Properties of nylon-6 based composite reinforced with coconut shell particles and empty fruit bunch fibres

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Novel natural fibre composites of nylon-6 reinforced with coconut shell (CS) particles and empty fruit bunch (EFB) fibres have been investigated. The fillers were washed and alkali treated before melt compounding with nylon-6. Composite test specimens were prepared by injection moulding prior to measurement of mechanical, thermal and rheological properties. Tensile modulus was found to improve with both fillers up to 16% for nylon-6/CS composite and 10% for nylon-6/EFB composite, whereas a moderate increase in tensile strength was observed only with CS composites. Differences in the strengthening mechanisms were explained by the morphology of the two fillers, empty fruit bunch fibres having a weaker cellular internal structure. Observation of composite morphology using SEM showed that both fillers were highly compatible with nylon-6 due to its hydrophilic nature. Both fillers were found to cause a slight drop in crystallinity of the nylon matrix and to lower melt viscosity at typical injection moulding strain rates. Moisture absorption increased with addition of both fillers, more so in the case of EFB composite.

Keywords: Nylon-6; polyamide; mechanical properties; thermal properties, rheological properties; coconut shell particle; empty fruit bunch fibre; composite

Introduction

Research into natural fibre composites is attracting increasing levels of interest, particularly the use of natural fibre waste by-products of the agricultural industry. The main advantages of these renewable fillers are their low cost and low specific density, provided that mechanical strength of the matrix can be maintained or enhanced. Applications of natural fibre composites include interior and exterior automotive components (Cunha, 2006, Agus et al., 2013). Hemp and flax fibre composites have also been utilised in sporting goods, mobility equipment, furniture, interior design, lighting applications (Pil et al., 2016 and Sanjay, 2016) and for energy absorption structures (Meredith, 2012). However, the compatibility between matrix and fibres is a

major problem in natural fibre polymer composites and chemical treatment of the fibres is often used to improve adhesion between the fibres and matrix. A commonly used alkali treatment involves immersing fibres in a concentrated solution of sodium hydroxide (NaOH). This reacts with the hydroxyl group of the natural fibres, removing some of the external surface of the fibres and leading to an increase in the surface roughness which improves interfacial bonding (Chandrasekar, 2017). Selection of a polymer matrix which has the same polar characteristic with natural fibre such as nylon can also improve interfacial bonding (Santos, 2007, Ajjima, 2016, Ozen, 2013). Attempts to enhance adhesion further by addition of compatibilizer to natural fibre reinforced nylon composite resulted in limited success (Ozen, 2013, Li, 2012, Mohsin, 2015).

Reinforcement of nylon matrix with treated and untreated pineapple leaf fibre (PALF) showed that alkali treatment promoted greater aspect ratio and better crystalline orientation of the nylon matrix. Further addition of silane did not improve the adhesion, other than increasing the aspect ratio of PALF fibre (Ajjima et al., 2016). Wood/nylon composites were found to produce an increase in tensile strength and modulus without requiring compatibiliser (Henry et al., 2003). The polar nylon matrix was found to provide good adhesion between the nylon and cellulose in the wood via hydrogen bonding. This finding was supported by micrographs showing reduced fibre pull-out in the composite. However, at higher fibre fractions, hydrolysis of the nylon negatively affected the mechanical properties of the composite.

Empty fruit bunch (EFB) fibre from oil palm and coconut shell is abundantly available as a result of agricultural industries especially in Indonesia and Malaysia. Most of this waste is currently burned to fuel power plants. Research has been carried out to

investigate EFB as a reinforcing fibre in polymer matrices such as polypropylene, polyester, polyvinylchloride, polyurethane, phenol formaldehyde (Hasan, 2010) and HDPE (Chinomso, 2012). However, these polymers are hydrophobic (non polar polymer) and thus have a low affinity to bonding with the natural fibres. Nylon is a polar polymer which has been found adhere well to natural fibres. Current, no attempts have been made to use EFB fibres or coconut shell as reinforcing fillers in nylon based composites.

Some research into natural fibre reinforced nylon matrices has been reported. A polyethylene-nylon blend composite was produced with banana stalk fibre (BaF) and tensile modulus, flexural strength and flexural modulus was found to improve with increasing fibre content, whereas impact strength decreased (Liu et al., 2009). Impact strength and interfacial bonding were enhanced by the addition of a coupling agent.

Studies on natural fibre reinforced nylon composites have investigated mechanical and thermal properties (Chen, 2008 and Erbas 2016 – which fibres; what were the findings?). The rheological behaviour of nylon based natural fibre composites has not been reported previously. Rheological properties of flax/PP composite were found to behave as yield stress fluids at low frequencies due to fibre-fibre interactions and show shear thinning at higher frequencies (Moigne et al, 2013).

The aim of this study was to investigate the potential of abundantly available coconut shell and empty fruit bunch fibre as renewable reinforcing fillers for nylon-6. Mechanical, thermal and rheological properties have been analysed and compared to those of the unfilled polymer. Scanning electron microscopy has been used to examine composite morphology and to relate this to the observed material properties.

Materials and methods

Empty fruit bunch fibre and Coconut shell particles

Empty fruit bunch (EFB) and coconut shell (CS) are waste products of the palm oil and coconut industrys respectively. EFB fibre used in this research contained 30.3 % cellulose and 38.3 % lignin; CS contains 49.8 % cellulose and 25.1 % lignin The ultimate tensile strength of EFB fibre ranges from 92.5 to 246.2 MPa with an elastic modulus of 11.9 GPa (Gunawan et al., 2009). An alkali treatment was applied to both fillers prior to compounding. The EFB fibres and CS particles were washed with distilled water and then immersed in 5% NaOH solution for 2 hours before drying at room temperature for 72 hours. According to SEM micrographs, the fibre diameter was in the range of 0.06 to 0.66 mm and the fibre length in the range of 1 to 4 mm.

A commercial injection moulding grade of nylon-6 (Schulamid 6NV12) was used as the polymer matrix. Based on the data from the supplier this polymer had a solid density of 1.13 g/cm^3, viscosity number of 130 cm^3/g, tensile strength at yield of 85 MPa, tensile strain at yield of 3.5 %, tensile modulus of 3 GPa and an impact strength of 3.5 KJ/m^2.

Fabrication of composite specimens

The nylon-6 was dried for 6 hours at 80°C prior to compounding. The fillers were dried in a vacuum oven for 1 hour at 80°C to remove residual surface moisture before compounding into the matrix polymer. For composite nylon-6/CS, the filler loadings were 5, 10, 15 and 20% by weight. For composite nylon-6/EFB, the filler loading was 5, 10 and 15% by weight. The maximum loading of EFB fibre examined was limited to 15% due to difficulties in achieving adequate dispersion at higher loadings during twin screw extrusion. The filler was manually pre-mixed with nylon-6 according to the

associated weight fraction. Composites were melt compounded using a twin screw extruder (Prism 16TC) with a screw diameter of 16 mm and a length to diameter ratio of 15:1. Each compound was manually fed into the extruder at a screw rotation speed of 150 rpm with barrel set temperatures of 200, 220, 230 and 240°C. The extruded strand was water-cooled and cut into uniform pellets 3 mm in length, which were then dried for four hours in advance of further processing.

Composite test specimens were prepared on an electric injection moulding machine (Fanuc Roboshot S-2000i5A) with a screw diameter of 14 mm and a maximum clamp force of 5 tonnes. The set barrel temperature was set to between 215-230 °C according to filler loading, to maintain the moulded part quality (i.e. no shrinkage). All other set moulding parameters were maintained at constant levels throughout the experiments; mould temperature 70°C, packing pressure 400 bar, packing time 24 s and injection speed 200 mm/s. These parameters were selected as optimum settings for injection moulding pure nylon-6.

Characterization of mechanical properties

Tensile tests were performed at room temperature using a Tensometer (Instron 5564) equipped with a 2 KN load cell. The tensile test specimen was moulded according to ISO 527-2:1996 type 1BA. A crosshead speed of 1 mm/min was selected according to the same standard. Izod impact tests were performed on notched beam specimens using an Instron-Dynatup POE2000 according to ASTM D 256-93a.

Thermal characterisation

Crystallinity and melting behaviour of the composites were assessed using differential scanning calorimetry (DSC) using a TA Instruments Q20. Pellet samples of

approximate mass 5 mg were heated from 40 to 300°C in a nitrogen environment. The samples were held isothermally at 300^{0}C for one minute before being cooled down to 40°C, held for three minutes and then reheated to 300°C. All applied heating/cooling rates were set to 10°C/min. Thermogravimetric Analysis (TGA) was performed on all samples using a TA Instruments Q500. Measurements of weight loss were made in the temperature range of 25 to 800°C. Dynamic Mechanical Analysis (DMA) was performed using a TA Instruments Q800 in cantilever bend mode, to assess the effect of moisture on mechanical properties. Tests were carried out on moulded bars in both dried and undried states at a constant frequency of 10 Hz at 1% strain, within a temperature range of 25 to 50°C.

Rheological characterisation

Rheological properties of pure nylon-6 and each of the composites were measured using a strain controlled rheometer (Anton Paar Physica MCR 301) at a set temperature of 240 ±0.1°C. Small amplitude oscillatory shear measurements were performed using parallel-plate geometry with plate diameter of 25 mm and a gap setting of 1 mm. Angular frequency sweeps were performed in the range of 0.1-100 rad/s. To ensure that all of the rheological experiments were performed in the linear viscoelastic region, strain sweeps were initially performed for each sample. As a result, a strain of 5 % was selected for all frequency sweep data.

Results and discussion

Filler morphology

SEM micrographs of CS particles are shown in Figure 1(a) and EFB fibres in Figures 1(b) and 1(c). Coconut shell particles were found to vary widely in size between 50 and

500 µm. This was attributed to the extraction method employed, being manual grating of the coconut shell rather than mechanical grinding due to the small sample sizes. Some particle agglomeration was also evident with the CS particles, agglomeration of particles tends to decrease the strength of the composite since load transfer is less efficient. The internal structure of the EFB fibres appeared to be cellular in nature, with cells increasing in size as fibre diameter increased. Cellular voids in reinforcing fibres have been shown to result in decreased fibre strength (Gunawan et al., 2009).

Mechanical properties

Elastic modulus of the composites increased with addition of the fillers as shown in Figure 2(a). The elastic modulus of EFB fibre composites increased up to 10% filler loading, above which it decreased. High standard deviation of measured modulus values at 15% EFB loading reflected irregularities in the compounding process; effective mixing proved to be difficult above 10% loading with EFB fibres. The nylon-6/CS composites showed a gradual increase in elastic modulus with filler content throughout the 0-20 wt% range examined. The increase in elastic modulus is indicative of effective stress transfer from the matrix to the filler suggesting good adhesion between matrix and filler. The tensile strength of nylon-6/EFB fibre was found to decrease with increasing filler content, as shown in Figure 2(b). As previously noted, both composites showed good compatibility with the nylon-6 matrix and therefore an efficient transfer of stress into the filler could be expected. However, other factors affect strength such as fibre length, distribution and structure.

The reduction of fibre aspect ratio during extrusion and injection moulding may affect the strength of the composite. Non-uniform fibre distribution, agglomeration and the cellular structure of the fibres may explain the observed lower strength and higher

variation observed for nylon-6/EFB composites. The tensile strength of nylon-6/CS composites increased with filler loading until reaching a maximum value at 15 wt%. Previous studies of natural fibre composites showed similar value of tensile srength (Ozen et.al, 2013). In nylon-6/CS composite, the hard CS particles behave as obstacles to yielding of the matrix, resulting in higher composite tensile strength. However, the increase in tensile strength was not significant because the filler particle size was relatively large (50-500 µm). Particle sizes up to a few microns have been shown to provide optimum reinforcement in polymer composites (Shao-yun et al., 2008). Particulate reinforcement also depends on the particle population and distribution. Tensile strength decreased at 20% loading probably due to particle agglomeration causing a reduction in stress transfer.

Strain at failure of pure nylon-6 was found to be 2.48 ±0.26. For nylon-6/CS and nylon-6/EFB composites, the strain at failure decreased gradually as the percentage of the filler content increased as shown in Figure 2(c). Strain at failure was significantly lower than for pure nylon-6, hence results for pure nylon-6 have been omitted for clarity. The presence of filler restrains matrix yielding which reduces the strain at failure. The measured values of tensile strength and strain at failure are comparable to those reported for pineapple leaf-nylon-6 composites (Panyasart, 2014), although a higher elastic modulus was observed in the current study. Impact strength of nylon-6/CS composites was found to be moderately lower than that for the pure nylon-6 in most cases, as shown in Figure 2(d). Nylon-6/CS composites behaved in a more brittle manner with the addition of CS particles resulting in lower impact strength. Addition of EFB fibres had negligible effect on the impact strength of nylon-6, although nylon-6/EFB specimens were less likely to break completely during impact tests than those containing CS filler.

Composite morphology and filler adhesion

Figure 3 shows SEM micrographs of cryo-fractured surfaces of pure nylon and nylon composites. The fracture surface of pure nylon is shown in Figure 3(a) for comparison. Figures 3(b) and (c) show examples of cryo-fractured surfaces containing 10% loading of coconut shell particles. These images show that the filler particles were well adhered to the polymer matrix at the fracture surface, without evidence of pull-out. Individual striations of polymer can be seen adhered to the particle surface, particularly in Figure 3 (b). This indicates good bonding between the NaOH treated filler particles and the nylon matrix, in agreement with previous research with nylon natural fibre composites. This adhesion has been attributed to the polar nature of the hydrophilic nylon matrix. It has also been suggested that the hydrolysis of the moisture in nylon can occur at high temperature to generate carboxylic acid end groups which are compatible with the –OH groups of cellulose in natural fibres (Santos, 2007). Figures 3(d) and (e) show cryo-fractured surfaces of 10% loading of nylon-EFB composites. The cellular nature of the fibres is clearly visible in these images, which is thought to reflect the lower mechanical properties observed. Interfacial adhesion to the polymer matrix appears to be good, although some evidence of fibre pull-out was observed at the edges of larger fibres, as shown in Figure 3(e). Good adhesion and limited fibre pull-out has also been observed in other studies of nylon-natural fibre composites (Ozen, et.al., 2013). The range of fibre diameters and the cellular nature of EFB fibres can explain the high deviation in measured impact energy values reflected.

Thermal properties

The melting temperature of nylon-6 was found to be relatively unaffected by addition of both filler types, with only a slight reduction observed at high filler loadings in Figure 4(a). Crystallinity was calculated from the measured melting enthalpies using Equations (1) and (2).

$$Xc_{(Nylon-6)} = \frac{\Delta H^{*}_{(Nylon-6)}}{\Delta H^{0}_{(Nylon-6)}} \qquad (1)$$

$$Xc_{(composite)} = \frac{\Delta H^{*}_{(composite)}}{\Delta H^{0}_{(Nylon-6)}} \qquad (2)$$

Where $\Delta H^{0}_{Nylon-6}$ is the enthalpy of melting per gram of 100% crystalline of 240 J/g, $\Delta H^{*}_{(Nylon-6)}$ and $\Delta H^{*}_{(composite)}$ are the measured enthalpy of melting for nylon-6 and its composite, respectively (Fomes et al, 2003).

Mass of the filler was subtracted from the total sample as the measured enthalpy of melting was assumed to result from the only polymeric matrix. From Figure 4(b), it can be seen that crystallinity of both composites was similar to that of pure nylon at high filler loading, suggesting that the filler particles were too large to significantly influence nucleation of crystal growth. Other studies have shown similar results (Nopparut, 2016; Panyasart, 2014). A reduction in crystallinity was observed in LDPE with addition of biocomposites (Shaji et al., 2010) whereas the reverse was observed when introduction of flax fibres were found to increase the crystallinity of PP/EPDM blends. For reinforced composites, particle size has been suggested as the primary factor for

determining the effect on crystallinity i.e. enhancement or reduction in the degree of crystallisation (Mihaela et al., 2007).

TGA results in Figure 4(c) and (d) showed that unfilled nylon-6 contained around 0.75 % moisture, which increased up to 2.3 % with addition of 15% EFB but only up to 1.2 % for 20wt% CS. This implies that EFB was more susceptible to moisture uptake than CS, possibly due to the larger filler particles and cellular structure of EFB. Similar results were observed with vegetal fibre composite (Fernandes et.al (2015). Thermal degradation of the composites was observed to start at lower temperatures than for pure nylon, in particular for 15wt% loading of EFB fibres. However, thermal degradation with both fillers started to occur at temperature above which the composite material was extruded and injection moulded. ***possibly add an extra figure (4e) showing peak degradation temperature against filler loading. Or alternatively a table summarising thermal properties such as Tg, Tm, degradation temperature?***

Rheological properties

Rheological characterisation at 240 ^{0}C showed that during an amplitude sweep at constant frequency, the storage modulus, G', for pure nylon-6, nylon-6/CS and nylon-6/EFB remained constant up to a strain of approximately 12 %. This suggested that the magnitude of the linear viscoelastic range of the materials was controlled primarily by the flow behaviour of the matrix and was not strongly affected by the fillers. In accordance with this test, a strain value of 5 % was selected for subsequent frequency sweep tests for pure nylon-6 and all composites. Results from frequency sweep tests for both composites are shown in Figures 5 (a) and (b). The complex viscosity, η*, of unfilled nylon-6 was found to be relatively independent of frequency. In contrast, the complex viscosities of nylon-6/CS and nylon-6/EFB composites were significantly

higher at low frequencies but exhibited shear thinning, becoming lower than that of pure nylon-6 at high rates. Similar behaviour has been observed for other polymer composites (Ayman et al., 2011; Marcovich et al., 2003; Min et al., 2008). At low frequency, the addition of filler disturbs the normal chain formation of nylon-6 resulting in a decrease of the polymer chain mobility hence the resistance to the melt flow is increased (Ayman et al., 201; Min et al., 2008; Smita et al., 2007; Yan-Hong et al., 2011). At higher frequencies the viscosity decreases since the number of polymer chain entanglements reduces with increasing shear rate (Marcovich et al., 2003). The magnitude of the observed shear thinning behaviour increased with increasing fibre content, with nylon-6/CS exhibiting more pronounced shear-thinning than nylon-6/EFB.

Experimental observations made during injection moulding of composite samples provided further evidence of this shear thinning behaviour; during injection (at high strain rates), measured injection pressure was found to decrease with addition of both fillers. Reduction of melt viscosity is beneficial to the injection moulding process, as cost savings may be achieved through lower set temperatures or a reduction of energy required for injection and plastication of the molten polymer. Figures 5 (c) and (d) show that the storage modulus, G', for both nylon-6/CS and nylon-6/EFB was higher than that of pure nylon-6 over the frequency range examined. Reinforcement by filler particles has been shown to decrease polymer chain mobility (Alvarez et al., 2005; Andrzej et al., 2009; Liang et al., 2005; Min et al., 2008). Storage modulus was found to increase slightly with increase in filler loading, for both composites. It was also observed that the storage modulus of pure nylon-6 was reduced following extrusion, particularly evident at low frequencies. This is presumed to result from a process-induced reduction in molecular weight, which also correlated to a drop in complex viscosity.

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Compared to storage modulus, the loss modulus was significantly less affected by the presence of fillers. In Figures 5 (e) and (f), the loss modulus, G'', for nylon-6/CS and nylon-6/EFB are shown to be slightly higher than those for the pure nylon-6 at low frequency. Increasing filler content did not significantly affect the G'' values for either composite.

DMA results are displayed in Figures 6 (a) and (b) and showed that drying of the moulded specimens had a significant effect on mechanical properties for pure nylon-6 and both composites. Storage modulus of nylon-6, nylon-6/CS and nylon-6/EFB composites were found to increase up to 45.4%, 35.1 % and 37.5 % respectively, after drying. Addition of both fillers increased the storage modulus in a relatively linear fashion both before and after drying. These results suggest that incorporation of the renewable fillers did not have a detrimental effect on the moisture sensitivity of mechanical properties.

Overall, the two fillers showed good potential for addition to nylon-6, demonstrating good adhesion to the polymer matrix, enhancing mechanical stiffness and strength (in the case of coconut shell), with only a moderate reduction in impact strength. Potential applications include interior automotive components and structural components in the building industry.

Conclusions

Two nylon-6 natural fibre composites were prepared by twin screw extrusion compounding and injection moulding. SEM micrographs suggested good adhesion between the alkali treated fillers and polymer matrix, particularly with coconut shell particles. Elastic modulus was enhanced with addition of both fillers, increasing by up to 16 wt% at the highest loading of coconut shell particles. Incorporation of fruit bunch

fibres adversely affected tensile strength of the composite whereas coconut shell particles increased tensile strength up to loadings of 15 wt%. Impact strength of both composites was marginally lower than that of unfilled nylon, and lowest in the case of coconut shell particles. The complex viscosity of both composites was lower than that of pure nylon at high strain rates. Thermal properties of the nylon matrix were relatively unaffected by the addition of fillers although a higher uptake of moisture was observed and thermal degradation was found to begin at lower temperature for both composites. The two low cost renewable waste materials showed good potential for use as fillers of nylon-6, without requiring the addition of compatibiliser. Further research is required to determine optimum parameters such as filler size and loading.

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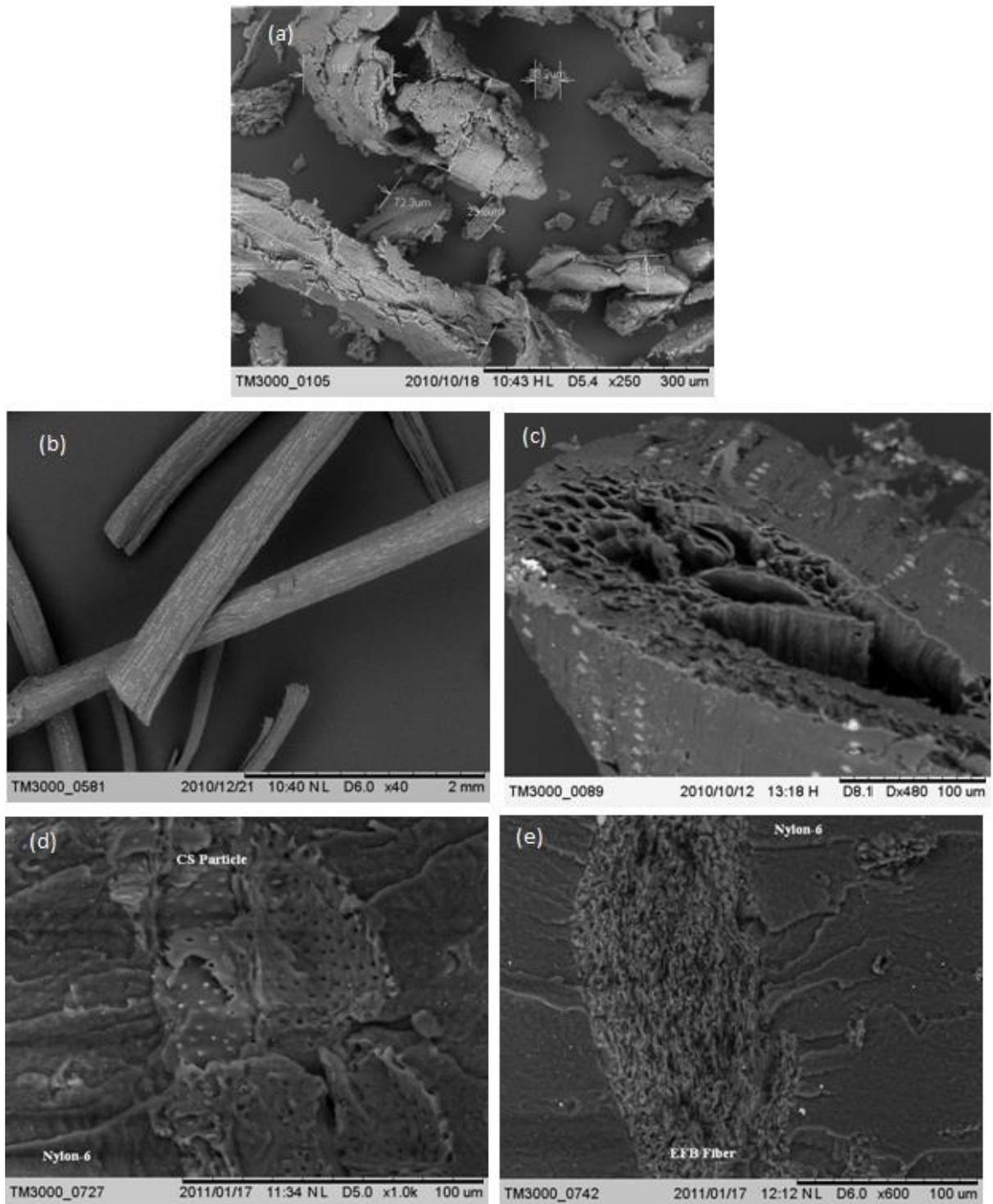
Figure 1: (a) Alkali treated coconut shell particle (b) Length of alkali treated empty fruit bunch fibre (c) Cross section of empty fruit bunch fibre (d) Cryo-fractured show the compatibility of nylon-6/EFB (e) Cryo-fractured show the compatibility of nylon-6/CS.
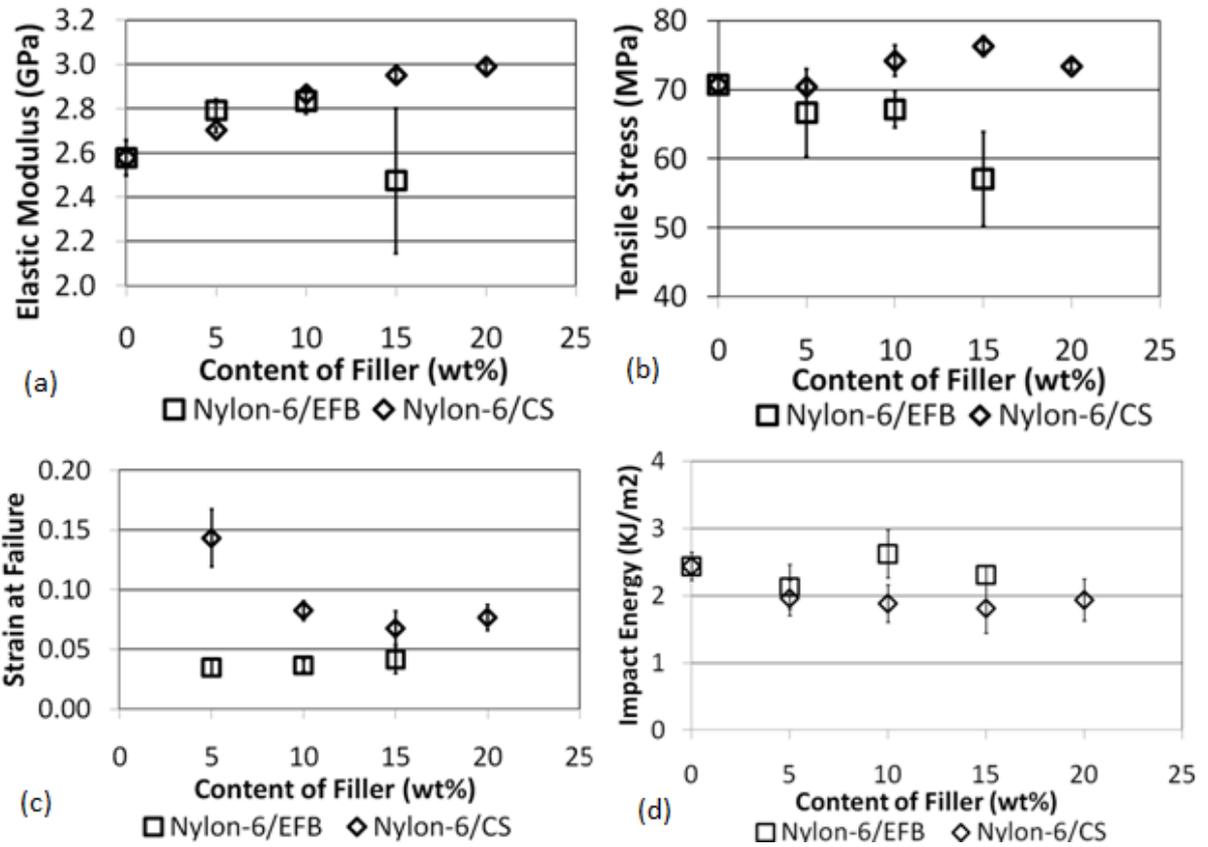
Figure 2 Mechanical and thermal properties of composite: Elastic modulus as a function of fillers content (b) Tensile stress as a function of fillers content (c) Strain at failure as a function of fillers content (d) Impact energy as a function of fillers content.
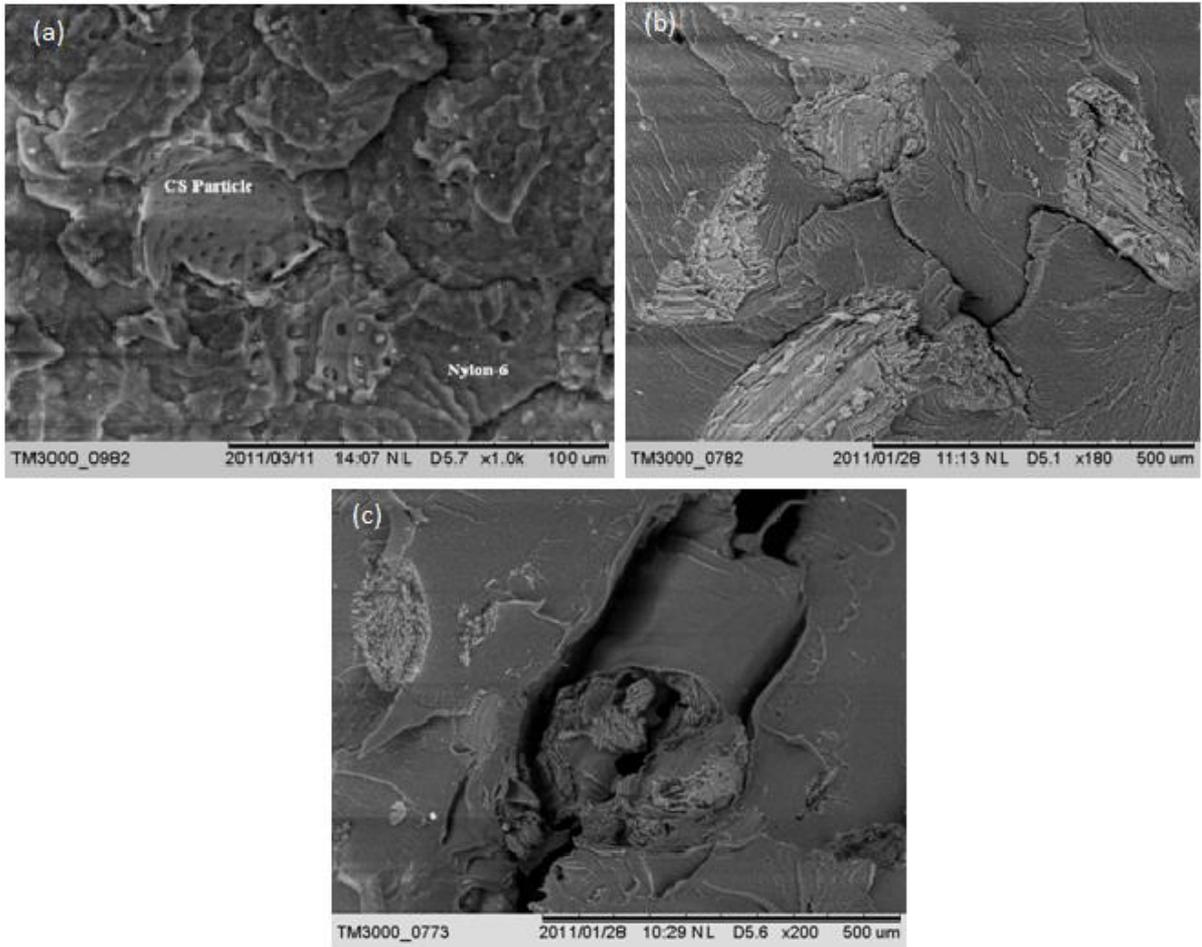
Figure 3 SEM micrograph of: (a) impact fracture of nylon-6/20% CS. (b) Impact fracture of nylon-6/15% EFB composite (c) Impact fracture of nylon-6/15% EFB composite: fiber bridging.

(a)

(b)
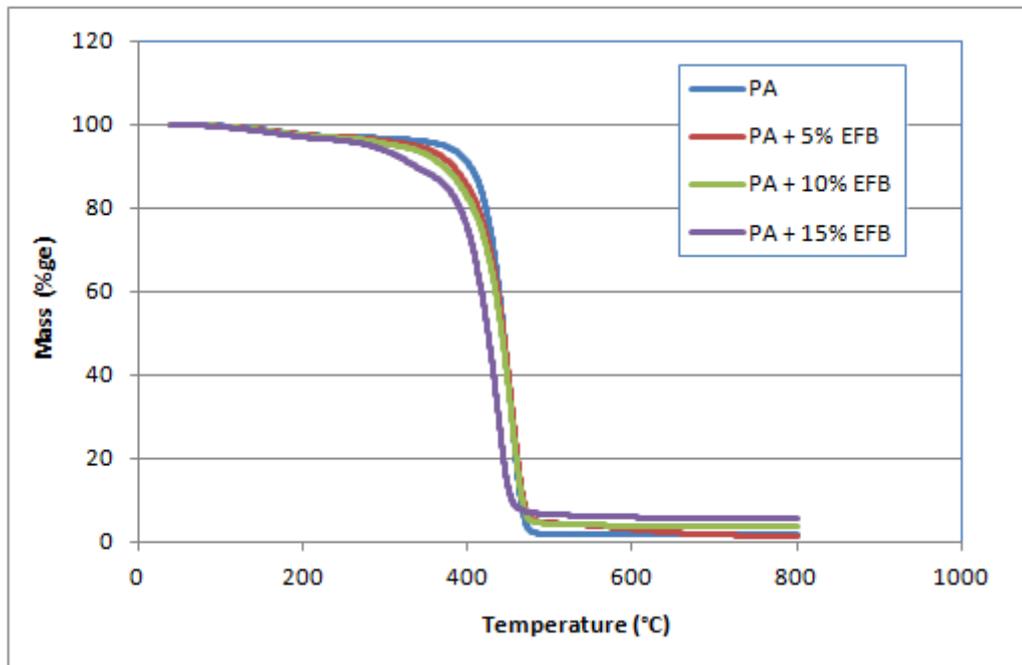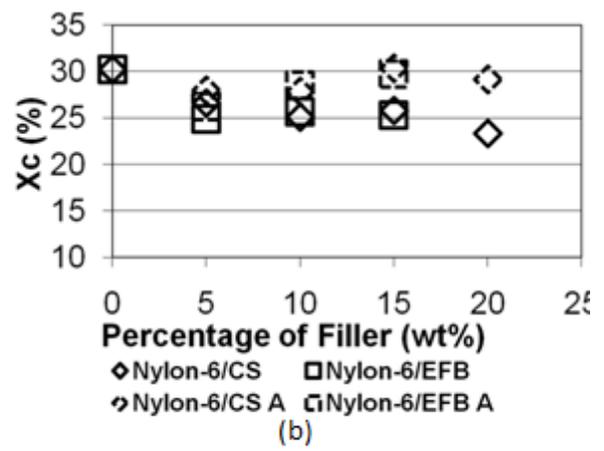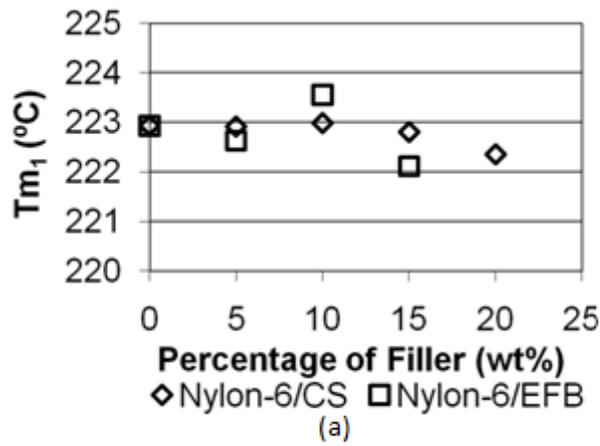
(c)
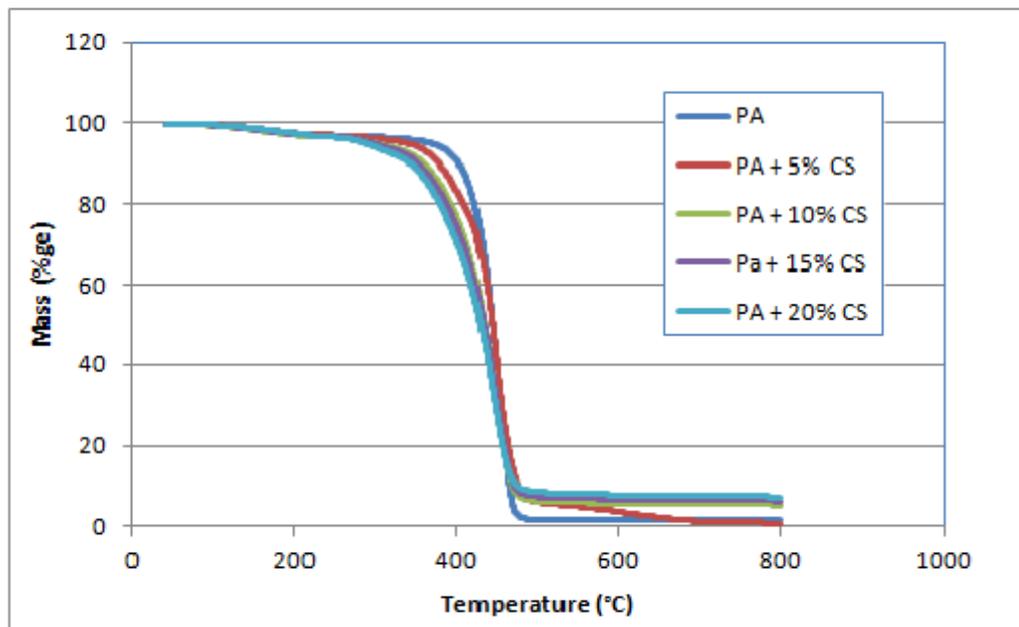
(d)

Figure 4 Thermal properties of nylon-6 based composite: (a) Melting temperature of composite (b) Crystallization of the composite (c) Weight loss of nylon-6 and EFB (d) Weight loss of nylon-6 and EFB
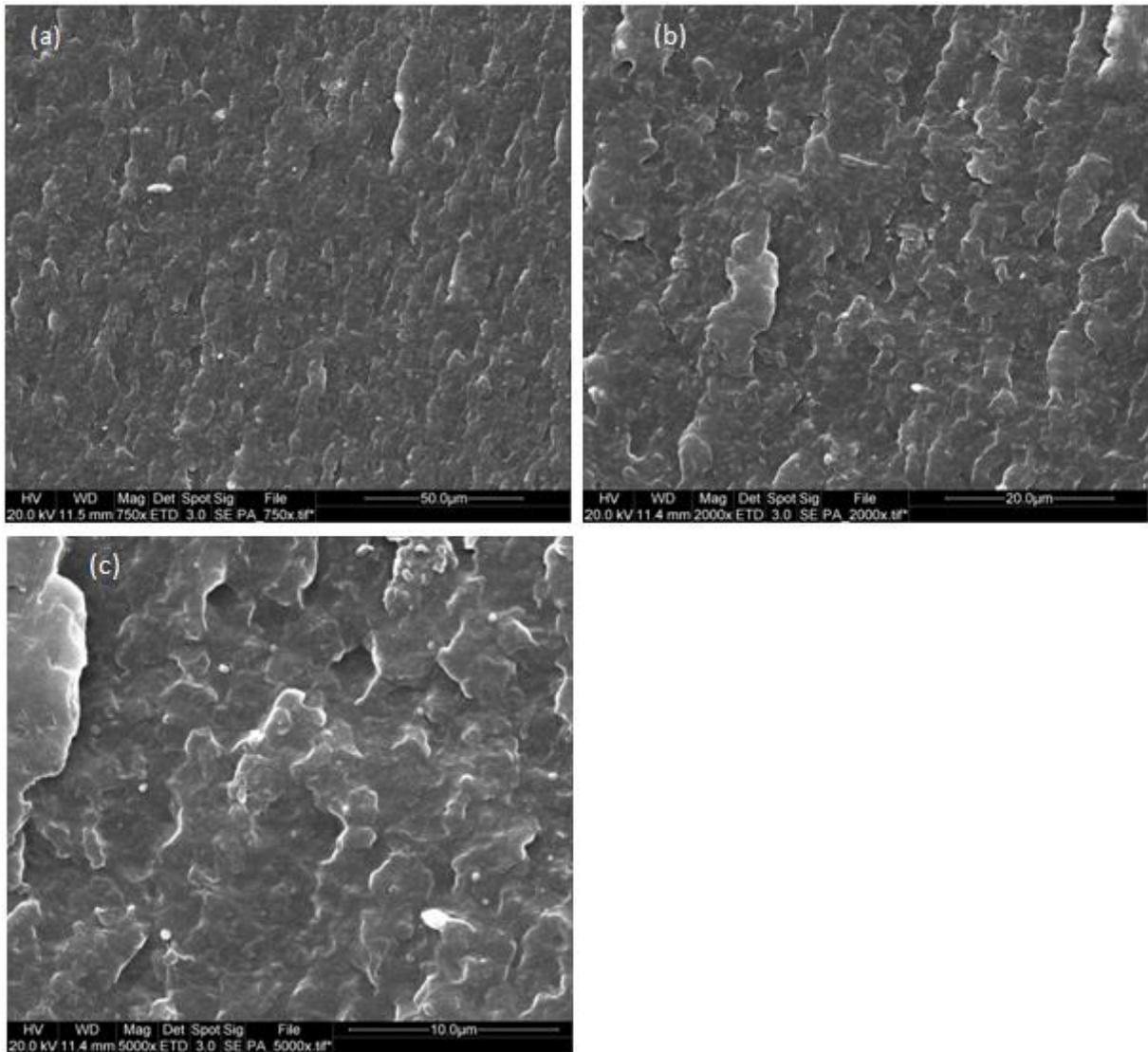
Figure 5 SEM micrograph of pure nylon-6.
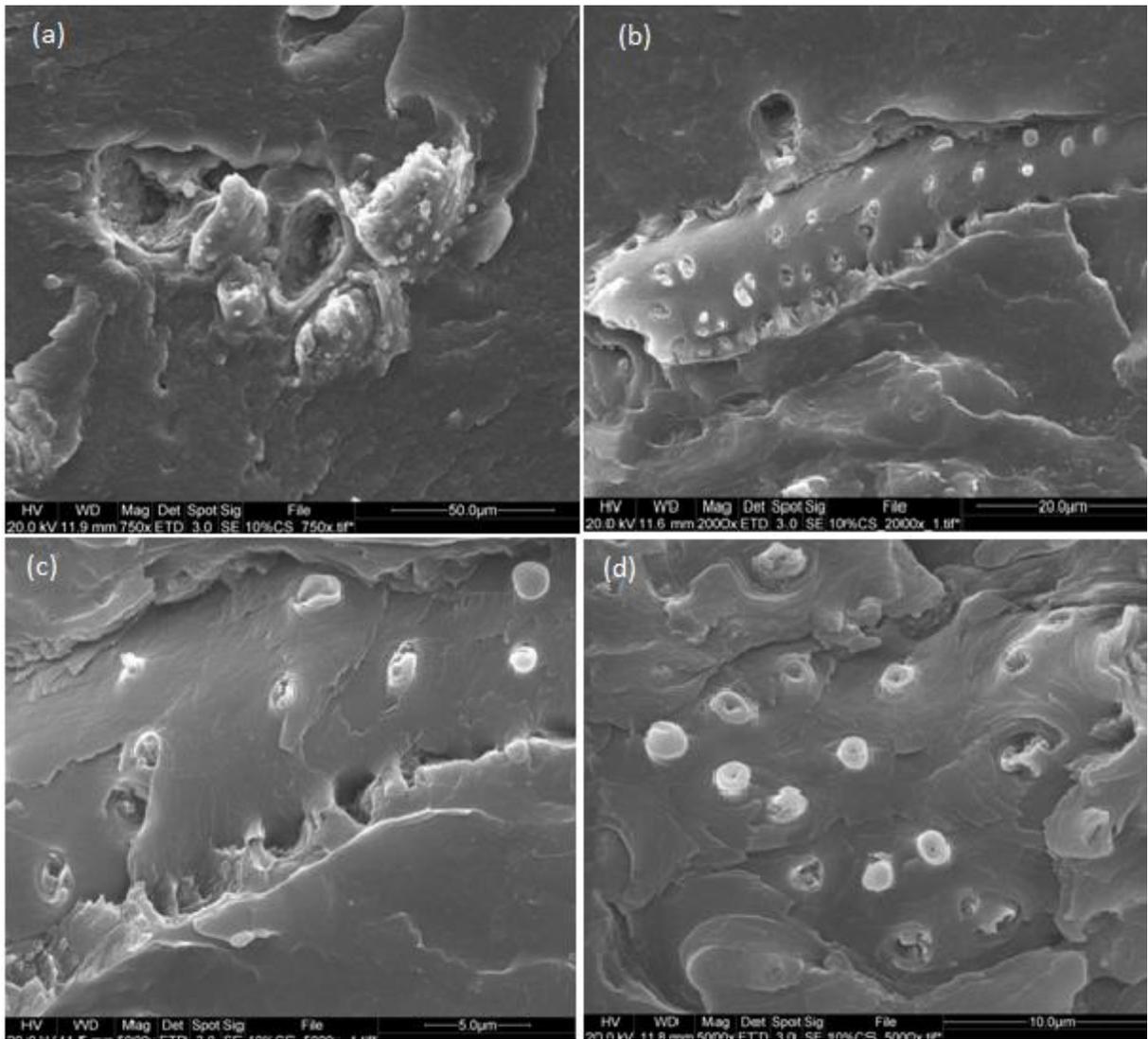
Figure 6 SEM micrograph shown an adhesion between nylon-6 and EFB fibres.
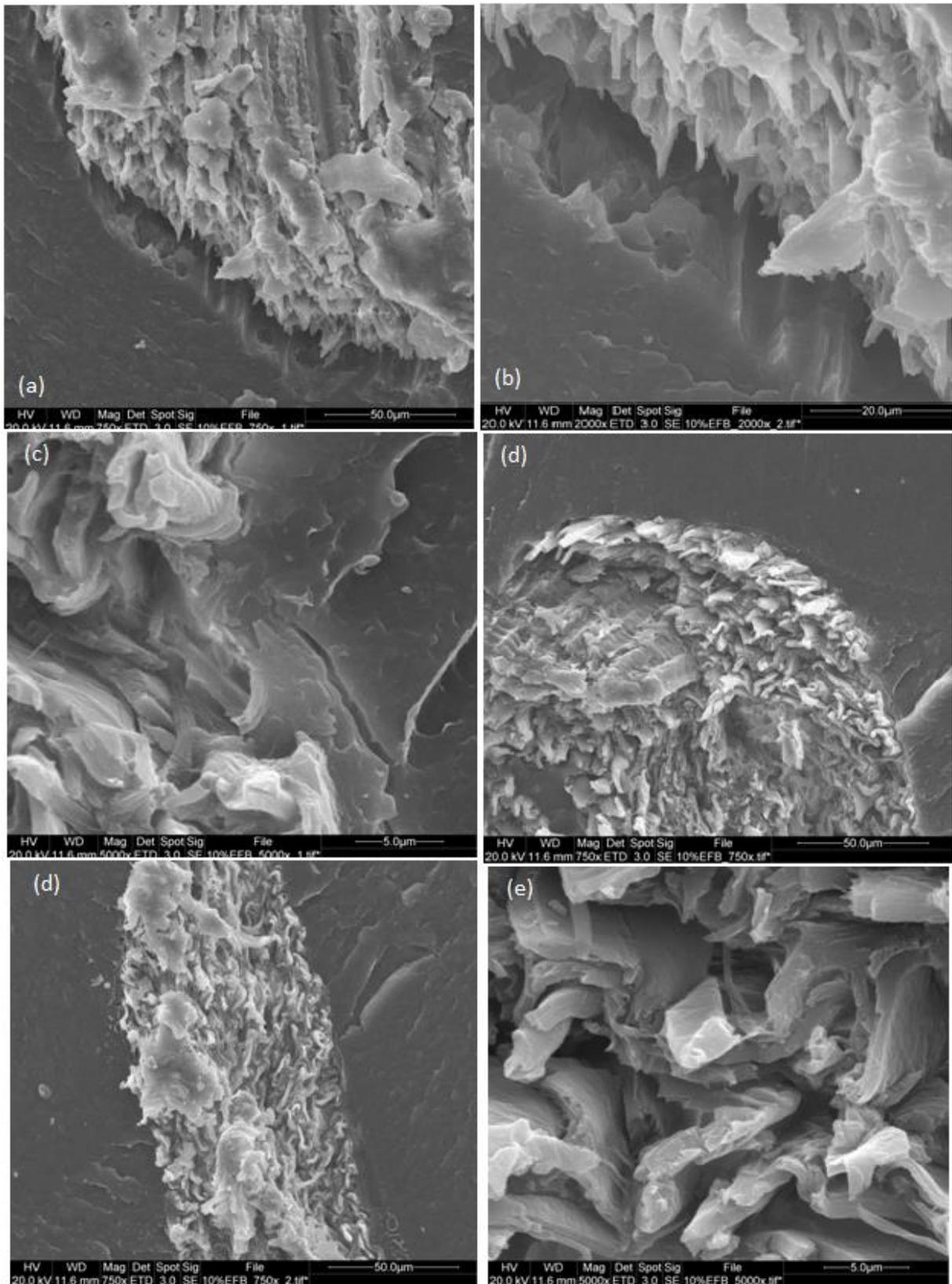
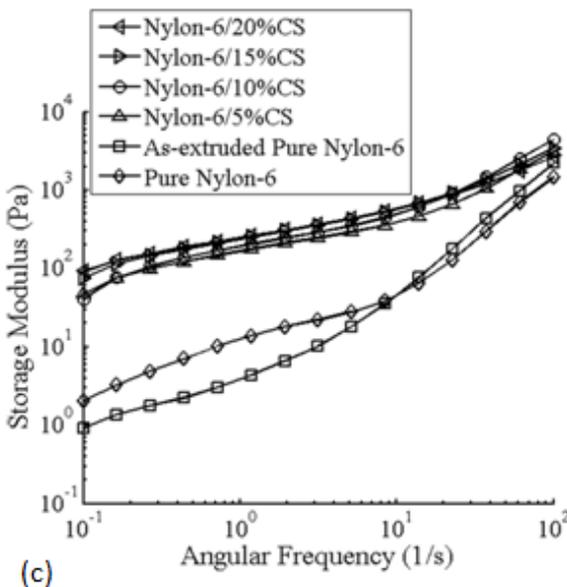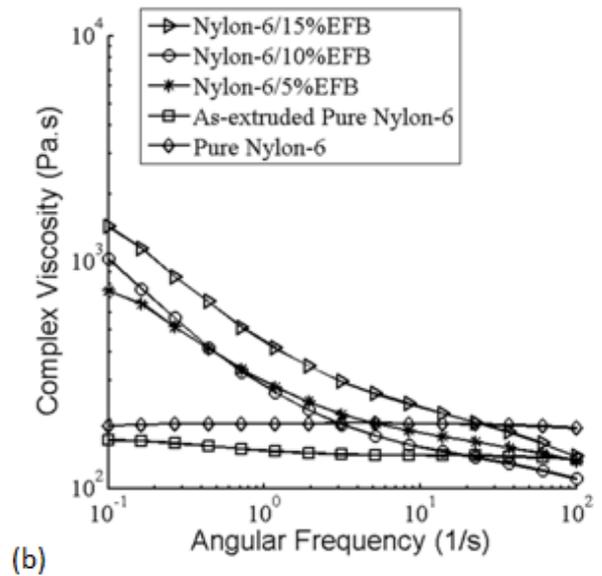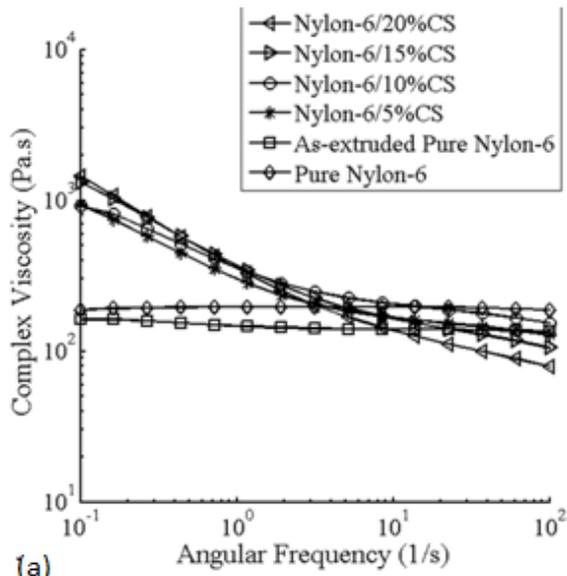Figure 7 SEM micrograph shown an adhesion between nylon-6 and CS particles.
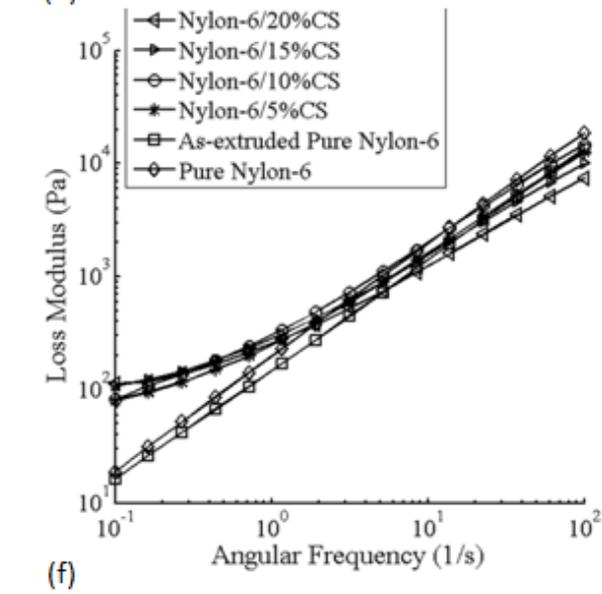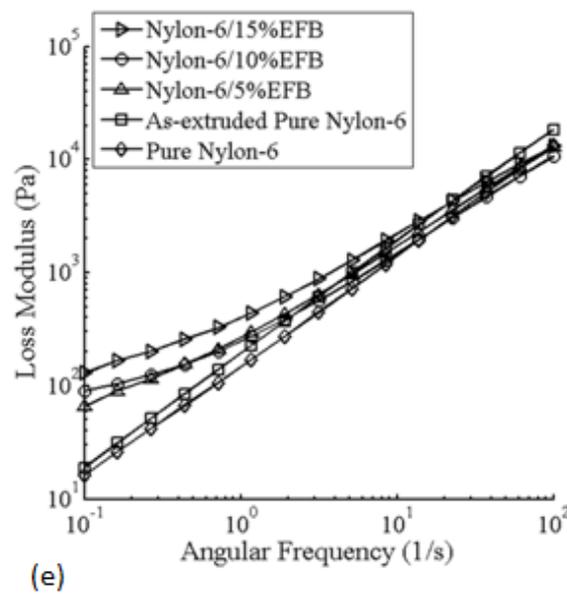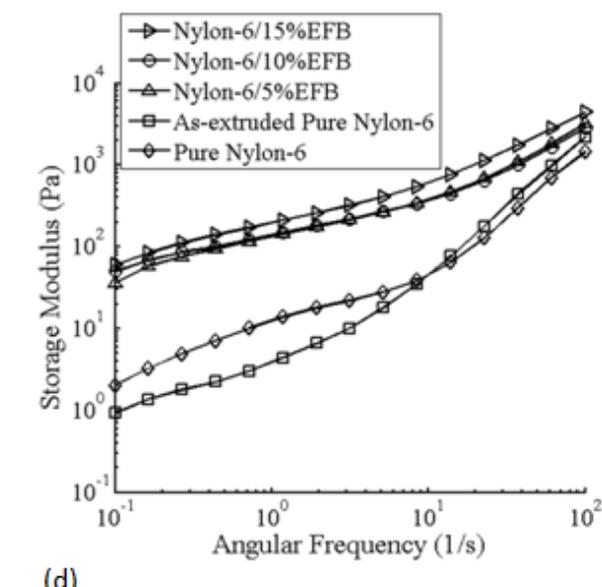
Figure 8 Rheological properties of nylon-6 based composite: (a) Complex viscosity versus angular frequency for pure nylon and nylon-6/CS (b) Complex viscosity versus angular frequency for pure nylon and nylon-6/EFB (c) Storage modulus versus angular frequency of pure nylon-6 and nylon-6/CS (d) Storage modulus versus angular frequency of pure nylon-6 and nylon-6/EFB (d) Loss modulus versus angular frequency of pure nylon-6 and nylon-6/CS (e) Loss modulus versus angular frequency of pure nylon-6 and nylon-6/EFB.
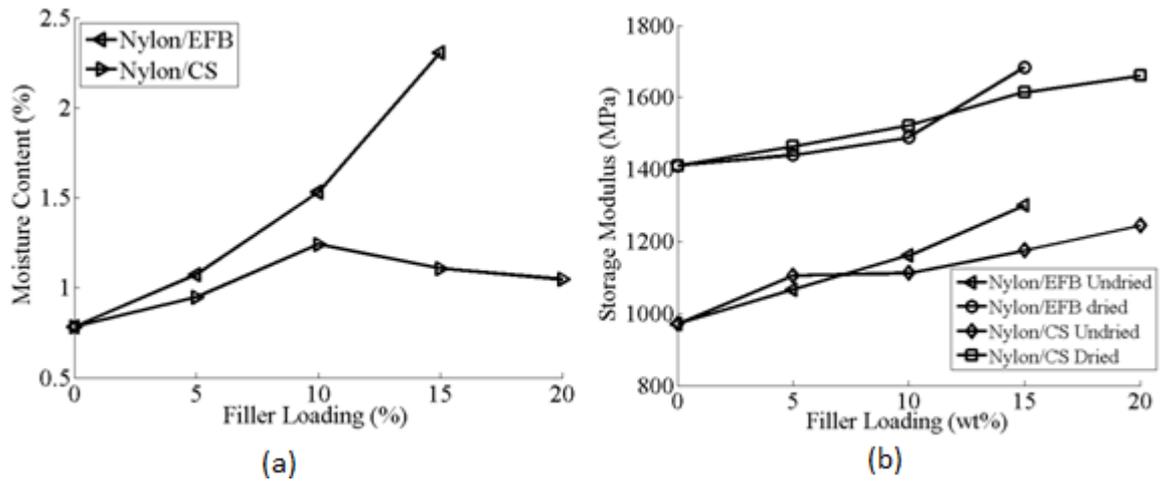

Figure 9 Moisture uptake as a function of fillers loading and affected storage modulus: (a) Moisture content of nylon/EFB and nylon/CS as a function of filler loading (b) Storage modulus of dried and undried nylon/empty fruit bunch and nylon/cocout shell as a function of filler loading.