TMMOB Tekstil Mühendisleri Odası
UCTEA Chamber of Textile Engineers
Tekstil ve Mühendis
Journal of Textiles and Engineer

Yıl (Year) : 2019/3
Cilt (Vol) : 26
Sayı (No) : 115

Araştırma Makalesi / Research Article

A NOVEL APPROACH FOR FABRICATION OF THERMOPLASTIC STARCH BASED BIOCOMPOSITES

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Gönderilme Tarihi / Received: 17.04.2019
Kabul Tarihi / Accepted: 10.09.2019

ABSTRACT: In this study, jute fiber/thermoplastic starch based (TPS) biocomposites were fabricated by using a novel mixing method. Dry mixing of filler and matrix was carried out by using a planetary high shear mixer. Various levels of fillers were used in order to observe the effect of fiber ratio on mechanical, structural and thermal properties of the composites. Both tensile strength and elastic modulus values of biocomposites were found to be improved by incorporation of jute fibers. The enhancement was attributed to the reinforcing effect of jute fibers and strong interphase between the filler and polymeric matrix that was also shown by morphology and FTIR analysis. In addition to those, thermal stability of the TPS composites increased by addition of the jute filler.

Keywords: Jute fibers, thermoplastic starch, biocomposites, morphological characterization, mechanical characterization

TERMOPLASTİK NİŞASTA ESASLI BİYOKOMPOZİTLERİN ÜRETİMİ İÇİN YENİ BİR YAKLAŞIM

ÖZET: Bu çalışmada, özgün bir karıştırma yöntemi kullanarak jüt lifleri ve termoplastik nişasta (TPN) esaslı biyokompozitler üretilmiştir. Kuru karıştırma işlemi, yüksek kayma hızında yörüngesel olarak çalışan bir karıştırıcıda gerçekleştirilmiştir. Lif oranının kompozitin mekanik, yapısal ve ısıl özelliklerine olan etkisini gözlemlemek amacıyla farklı dolgu oranları kullanılmıştır. Biyokompozitlerin hem kopma dayanımlarının hem de elastik modül değerlerinin jüt lifi ilavesi ile arttığı bulunmuştur. Bu artışın, jüt liflerinin takviyelendirme etkisinden ve morfoloji, FTIR analizlerinde de gösterilen lif polimer arasındaki güçlü arayüzeyden kaynaklandığı düşünülmektedir. Tüm bunların yanında jüt liflerinin ilavesi ile TPN kompozitlerin ısıl stabilitesi artmıştır.

Anahtar Kelimeler: Jüt lifleri, termoplastik nişasta, biyokompozitler, morfolojik karakterizasyon, mekanik karakterizasyon

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DOI: *10.7216/1300759920192611501, www.tekstilvemuhendis.org.tr*

1. INTRODUCTION

Thermoplastic starch (TPS) that is also known as plasticized starch is of great importance for many applications not only its easy processing and relatively low cost but also its biodegradability and sustainability. As known, starch is one of the most abundant natural polymers found on earth so, thermoplastic starch-based materials can be fabricated relatively cheap and easy when compared with traditional conventional thermoplastics. The main process, plasticization, can be carried out by different systems depending on the amount of the material [1-4]. However, the most common method used in the industry is the plasticization by an extruder. For this process generally, starch, plasticizer, and other additives are mixed prior to plasticization and fed into the extruder. By the synergistic effects of plasticizer, heat and shear deformation; gelation, fusion, and plasticization take place, respectively. Although starch cannot be melt processed, thermoplastic starch can be processed as traditional thermoplastics and TPS based products can be fabricated in various forms by using different systems including injection molding, extrusion blowing, high-temperature compression molding and so on [2]. Since TPS is relatively cheaper than other commercial biodegradable polymers, it can be used for many applications including packaging films and polymeric foams. Although TPS has become prominent because of its biodegradability and sustainability, its inherent properties are generally inadequate for some applications. In that case, TPS based composites draw attention because of their superior properties. In order to obtain desired morphological and mechanical properties, thermoplastic starch can be mixed with various fillers [1, 5-7]. If the biodegradability of the composite is considered; cellulose-based natural fibers become one of the first choices because of the chemical similarity to starch. As known, chemical similarity between filler and matrix is of significance in terms of obtaining a strong interface between cellulosic fiber and thermoplastic starch [5]. Although there are many studies in the literature related to natural fiber filled TPS composites [1, 5-8], number of the studies are very limited for jute fiber filled TPS composites. Jute fibers are cellulose-based bast fibers obtained from *Corchorus olitorius* plant and totally biodegradable, sustainable, strong materials. Jute fibers have antistatic character and they have resistance to microorganisms that enhance the range of the applications. They are used for many applications from ropes to carpet backings, floor coverings, and composite structures mainly because of superior mechanical properties [5]. When the jute fiber/TPS based composite studies are reviewed, jute fibers were found to be used in various forms including fiber [5, 7, 9] and fabric [10]. Iman and Maji, designed and fabricated nanocomposites by using nanoclay, thermoplastic starch, glutaraldehyde and jute fibers. Since jute fiber ratio was kept constant the effect of jute reinforcement was not very obvious in the study. Glutaraldehyde and nanoclay were found to increase mechanical strength and elastic modulus [10]. In another study, TPS was mixed with jute in an internal mixer and shaped by compression molding system. Results showed improved mechanical properties and reduced water absorption rate [5]. In a study carried out by Ray *et al.* jute fabric/TPS composites were prepared. Firstly solution cast TPS based films were prepared by using starch and poly(vinyl alcohol), water and glycerol. In the second step, jute fabrics were impregnated by gelatinized starch. In the final step, films and impregnated fabrics were laminated in a compression molding system. Mechanical properties were reported to be higher in the case of higher level of starch content. The interface between jute fibers and matrix was found to be good [3]. In another study, tube-shaped jute/TPS composites were fabricated by a pultrusion method. As reported in the study, the melt viscosity of the polymer and fiber ratio was found to be significant in terms of mechanical properties. In addition to that, the impregnation step in the pultrusion process was reported to be very critical in terms of morphology and interface properties of the composites. As concluded in that study interface and morphology determine the mechanical properties of the composite and the process used for composite fabrication [7]. As reviewed in the literature, mixing/compounding of TPS and jute fibers were generally carried out in an extruder. However, there are some limitations of extruder processing that are stemmed by cellulosic fibers and TPS. Cellulose based fibers (including jute) generally start to thermally degrade above 180°C and that is the main handicap for processing of cellulose based compounds in an extruder. Any polymer that is used as a matrix needs to have a melting point around 180°C. In addition to applied temperature, processing time is of significance in terms of morphological and chemical stability of the cellulosic fiber and filler-matrix interface. As reported in the literature, long residence time in the extruder leads to thermal degradation. In that case, various physical and chemical alterations can be observed including oxidation, dehydration and depolymerization. These not only affect the morphology and surface properties of the fibers but also filler-matrix interface and mechanical properties of the cellulosic fibers and composite. To summarize, higher process temperature values (more than 180°C) and/or longer residence/exposure times are very important and need to be taken into the consideration [11]. Also high viscosity of cellulosic fiber filled TPS is an important handicap for the mixing and homogeneous filler dispersion. As known, cellulosic fibers are materials with high rigidity because of their relatively high degree of crystallinity. That rigidity is the main reason for high viscosity during the compounding process in the extruder. Any increase in the temperature and/or shear lead to decrease in viscosity of the TPS. However, cellulosic fibers do not melt like traditional thermoplastics and they do not have important contribution for decrease in viscosity. Because of the facts mentioned above, homogeneous filler dispersion might be problematic for jute/TPS composites. Instead of classical extruder compounding, a novel approach for mixing jute fibers and TPS was developed in this study. A planetary high shear mixer was used in our process. As known filler dispersion in a matrix is one of the most important challenges in composite production.

Planetary high shear mixers are versatile devices that provide fast and homogeneous mixtures for both nano and micron-scale fillers. By the effect of planetary movement not only dispersion but also deaeration take place at relatively short times such as 60-90 sec. Since no blades or mixing rods are used, the aspect ratio of the fillers do not change. By this method fibers are well-dispersed in the matrix without any thermal degradation at relatively short time (90 sec). One novelty of the method lies in

the fact that both fillers and matrix do not thermally degrade and their chemical and physical structures are protected. The other novelty is that homogeneous mixing is carried out in a very quickly and total processing time is relatively short. As far as we know dry mixing method (by a planetary high shear mixer) was used for the first time in the literature for jute fiber/TPS composite fabrication.

In this study, biodegradable composites are fabricated by using jute fibers and thermoplastic starch. For the composite film production, dry mixing by a planetary high shear mixer and compression molding processes were combined. Jute fiber/thermoplastic starch composites were prepared at different filler ratios. According to outcomes, high shear mixing is found very effective regardless of the filler concentration and composites properties are directly affected by the filler ratio.

2. MATERIALS AND METHODS

2.1 Materials

Jute fibers were bought from a local store in the form of yarn and cleaned by acetone until a clear solution was obtained (3 cycles). After that, samples were dried in a vacuum oven for 24 hrs and carefully cut into short fibers by using a special cutting blade.

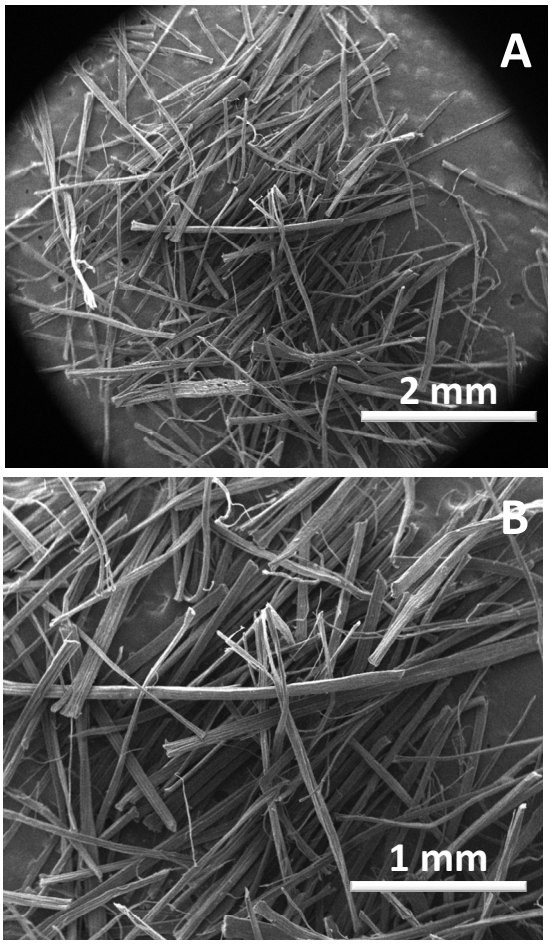

Figure 1. SEM images of jute fibers (a) 50x (b) 100x

In order to determine the distribution of fiber length and diameter scanning electron microscopy (SEM) analysis was carried out. The reason for choosing SEM is not only to determine the aspect ratio but also surface morphology of the jute fibers that are critical for the mechanical properties of the composites. According to SEM analysis, average length, average diameter and average aspect ratio were calculated as 62 µm 1612 µm and 26, respectively by using Image J software. SEM images of jute fibers at various magnifications, length and diameter distribution can be seen in Fig.1, Fig.2 and Fig.3 respectively.

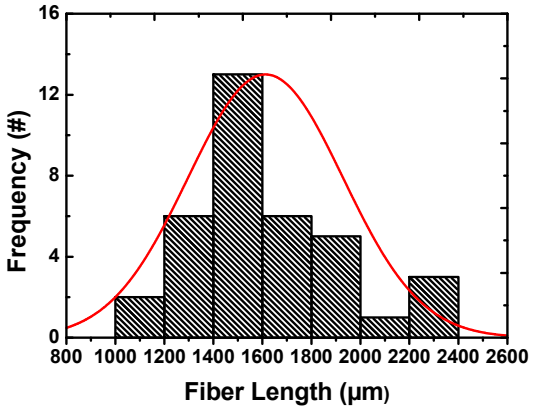

Figure 2. Length distribution of jute fibers

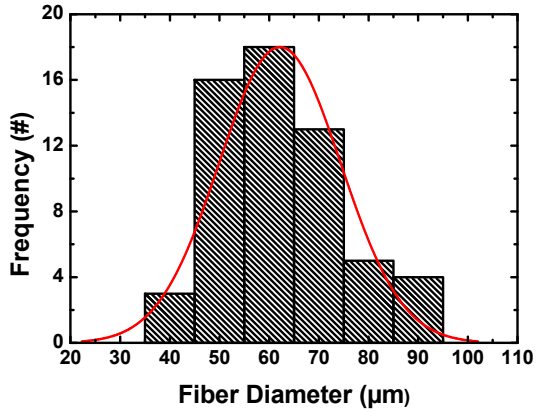

Figure 3. Diameter distribution of jute fibers

Thermoplastic starch was kindly supplied from Sunar Mısır. Glycerol was used as a plasticizer and the glycerol/starch ratio was given as 30/70.

2.2 Composite Fabrication

For the composite film production, dry mixing and compression molding processes were combined. Mixing was carried out by a planetary high shear mixer (Mazerustar KK-50S). Filler concentration was determined as 0, 2.5, 5, 7.5, 10 wt %. As can be seen from Fig. 4, determined amount of jute fiber is added into the TPS and mixed for 90 sec.at room temperature at 1600 rpm. Temperature was checked after mixing and 1-2 ºC increase was determined. (This was probably due to the high shear and friction between filler, matrix and container.)

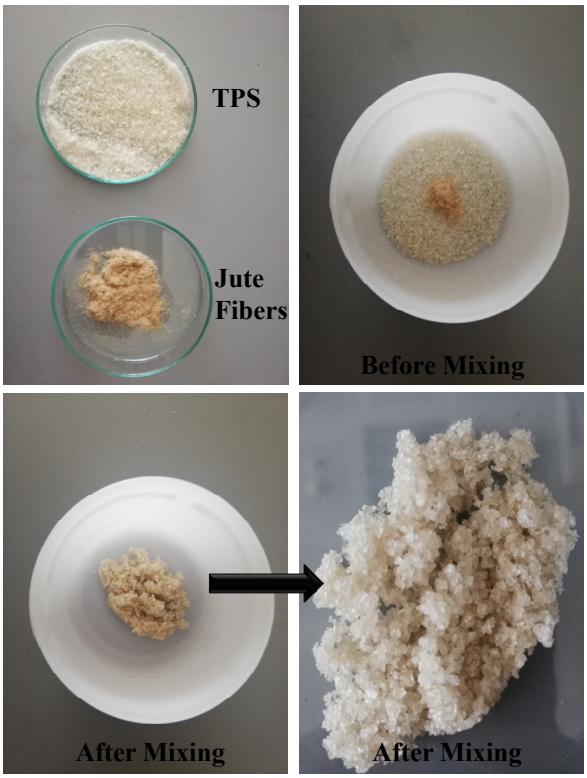

Figure 4. Dry mixing process of jute fiber/TPS

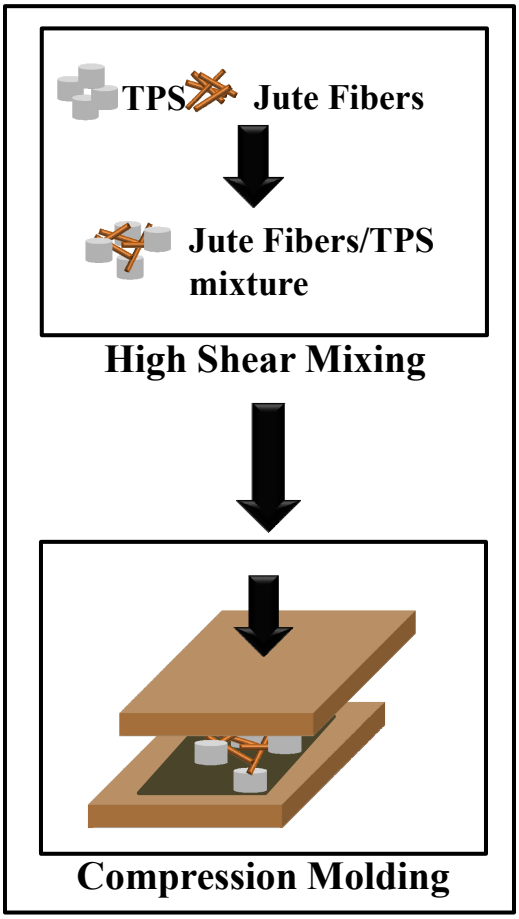

Figure 5. Jute fiber/TPS composite fabrication

After mixing, samples were compression molded between Teflon coated metal plates as shown in Fig. 5. In the first step, optimization of the process was carried out for thermoplastic starch films at various temperatures (170-185ºC) and time intervals optimum temperature was determined as 180ºC depending on the film formation and appearance. Following that all samples were compression molded at 180ºC for 3 minutes. During that process pressure was around 1 MPa.

2.3 Characterization

Morphology of TPS film and composites were analyzed by scanning electron microscope (SEM) (20 kV, Yalova University Central Research Laboratory). Prior to analysis samples were cut by a blade and cross-sections of the samples were sputter coated by Au/Pd alloy. Coating was around 3-8 nm.

Stress-strain behavior of the composite films was characterized by a universal load frame (Devotrans, DVT GPU/RD) with the strain rate of 10 mm/min. . Tests were carried out just after the composite fabrication. Film thickness was measured by a Mitutoyo digital micrometer from ten random locations. Average thickness was between 0.5-0.6 mm for all samples. For each composite type, 3 specimens with the dimensions of 25 mm x 5mm (length x width) were prepared and tested for load-extension behavior, average values were calculated. The mean values were calculated and the load-extension values were subsequently converted to engineering-stress and true-strain.

FTIR analysis was carried out by Perkin Elmer, Spectrum 100 IR spectrometer and attenuated total internal reflectance (ATR) spectroscopy was used. Spectra were recorded between 650 and 4000 cm^{-1} in the transmittance mode with spectral resolution of 4 cm^{-1}. at a scan rate of 4 scans.
Thermogravimetric analysis (TGA) was carried out by Seiko, TG/DTA 6300 between 25 and 650°C under nitrogen atmosphere with a gas flow of 20 ml/min. Approximately 3-5 mg samples were used.

3. RESULTS AND DISCUSSION

In order to investigate the dispersion of jute fibers and fiber-matrix interface, SEM analysis was carried out. Cross-sectional images are of TPS film and 7.5 wt% jute fiber-containing composite are given in Fig. 6a-c. As seen from the images, TPS was successfully melted regardless of the composition under determined conditions. That can be understood from the continuous matrix throughout the samples. Fig. 6b is a low magnification image of the composite film. This image is useful for understanding the filler dispersion and orientation. White arrows show the jute fibers. As obvious from the image, fibers are found to be well-dispersed in the matrix without any dominant fiber orientation.

That is probably due to effective high-shear mixing. In addition to that, no pores are observed in the composite structure. If the fiber-matrix interface is analyzed, a good interface can be seen clearly with good matrix wetting and continuity (Fig 6c). This is probably caused by three reasons. Firstly, jute fibers and TPS

matrix are members of the carbohydrate family and have similar chemical structures. That enhances the chance of formation of the strong interface by high levels of molecular interactions between two phases. Secondly, surface roughness of the jute fibers increases the adhesion [5]. Lastly, composites were fabricated at 180º C under the effect of compression. While temperature is responsible for the melting of the TPS, pressure enhances the wetting ratio of fibers by the molten polymer matrix. By the effect of pressure air, that was captured between the single fibers, can be removed from the composite structure and improves the interface morphology.

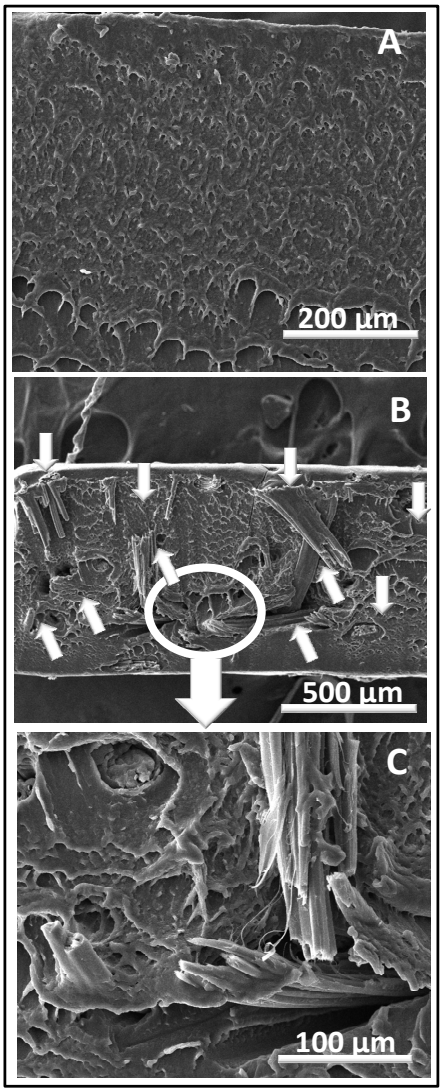

Figure 6. Cross-section SEM images of (a) 0 wt% and (b) 7.5 wt% jute fiber/TPS composite at 250x (c) 7.5 wt% jute fiber/TPS composite at 1000x

Mechanical properties of composites are of very important for providing insights into the morphology and determination of their mechanical performance limits. Typical stress-strain behavior of the TPS film, and jute/TPS composites for various filler content is presented in Fig. 7 and Table 1. As can be seen, tensile strength, elastic modulus and strain at breakage increased by the incorporation of the jute fibers. Reinforcement effect of jute fibers is reflected as the increased load-bearing capacity of the composites. As known jute fiber have a relatively high elastic modulus (10-30 MPa) [12] when compared with TPS. By increasing the ratio of the jute fibers in the TPS composite, tensile stress increased gradually. Tensile strength values of composites are given in Table 1. As obvious from the data, when compared with TPS film, tensile strength is nearly three times and five times higher at 5 and 10 wt % filler concentrations, respectively.

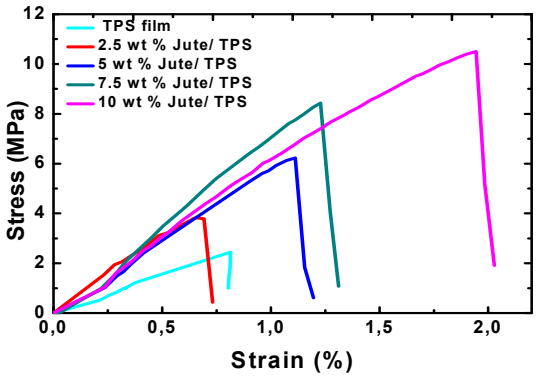

Figure 7. Stress-strain behavior of jute fiber/TPS composites as a function of filler ratio (wt %)

Increased ratio of fibers led to increase in the stress transfer from matrix to the fibers. In addition to high modulus, surface properties and chemical structure of the fibers are of significance in terms of mechanical properties. As previously shown from the morphological analysis, chemical similarity of filler-matrix and surface roughness of the jute fibers increase the ratio of mechanical interlocking because of higher adhesion and this makes the interface stronger. The surface roughness of jute fibers led to strong interaction and good interface. In addition to that tensile strain values increased gradually by the addition of jute fibers. This is probably caused by the good interface and good elongational properties of the jute fibers. The fibers that were aligned in the stress direction increase the load-bearing capacity of the matrix that leads to increase in both tensile strain and strength.

FTIR analyses was performed in order to study the effect of the addition of jute fiber into TPS, to determine possible interactions between jute fiber and starch. Since all composites showed very similar spectrum only the highest filler concentration is given.

Table 1 Mechanical properties of TPS film and composites and standard deviation (SD) values

Sample (Jute wt %)	**Elastic Modulus (MPa)/SD**	**Tensile Strength (MPa)/SD**	**Tensile Strain (%)/SD**
0	3.02/0,09	2.43/0,13	0.81/0,05
2.5	5.80/0,14	3.83/0,21	0.65/0,04
5	6.00/0,36	6.22/0,62	1.11/0,05
7.5	7.43/0,35	8.43/0,82	1.22/0,06
10	6.36/0,42	10.50/0,69	1.94/0,2

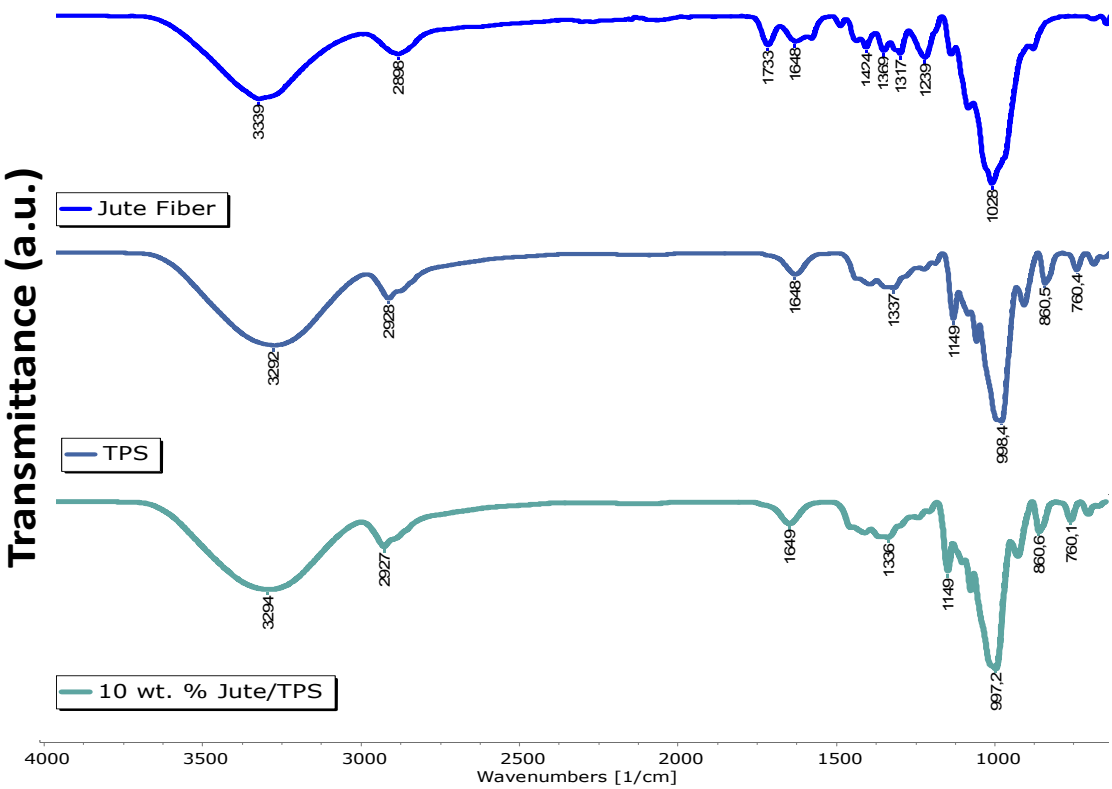

Figure 8. FTIR spectra of (a)jute fiber, (b)TPS film (c)10 wt% jute/TPS composite

Fig. 8 shows the FTIR spectra corresponding to jute fiber, TPS, and jute fiber/TPS composites. Jute fiber spectrum showed the characteristic peaks that were already reported [13]. The broad absorption band between 3000 and 3600 cm^{-1} is, due to OH stretching. As reported by many authors, this is caused by both absorbed water and alcohols found in cellulose, lignin, hemicellulose, and carboxylic acids [14]. The band around 2898 cm^{-1} is associated with CH_3 and CH_2 groups that is typical for cellulose based natural fibers [15]. The fingerprint region around 1800–600 cm^{-1}, is also parallel with the previous studies. The band at 1733 cm^{-1} is due to C=O stretching, the band at 1648 cm^{-1} is caused by H-O-H bending of absorbed water, the bands at 1424 cm^{-1}, 1369 cm^{-1}, 1317 cm^{-1} and 1239 cm^{-1} are caused by asymmetric C-H deformation [16], symmetric C-H deformation [16, 17] CH_2 wagging, C-O stretching of acetyl (lignin) respectively. The presence of the bands at 1157 cm^{-1}, 1103 cm^{-1}, 1027 cm^{-1}, are also associated with cellulosic structure and due to C-O-C antisymmetric bridge stretching, O-H stretching, C-O stretching respectively [18-22].

As seen from Fig. 8, jute fiber and TPS showed almost same peaks in the spectrum based on the functional groups of starch and glycerol. In addition to peaks explained for jute fiber, TPS showed couple of more peaks at 1148 cm^{-1}, 920 cm^{-1} that are associated with C-O stretching. Also the peaks at 860 cm^{-1} and 760 cm^{-1} are due to C-H deformation [16, 23] and -OH out-of-plane vibration or rotational vibration of water [24, 25] respectively.

The spectra of all composite samples are mainly dominated by the TPS bands. When the spectra of TPS and jute fiber/TPS composite are compared, no new peaks are observed. This is caused by the similarity of TPS and jute fiber. As mentioned previously they are both members of the carbohydrate family and have similar chemical structures. It is worth to note that only small shift was observed for OH stretching for jute fiber/TPS composites. While TPS film showed a peak at 3292 cm^{-1}, jute fiber and 10% jute fiber filled composites showed peaks at 3339 cm^{-1} and 3294 cm^{-1} respectively. Parallel with the previous studies [26, 27], TPS peak shifted to jute fiber and this is assumed to be caused by higher level of intermolecular hydrogen bonding that is stemmed from high compatibility of starch and cellulose. As mentioned in the morphological analysis, jute fibers and TPS formed a good interface.

Fig. 9 shows the TGA curves corresponding to jute fiber, TPS, and jute fiber/TPS composites. Jute fiber showed the highest thermal stability in all samples. As obvious from the curve, thermal decomposition of jute is observed in three steps. The first step until 100°C is due to the absorbed water. Weight loss caused by water removal is around 8 %. Second step is associated with decomposition of hemicellulose that starts around 290-300°C. At 300°C, total weight loss is around 20 %. In addition to that α-cellulose decomposition occurs around 365°C. The weight loss at that point is around 60% [28, 29].

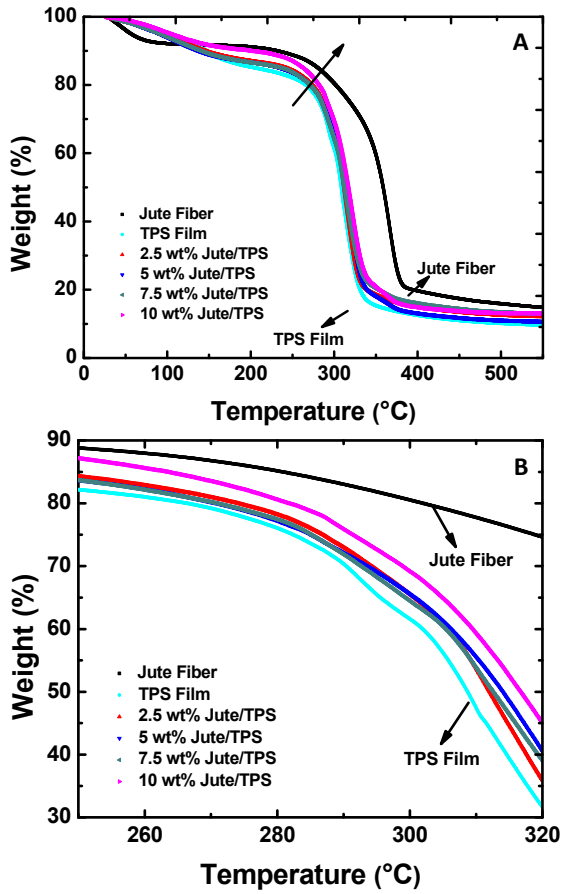

Figure 9. a) TGA curves between 0-550°C, of jute fiber TPS film, 2.5 wt% jute/TPS composite, 5 wt% jute/TPS composite, 7.5 wt% jute/TPS composite, 10 wt% jute/TPS composite b) between 250-320°C

For TPS film and composites around 175 and 300°C showed significant decrease in weight % caused by plasticizer (glycerol) and starch decomposition. The weight loss for TPS film is around 15 % and 40 %, respectively. On the other hand regardless of the concentration, addition of jute fiber leads to improvement in thermal stability. 2.5 wt% jute/TPS composite, 5 wt% jute/TPS composite, 7.5 wt% jute/TPS composite, 10 wt% jute/TPS composite show the following weight loss values respectively; 36 %, 37 %, 37 % and 30 %. In addition to high thermal stability of jute fibers, good fiber-matrix interaction (SEM images (interphase) and FTIR spectra (H-bonding)) leads to this improvement [30, 31].

4. CONCLUSIONS

Jute fiber reinforced thermoplastic starch composites were prepared by incorporation of different levels of fibers by a novel mixing method. In this study, dry mixing of fillers and matrix was carried out by a planetary high shear mixer for the first time in the literature. Good filler dispersion before compression molding process eliminates the compounding process in the extruder. As known, cellulosic fibers can be thermally degraded when the residence time is higher in the extruder. By using high shear dry mixing, total process time was decreased. Morphology, chemical structure, mechanical and thermal properties of the composites were investigated. Fillers were found to be well-dispersed in the matrix. Good filler/polymer interphase was observed without any dominant filler orientation. Since jute fibers provide substantial bridging in the matrix and enhance strength by increasing load bearing capacity of the composite by preventing or minimizing crack formation, mechanical properties were found to be affected by filler content. As given in the FTIR analysis, jute fibers and TPS matrix was found to form hydrogen bond linkages that are significant in terms of obtaining a good filler-matrix interface. TGA analysis showed that jute fiber addition leads to improvement in thermal stability.

This work is of importance in terms of providing alternative and quick route for fabrication of natural biocomposites. Future work will focus on industrial scale-up process of TPS based biocomposites based on our findings in this work.

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