenaf and aloe vera fiber · Montmorillonite clay · Hybrid nanobiocomposite · Thermo-mechanical properties · Water absorption and biodegradability properties

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Introduction

The improved utilization of plastics throughout the world has been resulted in enhanced plastic waste. The recent developments in recyclable polymers are playing a vital role as today there is an uncertainty of petroleum usage in the world (El-Shekeil et al. 2014). Due to higher cost, the bio-plastics are restricted to medical applications like sutures, drug delivery and orthopedic implant fabrications only (Guo et al. 2007; Chen et al. 2007; Greiner et al. 2007). Among all the bio-plastics or polymers, renewable resource based PLA (Polylactic acid) are more attracted by researchers due to its biodegradability, processability and biocompatibility. It is derived from corn starch or sugar beets by fermentation (Chandra and Rustigi 1998; Bogaert and Coszach 2000; Drumrit et al. 2000). Currently, the PLA biopolymer is not used extensively due to some negative aspects like its properties and high cost. Hence, the natural-fibers and nano clay inclusion in PLA is a right or an alteration system to develop cost-effective material, which has most recently attained thought to substitute carbon and glass fibers (Ochi 2008; Shanks et al. 2006). The foremost problem with natural fiber composites is incompatibility between hydrophobic polymer and hydrophilic natural fiber and poor wetting; it can reduce the thermal and mechanical properties of the composite. However, it can overcome with chemical and physical treatments, or incorporating of additives and compatibilizers (Ramesh et al. 2018a, b; Zhong et al. 2011; Vilay et al. 2008; Saba et al. 2015a, b; Balakrishnan et al. 2010; Mustapa et al. 2013).

The additives like synthetic aramid and glass fibers, nano clay, nano silica and nano tubes may be used to make hybrid composites through suitable material design (Ridzuan et al. 2016). The hybrid composite properties are exclusively managed by factors like matrix, type of filler, fiber dimension, fiber-matrix adhesion and nano filler-matrix adhesion (Saba et al. 2014; Mustapa et al. 2013).

At present, the hybrid composites are receiving extensive attention as the advanced constructional and structural materials with exact balancing better performance and cost effectiveness. The hybrid composite material is developed by combining of two or more dissimilar fibers in a common polymer matrix or reinforced with polymer blends (Sathishkumar et al. 2014; Panneerdhass et al. 2014; Siddika et al. 2013), and it offers the most attracting potential application in non structural, semi structural and structural industrial sectors (Saba et al. 2016).

The hybrid nanotechnology creates the new revolution in the area of material science developing the most high-tech advanced composites for future engineering appliances. The nanocomposites consist of at least one particle which contains nanometer range in dimension. The adding of nano particles demonstrates remarkable enhancement in the thermal, mechanical, physical thermo-mechanical properties due to better distribution, a high specific ratio and effective polymer filler interaction (Dueramae et al. 2014; Mustapa et al. 2013).

With rising ecological safety concern, the natural fibers are more and more in demand across a large of polymer composite appliances (Alwani et al. 2015). The natural fibers are ecologically better alternate to conventional fibers for the reason that they are biodegradable, cheaper, non-toxic, renewable, corrosion resistive, recyclable, abundant, and permeable and competitive properties (El-Shekeil et al. 2014). In the different types of natural resources, the plant fibers are low cost, low density, easy to handle and comparable mechanical properties over synthetic fibers (Sajna et al.2014). Among the plant based fibers kenaf and aloe vera are identified as the most important one for biocomposites study because of its availability, the acceptable specific strength and the low cost. Among the huge amount of residues in the agriculture crop, some quantity of residues is applied in household for domestic and remaining foremost part of residues is burned in the fields; this causes the air-pollution on the environment.

The essential alternative to resolve this dilemma is to apply the agriculture crop residues as reinforcement with polymers is to improve the mechanical properties of the materials. The kenaf plants have the fast growth in past years due to their rapid progress with consequence of low price under the extensive range of climatic circumstances. The kenaf fiber (KF) has a prospective alternating medium to replace the conventional fibers as reinforcement in composites; It diminishes the waste, creates the jobs and contributes healthier atmosphere (Khali et al. 2008; Joshi et al 2004; Aziz and Ansell 2004; Akil and Omar 2011; Yousif et al. 2008; Hong et al. 2008; Joh et al. 2008).

On the other hand, the aloe vera plant (Aloe barbadensis Miller) is also called medicinal plant and it is widely used in food, cosmetic and pharmaceutical or drug industries since thousands of years (Eshun and He 2004). The aloe vera plants are widely cultivated in India, Florida, South Texas, United States i.e. South California, Africa, South and Central America, Australia, Iran and Caribbean (Moghaddasi and Verma

2011). Its look like a sisal plant, but in reality different in nature; these are easily cultivated and low maintenance plant, and ability to stay alive through hard weather circumstances in a rocky terrain and provides economic benefit (Chaitanya and Singh 2016). The aloe vera gel was extracted from the leaves and leaving the outer shell (residue). The aloe vera fiber (AF) was extracted from these leftover leaf shells by using retting method and are currently used in the small textile industry in south India (Kumar and Sekaran 2014).

Chemical or surface modification methods can decrease the KF and AF disadvantages like hydrophilic nature, low thermal resistance, durability, low water resistance and poor compatibility between polymer and fiber. In case of plant fibers, the presence of lignin, wax, pectin and hemicelluloses are expected to hinder the edge bonding between the polymer and fiber. Chemical modification of fiber is a general technique to reduce moisture absorption and improves the adhesion between the hydrophobic polymer matrix and hydrophilic natural fibers (Kabir et al. 2012). In several cases, it is potential to make compatibility between the two materials by introducing maleic anhydride and silane type coupling agent, which can reduce the water absorption in the composites (Xie et al. 2010).

The various chemical modification methods such as alkaline, peroxide, acetylating, silane and benzoylation treatments have been studied in the past (Kabir et al. 2012). Among them, the alkaline treatment (NaOH) is inexpensive, easy and effective method when compared to other methods. Alkaline method expected to remove lignin, wax and pectin on the fiber as well roughen the surface, facilitating superior interlocking and adhesion between the polymer and fiber (Ahmed et al. 2011; Petinakis et al. 2013; Anbukarasi and Kalaiselvam 2015). The NaOH concentration, immersion time and temperature are influences the properties of composites (Ramesh et al. 2018a, b).

At present, the nano particles or fillers such as organically modified montmorillonite (OMMT) and montmorillonite (MMT) clay getting higher consideration as they possess the potential tendency to modify extensively the thermal, mechanical and functional properties of both thermoplastic and thermoset polymers (Souza et al. 2014; Saba et al. 2015a, b). The nanoclays are one of the nano-particles; it consists of layered silicate. The various nanoclays are pyrophyllite, organo clay, hectorite, saponite and nontronite nanoclay, montmorillonite (MMT) clay; among these MMT clay is the most commonly used layer silicate in polymer composites due to its high strength, low cost, high aspect ratio and high modulus (Saba et al. 2015a, b; Jahanmardi et al. 2013).

Several systems are being considered to get better properties and diminish of PLA cost. The produced PLA-biocomposites have been established to be a successful technique to attain ideal properties (Ochi 2008; Yussuf et al. 2010; Chaitanya and Singh 2016). Some scientists have achieved the preferred properties through natural fiber/synthetic fiber hybridization method (Samal et al. 2009; Dhakal et al. 2013). Hammad et al. (2019) concluded that the biodegradability of the cellulose/polyaniline/cobalt ferrite nanocomposite decreases by increasing of cobalt ferrite nanoparticle content. Zainudin et al. (2014) developed the coir/oil palm EFB/PP hybrid composite with enhanced higher mechanical properties. Jacob et al. (2004) investigated the sisal/ oil palm reinforced rubber hybrid composite with improved mechanical properties. Asaithambi et al. (2014) developed and studied the cause of hybridization on flexural, impact and tensile properties of the PLA/banana/sisal biocomposites. Aziz et al. (2018) investigated the effect of clay content on metal adsorption, FTIR, thermal and biodegradable properties of grafting cellulose/clay nanocomposite. Boopalan et al. (2013) examined the thermal and mechanical properties of jute/banana reinforced epoxy hybrid composite. Aziz et al. (2019) researched the influence of hydroxyapatite nanoparticle on water absorption and mechanical properties of RWP/CMC/HA-NPs and BWP/CMC/HA-NPs nanocomposites. El-Sayed et al. (2018) developed the polyaniline/tosylcellulose stearate composite with enhanced mechanical and thermal properties. Youssef et al. (2016) experimented the biological and electrical conductivity of paper sheet based on PANI/PS/Ag-NPs nanocomposite. Islama et al. (2015) studied the physical, biodegradable and mechanical properties of kenaf fiber/coir fiber/polypropylene/ montmorillonite nanoclay hybrid composite. Islam et al. (2017) have reported the influence of fiber hybridization (kenaf/coir/PP) and montmorillonite clay on mechanical, water absorption and biodegradable properties.

It is the evidence from the literature review that no study is carried out on treated kenaf fiber (TKF) and treated aloe vera fiber (TAF) hybridization in the absence of montmorillonite (MMT) clay. So far author's best knowledge this is the first article on TKF/TAF/MMT clay reinforced PLA hybrid nanobiocomposite prepared via compression molding method. Thus, the current study will give innovative information of hybridization with TKF and TAF in the absence of MMT clay to relevant research domain and industry. The PLA/TKF/TAF hybrid biocomposite and the PLA/TKF/TAF/MMT hybrid nanobiocomposite having total 15 wt% of TKF and 15 wt% TAF loading by weight together with 1 and 3 wt% MMT clay loading in PLA/TKF/TAF hybrid nanobiocomposites are fabricated by twin screw-extruder-compression molding method.

The main aim of present research is to investigate the effect of fiber hybridization and MMT clay content on mechanical, water absorption, thermal, and biodegradability properties of PLA/TKF/TAF/MMT hybrid nanobiocomposite. Besides these, the internal bonding behaviour is also obligatory to be investigated in order to synchronize properties. Finally, the fabricated composites were compared with PLA/TKF and PLA/TAF biocomposites and virgin PLA in order to develop an environmental friendly (fully biodegradable) composite with improved performance characteristics.

Experimental

Materials

In this research pellet formed 3052D PLA with 1.24 g/cm^3 at specific gravity, 145–160 °C of crystalline melt temperature, 200 °C of melt temperature and 55–60 °C of glass transition temperature material was acquired from Nature Tech, Chennai, India. The kenaf fiber (KF) and aloe vera fiber (AF) were supplied through Go-Green Products, Chennai, India. The NaOH was supplied by SR-Scientific Chemicals, Tirupati, AP, India. The 1–3 mm long chopped fibers was used. The 1.01 g/cm^3 density MMT clay (Nanomers® I.31PS) by modifying onium ion, powder formed with $\leq$ 20 μm sized was procured from Sigma-Aldrich at Bangalore, India. It contains 0.5–5 wt% aminopropyltriethoxysilane, and 15 to 35 wt% octadecylamine.

Methods

Fiber surface modification

The NaOH treatment method was applied for KF and AF surface modification. The pellet formed sodium hydroxide (NaOH) was provided by S.R.S. Chemicals, Tirupati, India. The KF and AF were immersed in NaOH solution with 6% (w/v) concentration for 3 h at room temperature (Mohd Edeerozey et al. 2007). The TKF and TAF then bathe with flowing distilled water. The pH value kept 7 as a constant. Afterwards, the TKF and TAF are kept in the oven at 100 °C for 8 h.

Lignocellulosic composition

Lignocellulosic compositions of treated and untreated KF and AF in terms of cellulose, lignin, hemicellulose, and extractive were determined using standard analytical test method (Li et al. 2004; Anil and Prasenjit 2016).

Preparation of PLA based composites

The produced composites formations are presented in Table 1. Prior to the fabrication of sample PLA, TKF, TAF and MMT clay are held in an oven at 110 °C for 1 h. TKF and TAF were hybridized by mixing them in 1:1 ratio, while 1 and 3 wt% is added into the fiber/PLA mixture for manufacturing hybrid nanobiocomposites. The PLA, TKF, TAF, and MMT clay are physically pre-mixed according to Table 1 and then compounded through (ZV 20 model) twin-screw extruder. The screw diameter and L/D ratio are 21 mm and 40, respectively. For compounding of all PLA based composites (Table 1), screw speed and temperature profile were set to 78 rpm and 155 °C to 190 °C, respectively. Then, the compounded pellets are kept in oven for dry at 80 °C for 4 h. After drying, the compound pellets are pre-melted at 185 °C in a counter rotating two roll mill internal mixer through a revolve speed of 50 rpm.

Afterwards, the compounded pellets are processed through compression molding machine. During the process, keep the temperature 185 °C and 30 ton force applied (up stroke) for 10 min, and then compacted at 165 bar pressure for 30 min followed by cooling under pressure. When the mold temperature is reached at 90 °C the platens are opened from the press; then

Table 1 Compositions and composite names

Code	Sample	PLA, wt%	TKF, wt%	TAF, wt%	MMT, wt%
P	PLA	100	0	0	0
S	PLA/TKF	70	30	0	0
A	PLA/TAF	70	0	30	0
H	PLA/TKF/TAF	70	15	15	0
H1	PLA/TKF/TAF/1MMT	69	15	15	1
H3	PLA/TKF/TAF/3MMT	67	15	15	3

the composite sheets (200 mm × 200 mm × 3 mm) are removed from platens and cut to desired form for tensile, flexural, impact, abrasion resistance, thermogravemetric analysis, water absorption and biodegradability evaluations. The virgin PLA sheet is produced through two roll mill and compression molding.

Mechanical characterizations

Tensile test of neat PLA and PLA based composites were carried out by Instron-3369 universal testing machine (UTM), USA according to ASTM D638 at 25 °C. Speed of the cross head was 10 mm/min. Flexural test of composites was also performed through the same UTM (50 kN load and 50 mm span length) according to ASTM D790-03 at 18 °C. Impact test of composites was finding through the Izod testing machine according to ASTM D256. For impact test, the maximum applied energy was 18 J. Abrasion resistance test was carried out through abrasion machine according to ASTM D1044 standards for 100 cycles.

Morphological characterizations

JSM-IT500 scanning electron microscope (SEM), Japan Electronics Optic Limited, USA, was used to examine the fracture surface of the tensile fracture specimen. Before the examination, the specimens were sputter coated with gold. Transmission electron microscope (TEM) of hybrid nanobiocomposites was examined by using a Philips CM 300 TEM (FEI Company, USA) at an accelerating voltage of 200 kV. Thin sections with thickness of 80 nm were cut from the fractured specimen by using diamond knife equipped ultramicrotome (Leica EM UC7) for consequent TEM observations.

Thermal characterizations

The PLA and PLA based composites thermal stability was carried out through TGA (Thermogravemetry analysis) by Perkin Elmer instrument. The samples (5–30 mg) were heated from 30 to 800 °C with a heating rate of 10 °C/minute under nitrogen gas flow.

Water absorption test

The water absorption test was carried out through direct immersion of neat PLA and PLA based composites in normal water at room temperature with dimension 10 mm × 10 mm × 3 mm for up to 30 days (Anuar et al. 2010). All specimens were first dried using a tissue to remove any excess water, followed by weighing using a digital scale. The weight gain of samples was calculated through the following Eq. (1).

$$\text{Water absorption } (\%) = \left(\frac{\text{W2} - \text{W1}}{\text{W1}}\right) \times 100 \quad (1)$$

where W_2 and W_1 are after and before immersion mass of the sample.

Biodegradability study

The biodegradability study of samples (1 cm × 1 cm) was performed through simple soil burial examination. Each sample weighed and then the samples are buried in ordinary soil in the garden at an average temperature 30 °C and 80% humidity for 10, 30 and 90 days (Yussuf et al. 2010). The humidity was kept to about 80% by regular watering. The mass loss percentages of biocomposites were calculated for 10, 30 and 90 days through the following Eq. (2).

$$\text{Weight loss } (\%) = \left(\frac{\text{Wi} - \text{Wf}}{\text{Wi}}\right) \times 100 \quad (2)$$

where Wi and W_f are before and after mass of the sample.

Results and discussions

Lignocellulosic composition

The lignocellulosic compositions of raw and treated KF and AF in terms of their cellulose, lignin, hemicellulose, and extractive were depicted in Table 2. It could be inferred from Table 2 that the KF and AF cellulose content were increased and hemicellulose content was decreased with sodium hydroxide (NaOH).

Mechanical characterizations

Tensile properties

Figure 1a-b presents the tensile strength and modulus of the PLA/TKF and PLA/TAF biocomposites, PLA/TKF/TAF hybrid biocomposite and PLA/TKF/TAF/MMT hybrid nanobiocomposites. Figure 1a shows that the tensile strength increased from 48.75 to 53.88 MPa when TAF was used instead of TKF. The PLA/TKF/TAF hybrid biocomposite tensile strength was depicted 54.36 MPa which was 11.51 and 0.89% higher than PLA/TKF and PLA/TAF biocomposites, respectively. Similarly, the fiber hybrid biocomposite (4.52 GPa) tensile modulus was depicted 24.72% higher than PLA/TKF biocomposite (3.63 GPa) and 38.75% lower than PLA/TAF biocomposite (3.28 GPa). The tensile strength of PLA/TKF biocomposite was increased from 48.75 to 54.36 MPa after TAF hybridization.

Additionally, the PLA/TKF, PLA/TAF and PLA/TKF/TAF hybrid biocomposite tensile modulus were improved 39.23, 25.76 and 73.46%, respectively than neat PLA. For finding natural fiber tensile strength cellulose content is played a vital role due to its high degree of polymerization and resistance, and linear orientation (Thakur and Thakur 2014; Maya et al. 2006). The TKF had a low amount of cellulose when compared to TAF; low amount of cellulose leads low tensile strength. Figure 1a-b shows that the optimum tensile properties bright to produce with fiber hybridization (Singh et al. 2010). This is confirmed that the elevated tensile properties of TAF are able to bear the low tensile properties of TKF. Figure 1a-b presents the hybrid nanobiocomposites tensile properties. The tensile strength of neat PLA was diminished with the addition of natural fibers. The PLA/TKF, PLA/TAF and PLA/TKF/TAF hybrid biocomposites tensile strength was decreased 14.57, 5.57 and 4.73% respectively than neat PLA. According to Yang et al. (2004) and Ismail et al. (2001) this diminishes was recognized due to the irregular shapes, fiber inability, stresses transferred (support from matrix), micro-voids and various processing procedures (Huda et al. 2006).

Also, the inclusion of 1 wt% MMT clay into PLA/TKF/TAF/MMT hybrid nanobiocomposite was leading to the modest improvement in tensile strength as shown in Fig. 1a. The tensile strength of 1 wt% MMT clay included PLA/TKF/TAF nanobiocomposite was improved 23.20, 11.46, 10.47 and 5.24%, respectively than PLA/TKF, PLA/TAF, PLA/TKF/TAF and neat PLA. As the MMT clay content beyond 1% (addition of 3%) by weight tensile strength was decreased 14.05% than neat PLA. The incorporation of the lower amount of MMT clay was reduced the micro-voids, uniform dispersion, and improved interaction, compatibility and adhesion among fiber-nano-matrix (Xu and Hoa 2008), which was enlarged the highest load transfer capacity. In another case, the addition of 3 wt% MMT clay included PLA/TKF/TAF nanobiocomposites was shown diminish tensile strength by having micro-voids and agglomerations formations; these were reduced both fiber-PLA bonding and load transfer capacity; similar mechanism observed in previous studies (Hakamy et al. 2013; Feng et al.

Table 2 Lignocellulosic compositions of raw and treated KF and AF

Natural Fiber	Extractive (%)	Hemicellulose (%)	Cellulose (%)	Lignin (%)
6% NaOH treated KF	0	20.2	62.73	17.07
Raw KF	6.1	25.9	50.93	17.07
6% NaOH treated AF	0	17.5	66.03	16.47
Raw AF	4.95	22.55	56.03	16.47

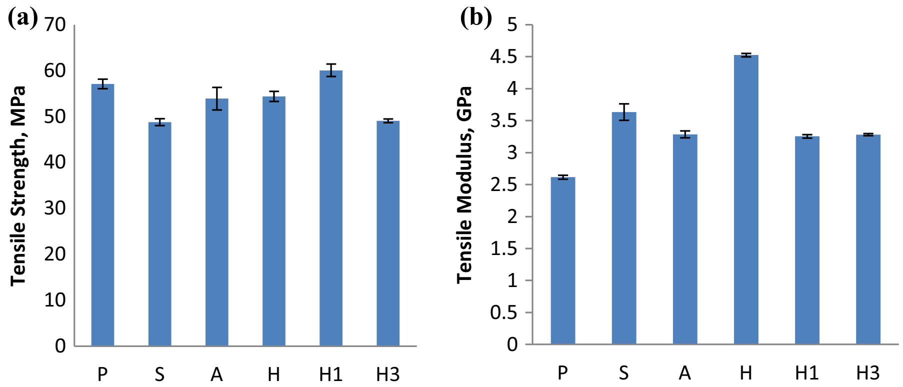


Fig. 1 Tensile **a** strength and **b** modulus of P, S, A, H, H1 and H3 composites

2014). These obtained results were confirmed with those obtained results by Ramesh et al. (2019) in their investigation, they conclude that 1 wt% MMT clay based PLA/treated aloevera fiber hybrid nanobiocomposite was shown the improved tensile properties than 2 and 3 wt% MMT clay based PLA/treated aloevera fiber hybrid nanobiocomposites. Alamri et al. (2012) studied and concluded that 1% nano filler epoxy based nanocomposites demonstrate enhanced mechanical properties than other (3 and 5 wt% nano filler) nanocomposites. Patel et al. (2018) found that bamboo /polyester with 1 wt% containing nano clay hybrid composites tensile strengths were improved; beyond 1 wt% nano clay the properties are diminished. Lei et al. (2007) observed that the higher amount of clay (beyond 1%) was affected the tensile strength of the wood/HDPE composites.

Tensile modulus was used as a sign for stiffness of the material (Chaitanya and Singh 2016). Figure 1b presents the tensile modulus of PLA/TKF/TAF/MMT hybrid nanobiocomposites. The 1 wt% MMT clay included PLA/TKF/TAF nanobiocomposite had been enhanced the tensile modulus by 24.61% over neat PLA. However, the presence of fiber hybridization had a marvellous effect on tensile modulus for PLA/TKF/TAF hybrid biocomposite; it was increased by 73.46% over neat PLA.

Flexural properties

Figure 2a-b shows the various manufactured composites flexural properties. Figure 2a presents that the flexural strength of PLA/TKF and PLA/TAF biocomposites were 68.24 and 94.76 MPa; when TAF was used instead of TKF flexural strength was improved to 94.76 MPa. The flexural strength of PLA/TKF/TAF hybrid biocomposite was demonstrated 40.20 and 0.95%, respectively higher than PLA/TKF and PLA/TAF biocomposites; that were attributed due to higher cellulose content in TAF. PLA/TKF biocomposite tensile strength was increased from 68.24 to 95.66 MPa after TAF hybridization. The flexural modulus of PLA/TKF, PLA/TAF and PLA/TKF/TAF hybrid biocomposite was improved 67.85, 61.42 and 69.52% respectively than neat PLA. The optimum flexural properties were able to develop by fiber hybridization; it was confirmed through bear the low flexural properties of TKF by TAF.

The PLA based composites flexural properties were illustrated in Fig. 2a-b. The flexural properties of neat PLA were declined with the addition of fibers, but increased with MMT clay. The virgin PLA flexural strength was leading towards the enhancement with 1wt% MMT clay as shown in Fig. 2a. The 1 wt% MMT included PLA/TKF/TAF nanobiocomposite flexural strength was improved 56.43, 12.63, 11.57 and 2.46%, respectively than PLA/TKF, PLA/TAF, PLA/TKF/TAF and neat PLA because of lower micro-voids, uniform dispersion and adhesion among fiber-nano-matrix (Xu and Hoa 2008); beyond 1% (addition of 3%) by weight which was decreased 31.55% than neat PLA due to micro-voids and agglomerations formations. The similar mechanism was observed in previous studies (Hakamy et al. 2013; Feng et al. 2014). These obtained results were confirmed with those were obtained results by Alamri et al. (2012) in their study, they concluded that 1% nano filler epoxy based nanocomposite exhibited enhanced flexural properties than 3 and 5 wt% based nanocomposites. Ramesh et al. (2019) researched and they conclude

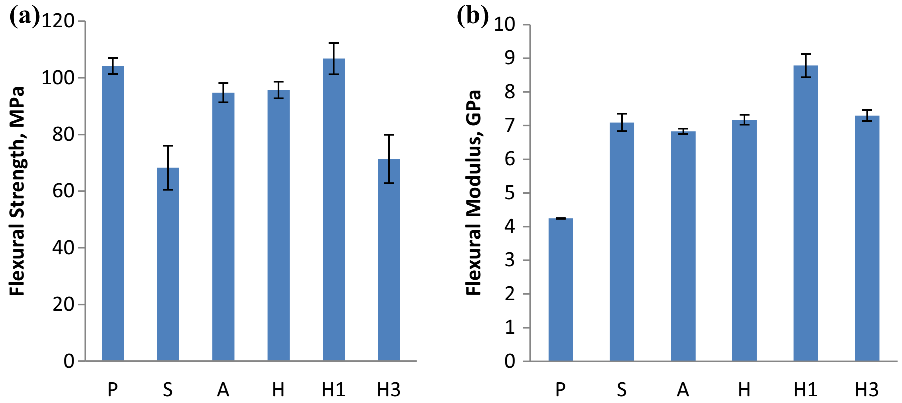


Fig. 2 Flexural **a** strength and **b** modulus of P, S, A, H, H1 and H3 composites

that 1 wt% MMT clay based PLA/treated aloe vera fiber hybrid nanobiocomposite shows enhanced flexural properties than 2 and 3 wt% MMT clay based PLA/treated aloe vera fiber hybrid nanobiocomposites. Bozkurt et al. (2007) reported that the glass fiber/nano clay/epoxy nanocomposite flexural properties were enhanced due to good bonding among matrix and fiber. Zulfli and Shyang (2010) examined that the nano clay was enhanced the flexural properties of epoxy/clay nanocomposite. The nanoclay presence was believed to enhance the adhesion between the epoxy and glass fiber (Zulfli and Shyang 2010).

The sign for material stiffness was used flexural modulus in static yielding condition (Chaitanya and Singh 2016). Figure 2b also presents the flexural modulus of PLA/TKF/TAF/MMT composites. The flexural modulus of PLA/TKF/TAF hybrid biocomposite and 1, 3 wt% MMT included PLA/TKF/TAF/MMT nanobiocomposites were improved 69.52 and 108.09, 72.61%, respectively than neat PLA. However, the presence of 1 wt% MMT clay had an outstanding effect on flexural modulus of PLA/TKF/TAF composite; it was improved 108.09% than neat PLA.

Impact properties

The unreinforced PLA and PLA based composites impact properties are illustrated in Fig. 3. The impact strength of PLA/TKF/TAF hybrid biocomposite was demonstrated 40.46 and 37.57%, respectively higher than PLA/TKF and PLA/TAF biocomposites; that were attributed due to higher cellulose content in TAF. PLA/TKF biocomposite tensile strength was increased from 49.34 to 69.29 kJ/m^2 after TAF hybridization.

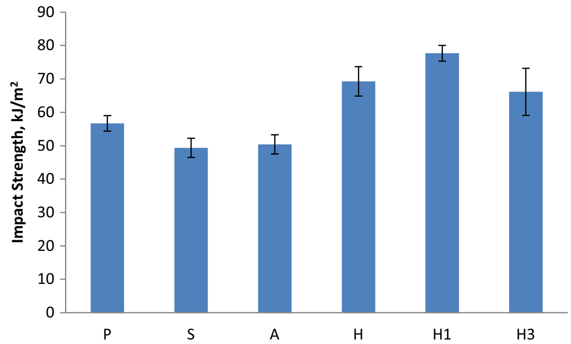


Fig. 3 Impact strength of P, S, A, H, H1 and H3 composites

The optimum impact properties were able to develop by fiber hybridization; it was confirmed through balance the low impact properties of TKF with TAF. As shown in Fig. 3 the addition of fibers into PLA impact properties was declined, but in addition of MMT clay it could be increased. Impact strength was leading towards enhancement with presence of MMT clay; in addition the 1 wt% MMT clay included PLA/TKF/TAF composite impact strength was enhanced 57.50, 54.27, 12.13 and 37.10%, respectively than PLA/TKF, PLA/TAF, PLA/TKF/TAF and neat PLA because of lower micro-voids, uniform dispersion, and adhesion among fiber-nano-matrix (Xu and Hoa 2008); beyond 1% (addition of 3%) by weight which was decreased 14.88% than 1 wt% MMT clay included nanobiocomposite due to micro-voids and agglomerations formations.

The addition of 1 wt% MMT clay included PLA/TKF/TAF composites was shown the improved mechanical properties because of high matrix-fiber bonding strength, which was enlarged the highest load transfer capacity. In another case, the addition of 3 wt% MMT clay included PLA/TKF/TAF composite was shown the diminish mechanical properties by having micro-voids and agglomerations formations; these were reduced both fiber and PLA bonding and load transfer capacity; similar mechanism was observed in previous studies (Ramesh et al. 2019, 2020; Hakamy et al. 2013; Feng et al. 2014). The fiber hybridization and MMT clay content was played a vital role in improved performance of PLA/TKF/TAF/MMT hybrid nanobiocomposites, the lowered content enhanced higher mechanical properties.

Abrasion resistance properties

Performed neat PLA and PLA based composites abrasion resistance results are shown in Fig. 4. The PLA/TKF and PLA/TAF biocomposites abrasion resistance were increased than neat PLA. The both PLA biocomposite abrasion resistance was equally decreased 62.85% after fiber hybridization. Additionally, the PLA/TKF/TAF hybrid biocomposite abrasion resistance is decreased by 23.91% over neat PLA but with the addition of MMT clay it can increase. It is obvious that 1 wt% MMT clay included PLA/TKF/TAF hybrid nanobiocomposite (H1) shows higher abrasion resistance than other; the improvement was 23.91% over neat PLA. The 1wt. % MMT clay improves the bonding between PLA, and TKF and TAF; it was evident from mechanical and morphological results. After more than 1 wt% MMT clay affects the abrasion resistance of PLA/TKF/TAF composite. The similar effect was observed in previous work (Ramesh et al. 2019, 2020; Brostow et al. 2017). The mechanical results also followed the same trend.

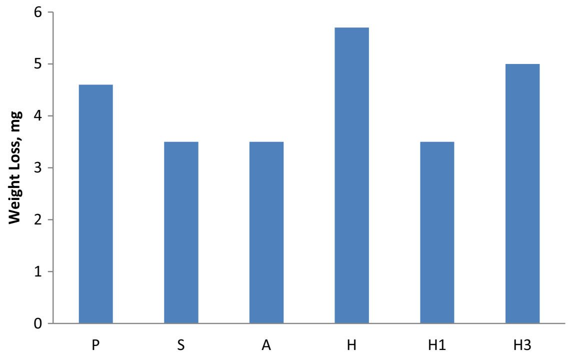


Fig.4 Abrasion resistance of P, S, A, H, H1 and H3 composites

Morphological properties

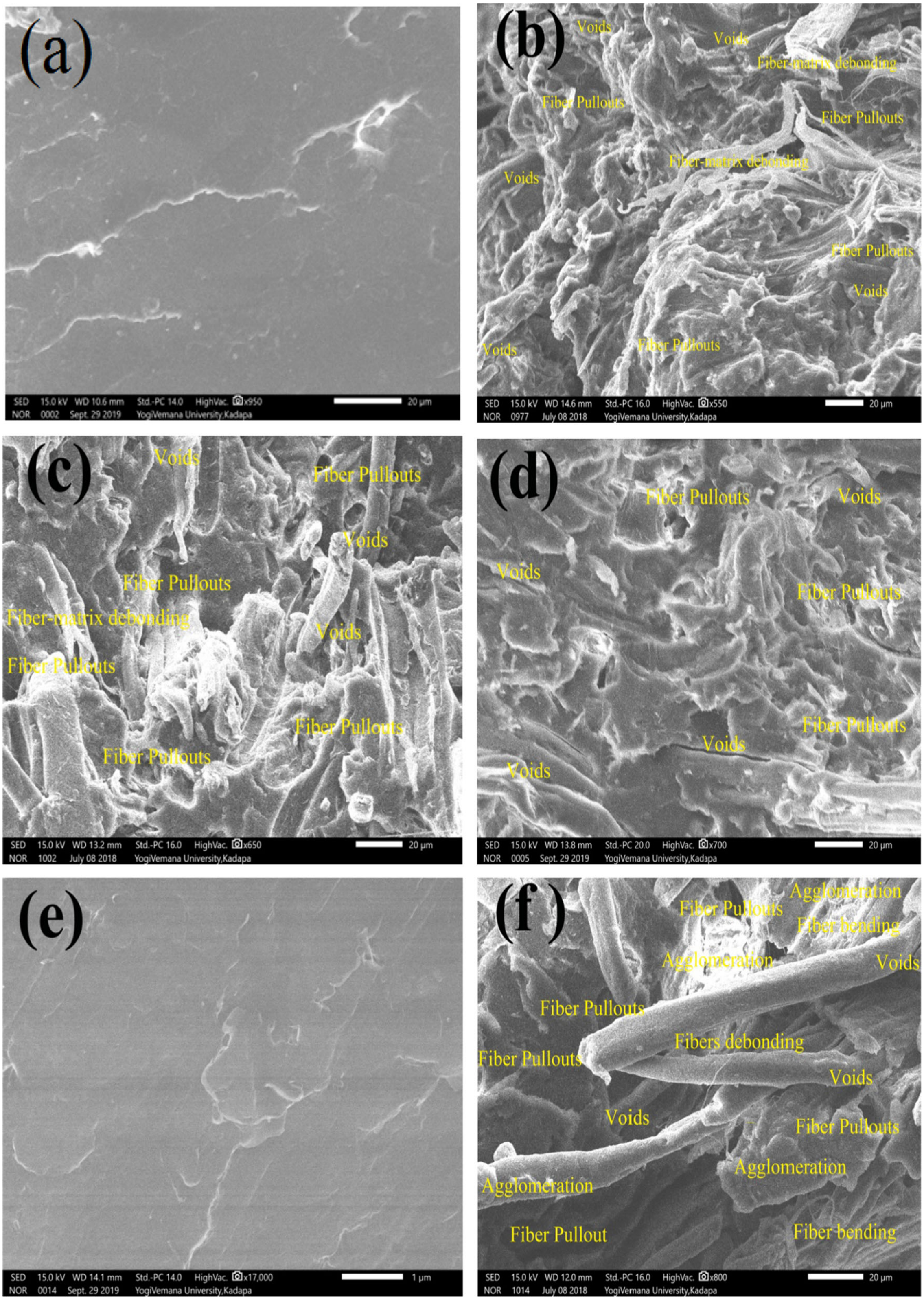
Scanning electron microscopy images

The SEM micrographs of virgin PLA, PLA/TKF, PLA/TAF, PLA/TKF/TAF hybrid biocomposites and PLA/TKF/TAF/MMT hybrid nanobiocomposites are presented in Fig. 5a–f. Multiplicities of failure mechanisms such as fiber pullouts, fiber-matrix debonding, voids, agglomerations and fiber bending were observed. Figure 5a shows the fractured surface of the unreinforced PLA, which was illustrated a regular and smooth surface and displays no significant defect on the fractured surface. The PLA/TKF and PLA/TAF biocomposites (Fig. 5b, c) were shown de-bonding between the fabrics and matrix, pullouts and voids. The interaction and bonding were not better after fiber hybridization (Fig. 5d).

However, the 1 wt% MMT clay included PLA/TKF/TAF hybrid nanobiocomposite (Fig. 5e) shows that the interaction are appreciably improved and thus the micro-voids, fiber fracture and pullouts were significantly reduced from the surface. After incorporation of MMT smooth fracture surface which was indicated good compatibility, interaction and bonding between TKF and TAF with PLA (Ramesh et al. 2019, 2020; Qutubuddin and Fu 2002). Thus, it could be believed that MMT clay had the ability to improve compatibility and adhesion of PLA with TKF and TAF. This result turn was enhanced the mechanical strength of 1 wt% MMT clay included PLA based hybrid nanobiocomposite. Addition of 3 wt% MMT clay included PLA/TKF/TAF hybrid nanobiocomposite was demonstrated a number of agglomerations, voids, fiber bending and pullouts (Fig. 5f).

Transmission electron microscopy (TEM) images

TEM images of PLA/TKF/TAF/1MMT and PLA/TKF/TAF/3MMT hybrid nanobiocomposites are shown in Fig. 6a-b. It can be seen that the MMT clay are uniformly dispersed within the PLA matrix (6a). However, the higher number of agglomerations can be clearly observed in the larger MMT clay content (6b). This observation clearly suggests that the quality of MMT clay dispersion decreases as MMT clay content increases. The similar examination was made by previous investigation (Alamri et al. 2012).


(a)
SED 15.0 kV WD 10.6 mm Std.-PC 14.0 HighVac. x950 20 µm
NOR 0002 Sept. 29 2019 YogiVemana University,Kadapa
(b)
Voids
Voids
Fiber-matrix debonding
Fiber Pullouts
Fiber Pullouts
Fiber-matrix debonding
Voids
Fiber Pullouts
Voids
Voids
Fiber Pullouts
SED 15.0 kV WD 14.6 mm Std.-PC 16.0 HighVac. x550 20 µm
NOR 0977 July 08 2018 YogiVemana University,Kadapa
(c)
Voids
Fiber Pullouts
Voids
Fiber Pullouts
Fiber-matrix debonding
Voids
Fiber Pullouts
Fiber Pullouts
Fiber Pullouts
SED 15.0 kV WD 13.2 mm Std.-PC 16.0 HighVac. x650 20 µm
NOR 1002 July 08 2018 YogiVemana University,Kadapa
(d)
Fiber Pullouts
Voids
Voids
Fiber Pullouts
Fiber Pullouts
Voids
Voids
SED 15.0 kV WD 13.8 mm Std.-PC 20.0 HighVac. x700 20 µm
NOR 0005 Sept. 29 2019 YogiVemana University,Kadapa
(e)
SED 15.0 kV WD 14.1 mm Std.-PC 14.0 HighVac. x17,000 1 µm
NOR 0014 Sept. 29 2019 YogiVemana University,Kadapa
(f)
Agglomeration
Fiber Pullouts
Fiber bending
Agglomeration
Voids
Fiber Pullouts
Fibers debonding
Fiber Pullouts
Voids
Voids
Fiber Pullouts
Agglomeration
Agglomeration
Fiber Pullout
Fiber bending
SED 15.0 kV WD 12.0 mm Std.-PC 16.0 HighVac. x800 20 µm
NOR 1014 July 08 2018 YogiVemana University,Kadapa

◀**Fig. 5** SEM images of **a** P, **b** S, **c** A, **d** H, **e** H1 and **f** H3 composites

Thermal characterization or TGA analysis

Thermal stability of developed virgin PLA and PLA based composites was determined using thermo gravimetric analysis (TGA). Figure 7 shows the neat PLA and PLA based composites TGA curve.

The hybridization improved thermal stability of biocomposite as evidenced from thermo gravimetric curve (Fig. 7). The onset temperature of 10 and 75% weight loss deviation from the baseline (T_{10} and T_{75} respectively) are used as the indicator for analyzing thermal stability of manufactured biocomposites which is based on previous research (Yussuf et al. 2010). The PLA/TKF biocomposite was improved from 280 to 291 °C and 337 to 343 °C after hybridization respectively at T_{10} and T_{75} Table 3 shows the T_{10} and T_{75} of the PLA and PLA based composites.

The Fig. 7 illustrates that the high thermal stability of TAF was able to balance the low thermal stability of TKF. The decomposition takes place in three-stages. In the primary stage moisture evaporation occurred up to 150 °C and in second phase due to lignin, cellulose and hemi celluloses. Finally, depolymerisation of PLA takes place. Generally, common trend was the polymer thermal stability decrement with the adding of natural fiber (Yussuf et al. 2010; Ohkita and Lee 2006; El-Shekeil et al. 2012). The virgin PLA thermal

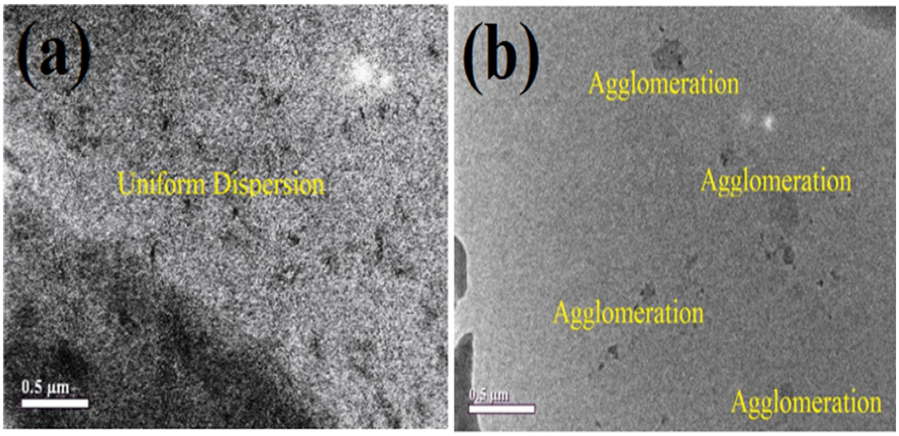


Fig. 6 TEM images of **a** H1 and **b** H3 composites

Fig. 7 TGA curves of P, S, A, H, H1 and H3 composites

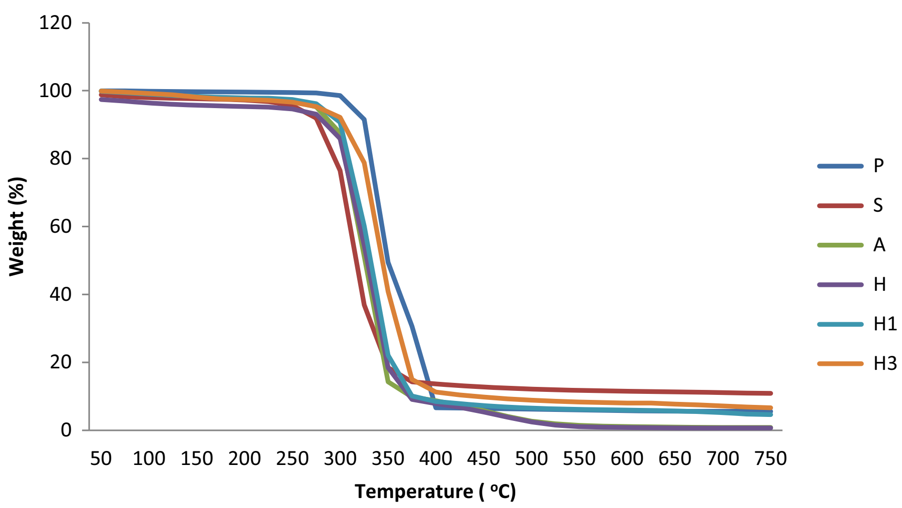


Table 3 TGA Characterization of P, S, A, H, H1 and H3 composites

Code	Sample	Weight loss, decomposition temperature (°C)	
		10 (%)	75 (%)
P	PLA	327	358
S	PLA/TKF	280	337
A	PLA/TAF	295	338
H	PLA/TKF/TAF	291	343
H1	PLA/TKF/TAF/1MMT	301	346
H3	PLA/TKF/TAF/3MMT	307	361

stability diminishes with adding of TAF and TKF but it can improve with adding of MMT clay.

It is obvious that 3 wt% MMT clay included PLA/TKF/TAF composite shows higher thermal stability than other nanobiocomposite; the improvement from 280 to 307 °C at T_{10} likewise 337 to 361 °C at T_{75} For 10 and 75% weight loss the pure PLA degrades at 327 and 358 °C respectively. From the results 3 wt% MMT clay included PLA/TKF/TAF hybrid nanobiocomposite shows higher thermal stability than neat PLA; the improvement from 358 to 361 °C at T_{75} This enhancement takes place due to MMT clay; it was acted as a barrier, constrained the mobility to chain and hinders decomposition process. The similar enhancement was observed in previous studies (Ramesh et al. 2019, 2020; Ismail et al. 2008; Madaleno et al. 2010; Yeh et al. 2006). The MMT clay was enhanced the thermal stability of PLA based biocomposites.

Water absorption test

The conducted water barrier properties of PLA and PLA based composites were displayed in Fig. 8. All samples absorption gain percentages were found with respect to submerging period. At the beginning period, the water absorption of all samples were improved significantly and then reached to equilibrium. Figure 8 shows that water gain percentage was increased with increase of submerging time. The water resistance of PLA/TAF biocomposite was increased after hybridization. The Fig. 8 illustrates that the higher water resistance properties of TKF was able to bear the low water resistance properties of TAF. The virgin PLA water absorption was increased by adding of TKF and TAF.

However, the incorporation of MMT clay effectively raises the water resistance of PLA based composites. The water resistance increased maximum with continuous adding of MMT clay. This phenomenon attributed because of fiber treatment and MMT clay presence in PLA/TKF/TAF/MMT hybrid nanobiocomposites; the clay acted as barrier medium and it restricts flow of water into biocomposites in all path ways, thus resulting in lower water uptake as described in the literature (Deka and Maji 2011; Zhao and Li 2008; Liu et al. 2005). As consider the effect of MMT clay on the water resistance of hybrid biocomposite, the 3 wt% MMT clay contained PLA/TKF/TAF composite exhibited excellent water resistance property than other composites. This could be attributed due to the barrier effects of the MMT clay, thus resulting in a decreased the water absorption. The similar examination was made by previous research (Alamri and Low 2013; Ramesh et al. 2019, 2020; Sajna et al. 2014).

Biodegradability test

Biodegradability test of the samples were performed without any composting and enzymatic material through simple soil burial investigation test (Yussuf et al. 2010). All samples weight loss percentages were found with respect to burial period time. Biodegradability (weight loss percentage) of the PLA and PLA based composites are improved with burial test time. Figure 9 shows the rate of biodegradability of PLA, PLA/TKF, PLA/TAF, PLA/TKF/TAF hybrid biocomposites and PLA/TKF/TAF/MMT hybrid nanobiocomposites with respect to time. The virgin PLA biodegradability was increased with adding of TKF and TAF. The PLA/TKF/TAF hybrid biocomposite had highest biodegradability or weight loss followed by PLA/TKF, PLA/TAF and virgin PLA. It was confirmed that after fiber hybridization composites

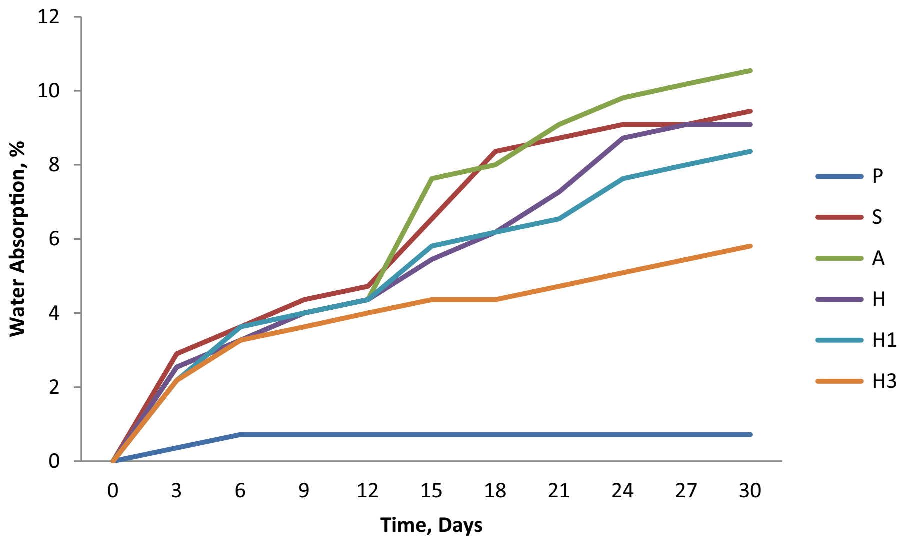


Fig. 8 Water absorption test of P, S, A, H, H1 and H3 composites

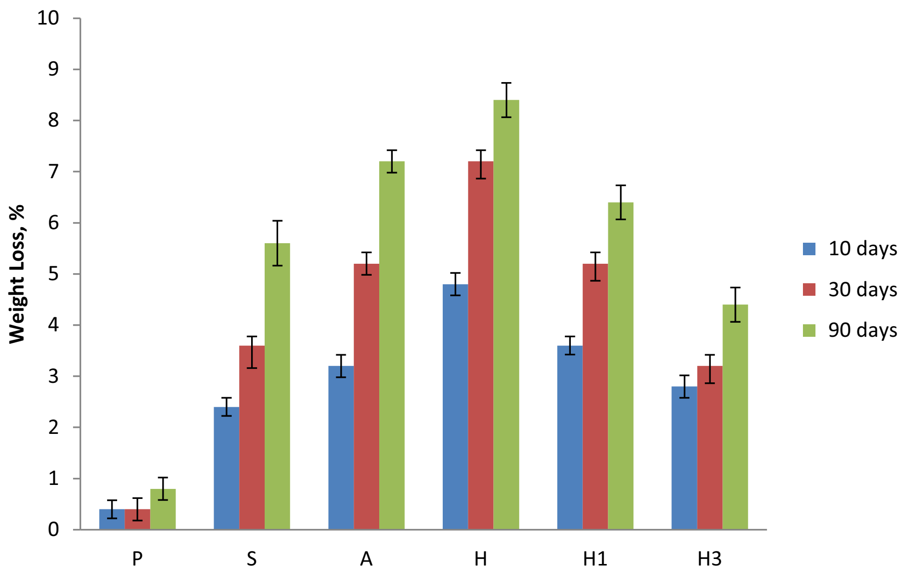


Fig. 9 Biodegradability test of P, S, A, H, H1 and H3 composites

physical properties were enhanced (Bledzki and Gassan 1999).

Then again, the addition of MMT clay into fiber hybridization the biodegradability was decreased. However, the incorporation of 3 wt% MMT clay negative effect on biodegradability properties of PLA/TKF/TAF composite. The higher amount of MMT clay content was leading to agglomeration, it raised owing to attractive force between PLA and MMT clay. Similar examination was made by previous investigation (Ramesh et al. 2019, 2020; Islam et al. 2017). However, the 1 wt% MMT clay included PLA/TKF/TAF composite was shows improved biodegradability properties than PLA/TKF, PLA/TKF/TAF/3MMT composites and unreinforced PLA.

Conclusions

The effect of fiber hybridization and MMT clay on mechanical, thermal, water absorption and biodegradable properties of PLA/TKF/TAF/MMT hybrid nanobiocomposites have been reported. The mechanical and thermal properties were found to be increased upon TKF, TAF hybridization and MMT clay incorporated. The optimum content of MMT clay was found to be 1 wt% for mechanical properties. The 1 wt% MMT included PLA/TKF/TAF/MMT hybrid nanobiocomposite exhibited increased tensile, flexural, impact, and abrasion resistance properties than neat PLA and other composites due to homogeneous dispersion of MMT clay in PLA matrix which was confirmed through TEM results. These remarkable improvements in PLA/TKF/TAF/MMT hybrid nanobiocomposite properties could be attributed to strong interactions of TKF, TAF and MMT clay, which was confirmed through SEM results. However, the tensile, flexural and impact strengths of PLA/TKF/TAF/MMT hybrid nanobiocomposite are negatively affected when the excess MMT clay was added. The PLA/TKF biocomposite thermal stability increased after TAF hybridization. The PLA/TKF/TAF/3MMT hybrid nanobiocomposite exhibited higher thermal stability. The scanning electron microscope analysis discloses that MMT clay strongly enhanced the bonding and compatibility among TKF, TAF and PLA. The fiber hybridization improved the biodegradability and water resistance properties of composites. The higher content of MMT clay minimizes the biodegradability rate and maximizes the water resistance of PLA/TKF/TAF/MMT hybrid nanobiocomposite. The enhanced mechanical properties in combination with their fully biodegradable PLA/TKF/TAF/1MMT hybrid nanobiocomposite have high potential in automobile, electronic components, household applications and food packaging applications. Hence, the author's proposed that the low quality natural fiber composite overall properties could be enhanced by hybridizing with high quality natural fiber and MMT clay.

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