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ORIGINAL ARTICLE

Investigation on the flexural properties of nanofillers loading on the Jute/Carbon/PLA nanocomposites

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ABSTRACT – Presence of fibers and fillers in a composite can be an efficient way to arrest crack either at macro or micro levels. In this work, woven jute and carbon fibers were arranged alternately in PLA (Polylactic Acid) nanocomposite. Graphene or nanoclay was embedded into PLA matrix to make polymer nanocomposite. Fiber reinforced polymer nanocomposites were prepared by varying the concentration of graphene or nanoclay in the PLA matrix and alternate woven jute/carbon fibers was then bind with the PLA nanocomposite. Influence of graphene or nanoclay concentration and presence of woven fibres in the composite was quantified by flexural analysis. Flexural strength and flexural modulus were found to increase at 3wt% of nanofiller concentration for both graphene/jute/PLA and nanoclay/jute/PLA nanocomposites with increment up to 37% and 31%, respectively. FTIR was used to determine the interaction between PLA and nanofillers. Morphology observation by Scanning Electron Microscopy (SEM) was done to investigate the fractured surface of the hybrid jute/carbon fibres reinforced PLA nanocomposite.

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INTRODUCTION

Currently, the lack of resources on crude oil has been escalated the global demand for synthetic based material [1] . Hence, this leads to the awareness of global communities to reduce the dependency of the synthetic materials which subsequently urged the plastic manufacturing industry to search for new alternatives and immediate actions [2]. Apart from that, the application of the conventional synthetic based composite has induced various environmental issues such as waste disposal and environmental sustainability [3].

To engulf this problem, necessary actions have been proposed by several researchers including the substitution of natural-based material and the application of recycle material from waste products. For instance,[4] have performed research on the fabrication of polymer composites with polylactic acid and cellulosic natural fibres. The wet-laid fibre sheet method with film stacking composite was used for the process making. The highest tensile strength result was shown about 121 MPa was achieved compared to the neat PLA. Moreover, a study conducted by Hinchcliffe et al.[5] have shown a research on the mechanical properties of natural fibre reinforced polylactic acid. The data research show increases of 14% and 10% respectively for flexural specific strength and rigidity while 116% and 62% were increased as well for the tensile specific strength as compared to solid and unreinforced PLA. Other than that, about 59.32% of tensile strength and 100% elongation were observed as the maximum increase for nano fibrillated kenaf cellulose (NFKC)-PLA composite with NFKC yielding at a blending speed and time of 15000 rpm and 15 minutes as compared to pure PLA. Nano fibrillated kenaf cellulose (NFKC) was derived from kenaf fiber after varying chemico-mechanical treatments were introduced into polylactic acid (PLA) as reinforcements to improve the mechanical and morphological properties of the biocomposites [6]. Thus, the implementation of biopolymers such as PLA is one of the suitable methods in reducing the dependency toward non-biodegradable polymer used in various sectors application.

The most recognized biodegradable polymers are aliphatic polyesters as for the example are polylactic acid (PLA), polyglycolic acid (PGA), polycaprolactone (PCL), and polyhydroxybutyrate (PHB) [7,8]. Due to good aesthetic, mechanical strength, thermal plasticity, biocompability and easy processability in most equipment, PLA was known that has the greatest commercial potential [8]. Polylactic acid (PLA) extracts from renewable sources by fermentation under controlled conditions of a carbohydrate source, including sugar cane, corn sugar and potato [9,10].

On the other hand, the development of natural fibre composites has been a topic of concern [11]. Because of their efficiency in composite materials, the use of natural fibre attracts interest for the researcher to study [12]. As a sustainable structural material, natural fibres have excellent potential choices with low density, biodegradable, ability to increase the composite strength, lightweight, low abrasiveness, reduce the cost of material and have a low environmental impact [13,14]. A huge range of natural fibre applications in composite materials can be found, for instance, housing construction materials, automotive parts, food packaging, and furniture [9,15].

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Jute is one of the examples of natural fibre. It is obtained from the jute plant's stem. The benefits of jute are it has high tensile strength, modulus, and high cellulose content. Jute can also be considered as an alternative material for reinforcement synthetic fibreglass and carbon fibre as jute has good specific mechanical properties, biodegradability, low cost of production and low density [16]. In this view, research by Berganu et al. [15] was carried out on the mechanical behaviour of jute fibre reinforced polypropylene composites. The result of this research reveals that the addition of the jute fibre reinforcement significantly enhanced the mechanical characteristics of composites based on polypropylene. It also observed that the weight percentage of the jute fibre reinforcement increases up to 40% and make an increase in the mechanical properties as well. This was due to the high interfacial bonding between the fibre and the matrix which was when the increment up to 40% of the jute fibre reinforcement in the polypropylene-based composites [17].

The main component that provides high tensile and flexural strength to jute fibre is cellulose. The cellulose content in jute fibre was found to be in the range of 61-71% from overall constituents. With a large number of impurities like pectin, lignin, hemicellulose, oily, and waxy substances that covered the fibre surface, it reduces the compatibility of hydroxyl group which is present on the fibre surface to react with the hydrophobic polymer matrices [18]. Thus, it forming mechanical interlocking adhesion with non-polar matrices because of the hydroxyl groups are not being able to react with the polar matrices [11]. As a result, this impurity will lead to poor interactions between the fibres and the matrix and in fact, there will be a void formation in the polymer composite. To overcome this problem, surface treatment can be carried out to improve the fibre/matrix adhesion, while subsequently removed the impurities that covered the fibre's surfaces [18].

Gopinath et al. [19] examined that jute treated with 5 wt% NaOH perform better tensile strength than jute treated with 10 wt% NaOH. This occurred due to the factors like presence of air voids, fibre agglomeration, fibre-matrix adhesion, dispersion and the orientation of the fibres. It was expected that treatment with NaOH tends to remove impurities such as pectin that can aid in increasing the tensile properties of the jute fibre composite [11]. Moreover, the young modulus of the polymer composite filled with treated jute was found high up to 17% compared to untreated jute filled in polymer composite. It has been suggested that alkali treatment on the jute fibre can leach out the fatty acids and some other lignin components that can increase the jute amorphous region [20].

Carbon is one of the most common reinforcements for polymer matrix composites. Besides, the use of carbon fibre reinforced polymer matrix (CFRP) composites as well could result in a weight decrease of 40-60% notably[21]. Other than that, this reinforcing material is well recognized as it has superior properties such as high mechanical strength, high elasticity modulus, have good flame resistance and low density. Carbon fibre has huge application in the sector of engineering, for instance, are constructions, sports equipment, ships, aircraft and automobile. However, the brittleness of carbon fibre makes carbon fibre composites very susceptible to stress concentration. Additionally, it requires expensive processing of carbon fibre. There is one way how to strengthen the weakness of the carbon fibre reinforced plastic (CFRP) which is replacing some of the carbon fibre with the ductile fibre. This process is called hybridization [22].

Hybrid composite is a combination of two or more types that reinforced a prevalent matrix. Although in many distinct schemes, hybrid composites can be generated, the most studies on their mechanical characteristics was restricted to the settings in which fibres were closely blended or purposely arranged within the matrix. A general rule of mixtures method may be used to quantify a material property with regard to volume concentration of its constituents when assessing the mechanical characteristics of hybrids [23,24]. In several lightweight and high-strength applications, hybrid composite materials effectively replaced traditional materials. The growth of composite materials increases their efficiency by reinforcing two or more fibres in a single polymer, resulting in the advanced material scheme called hybrid composite with a wide variety of material characteristics. Thus, the mechanical characteristics such as flexural strength, tensile strength, hardness and compressive strength need to be investigated. A three-point bending is used most commonly to determine the flexural strength in which a loading nose deflects a specimen up to fracture at a fixed span and load rate [25].

Zhang et al. [26] reported that the outcomes indicate that when the carbon layers are on the outside, hybrid composite laminates with a 50% carbon fibre reinforcement provide the best flexural characteristics, while the alternating carbon/glass lay-up offers the greatest compressive strength. In an epoxy matrix, hybrid composite laminates were produced using a varying ratio of glass woven fabric and carbon are woven fabric. In composite coupons comprising multiple proportions of carbon fibres to glass fibres, static experiments including three-point bending, tension, and compression were performed.

Use of nanofiller in polymer matrices has been one of the well-known material used by many researchers to form advanced materials properties applications. Graphene is a material that has the multifunctional reinforcement for polymers. From previous studies, the inclusion of graphene in polymer shows improvement in mechanical, thermal and electrical properties compared to the blank polymer [27-29]. For instance, Zhao et al. [30] investigate the variation of mechanical and thermal properties in sustainable with the presence of graphene oxide in epoxy. The result obtained from this study stated that 49% of flexural strength, 21% of flexural modulus, 80% of tensile strength, 97% of critical strain energy and 69% of critical stress intensity has ascended due to the addition of 0.05-0.5 wt% of the functional graphene oxide. For thermal analysis, it was observed that there is 17°C increment in the thermal decomposition temperature of functional graphene oxide/bio-based bis-furan di-epoxide compared to a blank sample of bio-based bis-furan di-epoxide itself. Thus, they concluded that the inclusion of graphene oxide tends to improve the mechanical and thermal properties of the epoxy.

From an experimental study done by Dominick et al. [31], they found that the addition of only 2 wt% of nanoclay, showed improvement in the glass transition temperature (Tg), modulus, thermal stability, electrical conductivity and free

volume of the epoxy nanocomposite. Next, Lim et al. [32] demonstrates that the effect of nanoclay filler on the mechanical and morphological properties of Napier/epoxy composites. The author stated that the increase of the interfacial bonding is due to the better dispersion and the distribution of nanoclay in the epoxy resin. The highest flexural strength obtained is with the inclusion of 3 wt% of nanoclay which showed about 163% improvement compared with neat Napier/epoxy composites. Therefore, in this study, the effects of different loading of nanofillers on the flexural properties for Jute/Carbon/PLA nanocomposites were investigated.

METHODS AND MATERIALS

Sodium hydroxide (NaOH) and distilled water are used to treat jute mat surface treatment. The carbon fibre mat was obtained from pyrofil department, Tokyo Japan. Polylactic acid (PLA) was supplied from a NatureWorks LLC product (Ingeo biopolymer 7001D). Graphene and nanoclay were used as nanofillers in the hybrid composite and were purchased from United Nanotech Innovations and Aldrich, respectively.

Treatment of Woven Jute Fibres

For a treatment process on the woven jute fibre, 5% of NaOH solution were prepared. Then, the jute fibre mat was soaked into the container filled with the NaoH solution as shown in Figure 1. The fabrics were kept for 2 hours in NaOH solution and after that, the woven fibre mats were rinsed thoroughly with distilled water until reached pH 7. The fibres were finally dried for 24 hours at room temperature, followed by oven drying for 2 hours at 100°C.

Figure 1. Jute treatment

Fabrication of Composites

Nanofillers with designated concentration from 1 to 5wt% (nanoclay or graphene) was mixed with chloroform. The solution was then mixed using the ultrasonic probe for 30 minutes to improve the nanofiller dispersibility and reduce agglomerations of the nanofillers. Next, polylactic acid (PLA) was added into the mixing solution and continue stir by using a mechanical stirrer for another 2 hours. Then, the nanofiller/PLA solution was cast onto a mould to make a thin film. The thin film thickness is approximately 1mm. The thin film was left in the oven at 40°C for 12 hours to dry. Then, the thin film was applied alternatively to the fibres. The arrangement of woven jute and carbon fibres as shown in Figure 2. The combination of woven jute, carbon fibre and PLA nanocomposites were stacked together and placed onto hot press equipment. The hot press was set to preheating at 185°C for 20 minutes and followed pressing for another 30 minutes to obtain the desired thickness according to ASTM D790. Table 1 shows the designation for every sample.

Table 1. Sample abbreviation name for hybrid polymer nanocomposites

Sample abbreviation	Sample name
CH	jute fibre/carbon fibre/PLA
G 1	1wt% of graphene in jute fibre/carbon fibre/PLA
G 3	3wt% of graphene in jute fibre/carbon fibre/PLA
G 5	5wt% of graphene in jute fibre/carbon fibre/PLA
NC 1	1wt% of nanoclay in jute fibre/carbon fibre/PLA
NC 3	3wt% of nanoclay in jute fibre/carbon fibre/PLA
NC 5	5wt% of nanoclay in jute fibre/carbon fibre/PLA

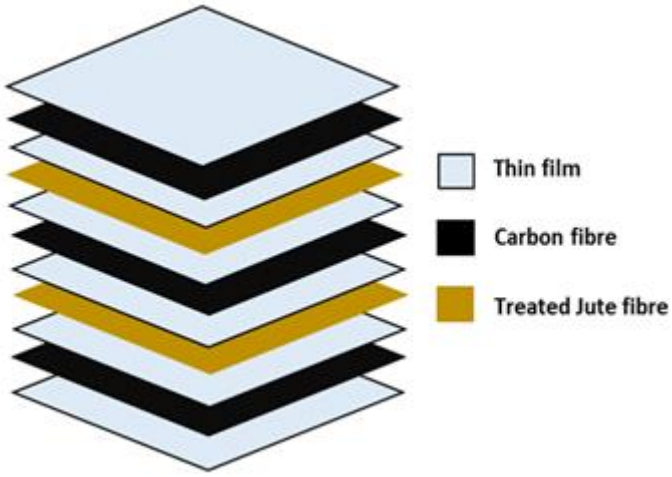

Figure 2. Fibre reinforced PLA nanocomposite mat arrangement

Flexural Test

The flexural test was done by using three-point bending according to ASTM D790. The crosshead speed and support span were 1.28 mm/min and 48 mm, respectively. All specimens were cut following the dimension of rectangular shape which was 3.5 mm (T) x 12.7 mm (W) x 127 mm (L). The flexural test was conducted as shown in Figure 3.

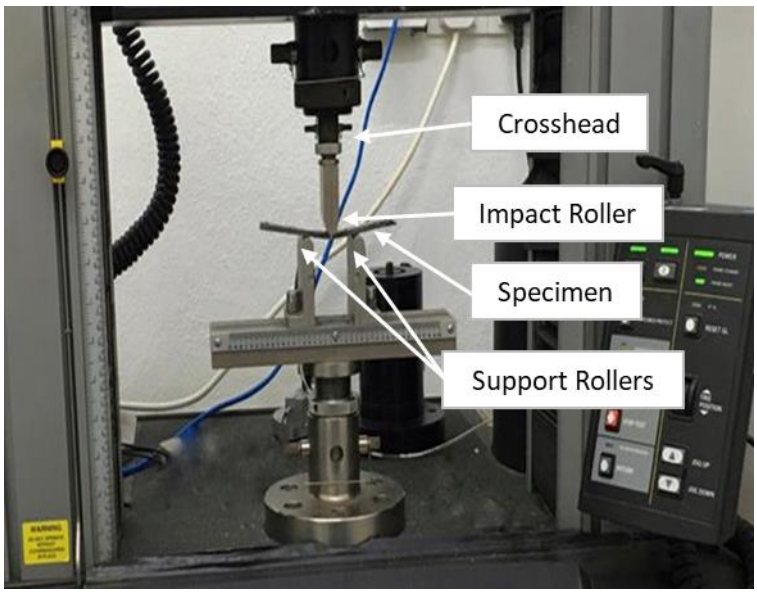

Figure 3. Three-point bending test on fibre reinforced PLA bionanocomposite

Morphology Analysis

The morphology of the flexural fractured surfaces of the fibre reinforced PLA nanocomposite was observed and analysed using scanning electron microscope (SEM) type EM-30AX. Initially, the samples were sputter-coated with the gold mixture to avoid subsequent charging before measurement by SEM. Then, the nanocomposite sample was ready for viewing using SEM.

FTIR Analysis

Possible bonding interactions and the presence of functional groups were characterized by using fourier transformed infrared spectroscopy (FTIR, Nicolet Is10 OF Nexus analytics). For the nanofillers, approximately 2mg of the nanofiller powder was mixed and grounded with KBr powder. Then the mixture formed into a thin transparent pellet. The spectra were performed at a resolution of 4cm^{-1} for 16 number of scan in the spectrum range of 280 to 4000cm^{-1}.

RESULTS AND DISCUSSION

Mechanical Properties in the Flexural Analysis

The mechanical properties of the hybrid fibre PLA nanocomposite were analysed by flexural test. In the flexural test, initially the surface of the specimen will be subjected to greater strains than at the centre. The surface of the composite for flexural analysis whether on top or bottom, faced the compressive fracture. In this work, the polymer binder consists of a PLA mix with either nanoclay or graphene. Figure 4 illustrates the overall flexural behaviour for hybrid fibre PLA nanocomposite filled with nanoclay or graphene until fracture. For the PLA filled with fibre and nanofiller, it was found that the stress of the hybrid fibre PLA nanocomposite start to increase as highlighted in the red box region. This indicates a stress transfer from the PLA nanocomposite (either at the top or bottom surface) to the next layer of the composite which is the woven fibre mat which determine the initial strength of the hybrid fibre PLA nanocomposite. High aspect ratio and good miscibility of nanoclay or graphene in PLA, will subsequently boost the stress transfer within the materials in the composite and cause the flexural strength to increase. The addition of nanofiller makes the hybrid fibre PLA nanocomposite become stiffer and enable the stress transfer from nanofiller to the molecular structure of the polymer [32]. Good interfacial adhesion between nanofiller and PLA matrix, can increase the efficiency of stress transfer compared to the hybrid fibre PLA composite (unfilled with nanofillers, CH sample). For the CH sample, the stress was found to be the lowest while the strain of the composite was the highest. Without the presence of nanofillers, the PLA molecular chain is free to move without restriction which make the strain value higher compared to other samples.

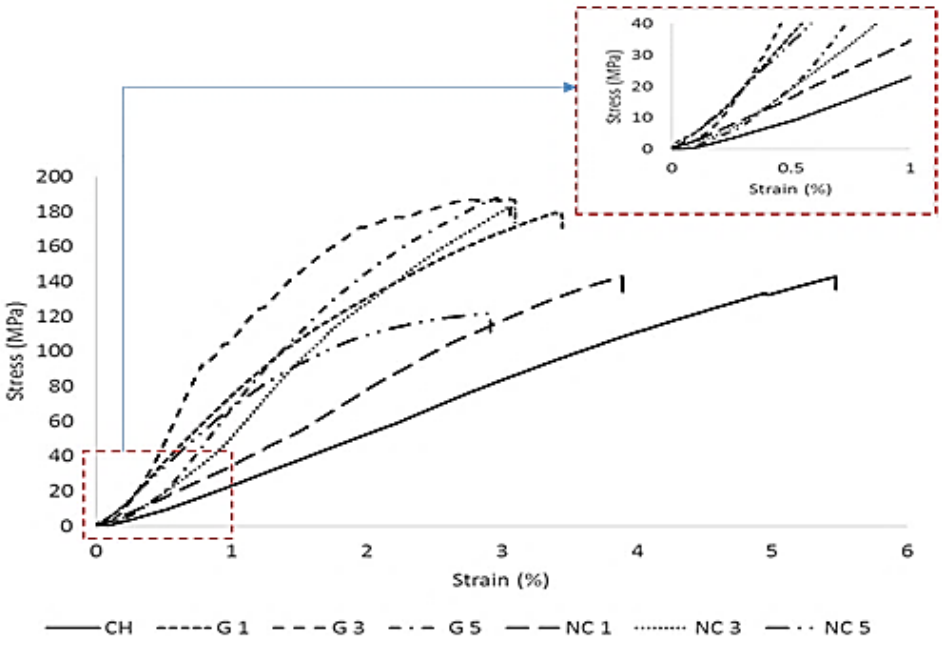

Figure 4. Stress strain behavior of hybrid fibre PLA nanocomposite

From Figure 4 also can be observed that sample G 3 which is filled with 3 wt% of graphene shows the highest ultimate flexural strength value compared to others. For the hybrid fibre PLA nanocomposite filled with nanoclay, sample NC 3 which consist of 3 wt% nanoclay present the highest value which is 182.771 MPa. As shown in Table 2, sample G 3 value

is about 5 % higher compared to sample NC 3. Thus, can be presumed that the presence of nanoclay or graphene can affect the properties of the hybrid fibre PLA composite. Increase in flexural strength for both hybrid

fibre PLA nanocomposite filled with nanoclay or graphene can be described in a way that the nanofillers act as load carriers which transfer stresses from PLA through the nanofiller, that lead to the effective and uniform stress distribution [33]. Further increase in concentration of nanofiller to 5 wt%, results in deterioration of the flexural strength. Drop in flexural strength was significant for sample NC 5. By increasing the nanofiller concentration, the distance between particles become smaller, thus increase the possibility of the formation of agglomeration. This occurs due to the Van der Waals forces that attract the particles together and Lead to low flexural strength [34]. The effect of the agglomeration due to poor dispersion of the nanoclay in the matrix which results in non-uniform stress distribution across the laminate and exhibits poor mechanical properties.

Table 2. Influence of graphene and nanoclay nanofiller on flexural properties of nanocomposite

Sample	Flexural properties	
	Flexural strength (MPa)	Flexural modulus (GPa)
CH	140.034	3.179
G 1	179.948	7.096
G 3	192.164	9.125
G 5	161.368	7.243
NC 1	143.282	5.972
NC 3	182.771	8.133
NC 5	126.174	6.247

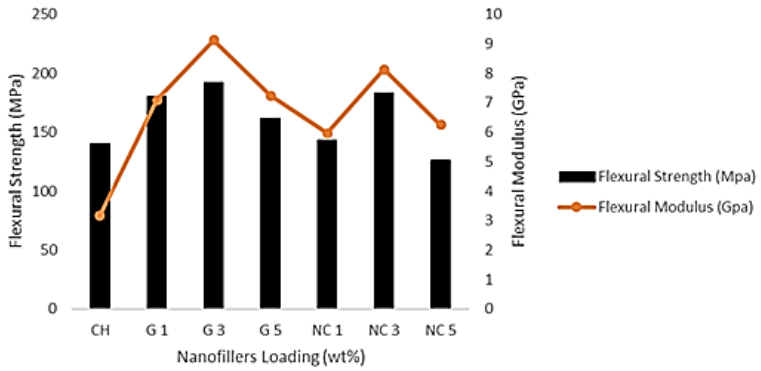

Figure 5. Relationship between flexural strength and flexural modulus of hybrid fibre PLA nanocomposite

Figure 5 shows the relationship between flexural strength and flexural modulus of the hybrid fibre PLA nanocomposite samples. As can be seen the trend for both flexural strength and flexural modulus seems to be nearly similar. The hybrid fibre PLA nanocomposite filled with graphene or nanoclay shows an increment in flexural modulus as stated in Table 2 for about 187% for G 3 sample and 96% for NC 3 sample compared to the hybrid fibre PLA composite (CH). As the amount of nanofiller increase, the flexural modulus was found to be increased. Increment in flexural modulus can be due to the addition of nanofillers which makes the composite become more stiffer than unfilled nanofiller composite sample. Formation of hydrogen bonding between nanoclay silicate layers with PLA matrix and degree of dispersion of the silicate layers plays an important role in improving the flexural modulus of the NC sample up to 3 wt% nanoclay concentration. However, it has been shown in Figure 5 as well that the G 3 sample exhibited larger flexural modulus which indicate that the sample has a higher stiffness compared to unfilled and NC samples. Increase in stiffness of the hybrid fibre PLA nanocomposite filled with graphene can be attributed to effective stress transfer and high resistance to plastic deformation [35]. By increasing further the nanofiller content to 5 wt%, the viscosity of the PLA during processing was found to be increased which lead to the poor dipersibility in PLA media. Thus, the nanofillers tend to agglomerate and reduce the flexural modulus value. Increasing the concentration of nanofiller, can increase the stress concentrations in the hybrid fibre PLA nanocomposite and results in reducing flexural modulus [36].

Scanning Electron Microscopy (SEM) Analysis

Figure 6 shows the micrographs of the specimen subjected to fractured from flexural test. It can be seen clearly in Figure 6 shows the layered of hybrid fibre polymer composite which consist of only carbon and jute fibre as reinforcement materials and polylactic acid (PLA) as a binder. Synthetic fibre such as carbon fibre has a smooth fibre structure and the SEM image indicate a small gap between carbon fibre and PLA matrix which explained poor wetting by PLA (red arrow). In addition, lack of PLA matrix can be observed in the carbon fibre region. From Figure 7 also can be clearly seen that the treated jute fibre is not straight as carbon fibre and the PLA is attached well on the fibre surface. The chemical treatment which has been done on the jute fibre tend to increase the concentration of oxygen functional groups and imparts better compatibility with PLA matrix.

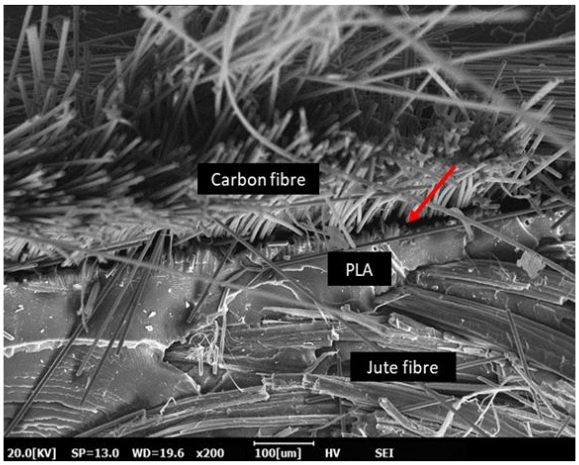

Figure 6. SEM micrograph of hybrid fibre PLA composite

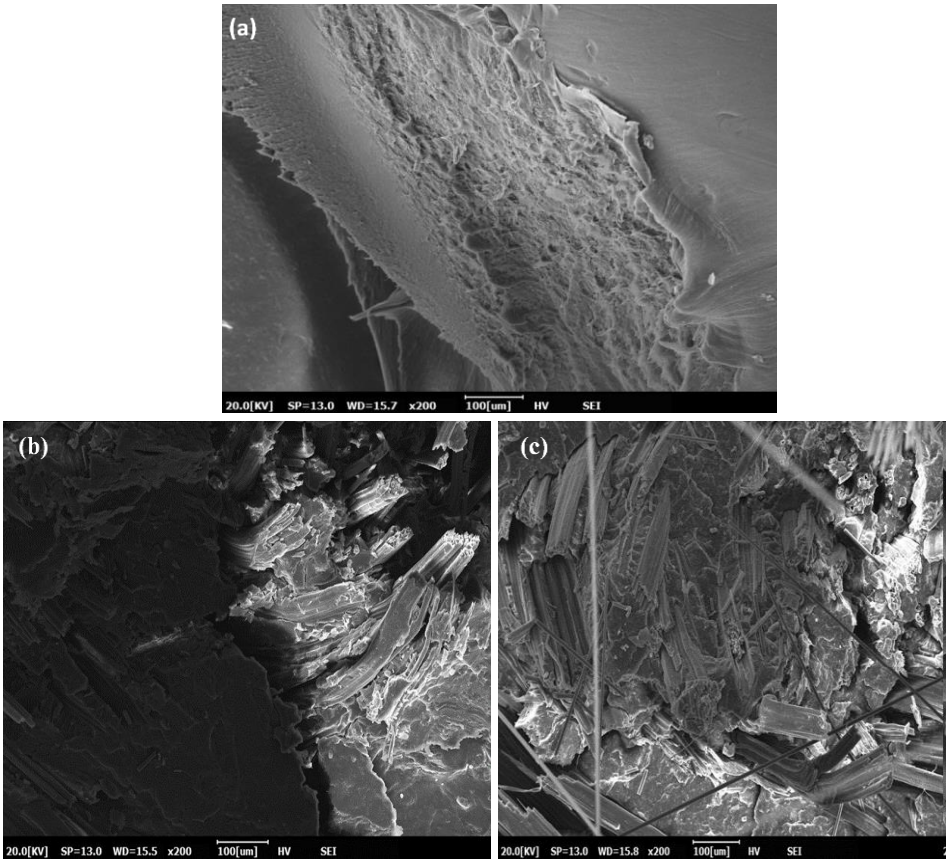

Figure 7. SEM micrographs: (a) PLA matrix, (b) hybrid fibre PLA nanocomposite filled with graphene and (c) hybrid fibre PLA nanocomposite filled with nanoclay

Figure 7(a) shows the SEM images of the PLA matrix film. As can be seen clearly the fractured surface consist of smooth and rough region. Presence of rough surface indicates, may suggest a good interracial interaction when jute fibre is added as reinforcement. Surface modification on the jute fibre tend to disrupt the hydrogen bonding on the fibre structure and create better interfacial interaction with PLA matrix. The surface treatment on jute fibres not only improve the wettability with PLA, but also remove or reduce the presence of unwanted material such as lignin, wax and oils that covering the jute surface.

Figures 7(b) and (c) shows the fractured surface of the nanocomposite. As has been discussed earlier in flexural modulus, addition of 3wt% of graphene (Figure 7b), increase the viscosity of the PLA matrix and make the composite become stiffer. The dark region on the left side shows the brittle failure mark of the sample. In addition, the presence of gap indicates high local stress concentration and lead to initiation of crack. The crack is branched and deflects its direction until it reach failure. Micrograph in Figure 11c shows the fractured surface of hybrid fibre PLA nanocomposite filled with 3 wt% of nanoclay. The sample exhibit a ductile fracture pattern in which necking, and stretching marks can be observed in the fractured sample. This is in agreement with the flexural strain analysis, which shows that hybrid fibre PLA nanocomposite filled with NC have higher strain value compared to hybrid fibre PLA nanocomposite filled with graphene. Thus, can be assumed that incorporation of different types of nanofillers in PLA matrix can change the fractured surface pattern.

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FTIR was performed on PLA thin film NC/PLA and G/PLA nanocomposite to understand the chemical compositions of the materials (Figure 8). In the FTIR analysis, 1 wt % of nanofiller was added into the PLA matrix. A strong absorption band for NC/PLA, G/PLA, and PLA were at around 2997.14, 2997.07, and 2997.06 respectively which was corresponding to stretching vibration of C-H from alkene. The stretching frequencies for the PLA thin film were C-O, C=O and C-H at 1081.03, 1382.59, 1747.13, and 2997.06, respectively. Bending frequencies for C-H has been identified at 1382.59. The absorption peaks between NC thin film and G thin-film almost the same. Both thin film exhibit strong absorbance peak at 1747.24, and 1747.25, respectively, which indicates the presence of C=O stretching from the ester. It was found that peak intensity was increased than PLA thin film. There is strong interaction in polymer blends when shifting of peaks in IR spectra is observed [37]. In the case of the most hydrophilic nanoclay composites, the wideband at 1350−1400 in PLA, corresponding to the symmetric deformation vibration [38]. Thus, lead to an increment in flexural result properties of hybrid composite even though at 1wt% concentration of nanofiller into the PLA matrix as have been shown in Figure 8. The nanofillers in the PLA seems to create an interpenetrated network of particle and small aggregates. Thus the addition of nanofillers created a possibility of improving the synergistic effect between nanofillers and PLA.

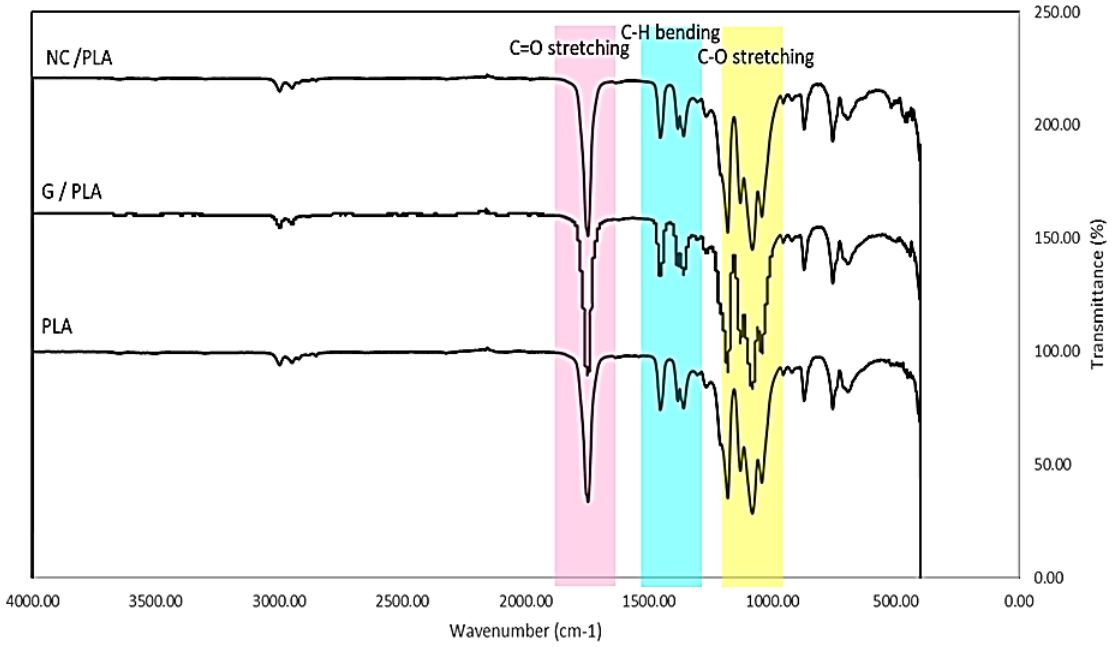

Figure 8. FTIR spectra of PLA thin films, NC/PLA and G/PLA nanocomposites

CONCLUSIONS

The treated jute fibre and carbon fibre reinforced hybrid composites have been fabricated by using a hot press machine. Experimental evaluation of flexural testing of hybrid composites as per ASTM standards has been completed. As a conclusion, from this research, the experimental result showed that the highest flexural strength was the inclusion of 3wt% of graphene loading in the hybrid composite which is 192.164 MPa approximately. While the highest inclusion for the inclusion of nanoclay nanofiller in hybrid composite was NC 3. The fact that interfacial interaction, the concentration of nanofiller, and the homogenous properties with good dispersion were can affect the flexural properties. As concluded, the presence of nanofiller shows significant improvement in the hybrid composite than the neat composite.

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