PERFORMANCE OF KAPOK FIBER REINFORCED POLYVINYL ALCOHOL BICOMPOSITE BY ALKALI TREATED

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ABSTRACT. *Raw Kapok (Ceiba pentandra) fibre was initially washed and dried before undergoes chemical treatment. Upon dried, the kapok fibre was bleached and delignified at room temperature, in an acidic solution containing 6% of sulphuric acid and 4% hydrogen peroxide to remove hemicellulose and wax. The treated kapok was filtered and washed thoroughly with distilled water and vacuum dried at 60 ^oC for 10 hours. Finally, the treated kapok was converted to alpha-cellulose (α-cellulose) by alkali treatment. In this step, treated kapok was immersed in 17.5% of sodium hydroxide solution for 30 minutes at a temperature of 50 ^oC to remove alkali-soluble components. The obtained α-cellulose, termed as alkali-treated kapok fiber (AKTF) was filtered, washed thoroughly with distilled water until pH is neutral and vacuum dried at 60 ^oC for 10 hours. In this stage, a certain weight of ATKF (0%, 10%, 20%, 30%, and 40%) were mixed with a hot solution of PVA and dried at room temperature. In the mechanical test, ATKF – PVA biocomposite shows an increase in tensile strength and elastic modulus up to 30% content of kapok fibre but drop at 40% kapok loading. The result shows that both ATKF – PVA biocomposite film (30%) were having the highest mechanical properties among the others and was chosen for next characterizations. It is evidence in FTIR spectra that the composites indicate the formation of new hydrogen interaction between kapok fibre and PVA which might help to improve the mechanical properties. As for XRD analysis, the ATKF – PVA biocomposite film (30%) blend was found to be a heterogeneous as the peaks of diffractogram were overlap each other. This is supported by SEM micrograph in which ATKF – PVA biocomposite (30%) show a heterogeneous phase. Additionally, in the TGA data, ATKF – PVA biocomposite (30%) was founded less thermally stable than raw kapok and pure PVA is the least thermally stable among other samples.*

KEYWORDS. PVA; Kapok fiber; Biocomposite; Mechanical Properties

INTRODUCTION

Kapok tree or *Ceiba pentandra* is belong Bombacaceae family which mostly grows in Asia, West Africa and Central America region. Kapok also refers to the fluffy cotton-like that obtained from seed pods. The physical appearance of kapok is silky, yellowish or brownish colour (Zheng *et a*l., 2015). According to Fengel and Przyklenk (1986), main composition of kapok fiber consists of cellulose, lignin and xylan. Their composition percentage however, were *inconsistently reported. A study conducted by* Kobayashi *et al.*, (1977) has revealed that kapok fibre is composed of about 64% of cellulose, 13% of lignin and 23% of pentosan on a weigh basis, while another study found that kapok fiber composed of cellulose (35 %), xylan (22%), and lignin (21.5%) (Hori *et al.*, 2000). Recent study shows that cellulose content in the kapok fiber is 50.7% (Tye *et al.*, 2012). Kapok fibre is mostly used as filling material in mattresses, pillows or as oil absorbing materials (Abdullah *et al.*, 2010) and water-safety equipment due to its excellent buoyancy (Zhang *et al.*, 2013).

Polyvinyl Alcohol (PVA) is a water-soluble synthetic polymer which widely used as a matrix in the composite. It possesses many excellent properties such as non-toxic, biocompatible, good mechanical properties and good film-forming capability (Srinivasa *et al.*, 2003). PVA also water-soluble and hydrophilic in nature (Qiu, & Netravali, 2013) make it easier to work on. Compared to other man-made polymer such as polyvinyl chloride (PVC) and polyethylene (PE) which are non-biodegradable, PVA can be considered as biodegradable synthetic polymer due to the hydroxyl groups that presence on carbon atoms and its degradability can be improved by hydrolysis process (Gaaz *et al.*, 2015). PVA has the idealized formula $[CH_2CH(OH)]n$ and can be combined compatibly with organic or inorganic materials due to the presence OH groups and formed hydrogen bond to form PVA composites. Multiple studies showed PVA based composite was successfully applied in tissue scaffolding, filtration material, protective coveralls and pharmaceutical applications (Abdulkhani *et al.*, 2013). Many previous researches were using PVA as matrix in combination with different fillers such as seaweed, cotton and kenaf. For example, Puttaswamy *et a*l., (2017) has fabricated PVA reinforced cellulose microfibers from jatropha seed shell in his research. He found that the tensile strength of the composite was increasing as filler content increase. Another study by Laxemeshwar *et al.*, (2012) using microcrystalline cellulose blend with PVA, shows that the tensile strength of modified cellulose to PVA, 95 + 05 is the strongest while 10 + 90 is the weakest.

METHODOLOGY AND MATERIALS

1. Materials

Chemicals used in this research were of analytical grade. They were purchased from Aldrich Co. and used without further purification. Kapok fiber was purchased locally at Kota Kinabalu Sabah market. Before chemical treatment, all the impurities such as leaves, fruit skin and seed was manually removed.

2. Preparation of Kapok Fiber

a) Delignification and Bleaching

About 7 g of raw kapok fiber was heated in 100 ml of solution (a combination of 25 ml of 4% hydrogen peroxide, H_2O_2 and 75ml of 6% sulphuric acid, H_2SO_4) (Asik *et al.*, 2016). The solution was stirred and heated at 70 ^oC for 1 hour. In order to recover the loss of solution during heating process, 100 ml of the solution was added into the solution every 20 minutes. Next, the slurry was vacuum filtered by using polyester cloth and then washed with distilled water several times until the pH of the filtrate reaches a neutral condition at pH 7. Finally, the wet cellulose was vacuum dried for 10 hours at 60 ^oC.

b) α-Cellulose Conversion

The slurry obtained from section 3.4.1 was further treated again with 17.5% of sodium hydroxide, NaOH to obtain α-cellulose. The solution was stirred for 30 minutes at 50 ^oC until the colour of the mixture turn to milky white. The slurry was vacuum filtered using polyester cloth and then washed with distilled water several times until the pH reaches 7. Lastly, the wet α- cellulose was vacuum dried for 10 hours at 60 ^oC. The cellulose obtained in this section was termed as alkali treated kapok fibre (ATKF).

c) Preparation of Kapok Fiber/PVA Biocomposite

A series amount of an alkali treated kapok fibre (ATKF) was weighed and soften by addition of 2 ml of boiling water. Meanwhile, PVA solution was prepared by dissolving 4% of PVA pallets in 100 ml distilled water which make the concentration of PVA solution produced is 4 wt % (w/v). PVA solution is heated at 75 ^oC and stirred with magnetic stirrer to ensure PVA solid is fully dissolved. Next, the wetted alkali treated kapok fiber (ATKF) was mixed and stirred for 10 minutes until the mixture became homogenous. The mixture was casted on metal tray and let it dried in fume hood for 24 hours at room temperature. The dried ATKF biocomposite was peeled, cut into size and subjected for characterization.

3. Characterization of Kapok Fibre - PVA Biocomposite Film

a) Mechanical Properties of Kapok Fibre - PVA Biocomposite

The tensile test was carried out using universal testing machine (Gotech AI-7000M) according to ASTM and all testing was done at room temperature. Tensile strength was expressed as the maximum tensile strength divided by the initial cross-sectional area. The specimens were cut at dimension 110 mm (L) x 30 mm (W) x 0.5 mm (T). Each test was repeated 3 time with different specimens and the average value was calculated.

b) Functional Group Determination of Kapok Fibre - PVA Biocomposite

Fourier Transform Infrared Spectroscopy (FTIR) was used to detect the presence of functional group in kapok/PVA biocomposite thin film. 2 mg of sample was weighed and analyzed with FTIR (100 series, Perkin –Elmer) and pressed onto disc with 1 mm thickness. The range of absorption band recorded in between 600 – 4000 cm^{-1}.

c) CrystallinityDetermination of Kapok Fibre - PVA Biocomposite

X-ray diffraction (XRD) is a method in determination of crystallinity of a compound. The X-ray diffraction measurement were carried out using Expert Pro (Philipes EW3040/60) with Cu Kα radiation operating at 40 kV. The scattering angle covered the range from 2ϴ of 5^{o} to 60^{o} and the samples were cut into 3 x 3 cm dimension for length and width.

d) Morphology Behavior of Kapok Fibre - PVA Biocomposite

The study on morphology of dried samples were carried out using field electron scanning electron microscopy model LEO SUPRA 55, Carl Zeiss. The sample was cut into small dimension and coated with gold coating to improve electrical conductivity. All the samples images were taken at 15 kV of accelerating voltage with various magnification image of the samples.

e) Thermal Behavior of Kapok Fibre - PVA Biocomposite

The thermal behaviour of kapok fibre was determined using Thermogravimetric Analysis (TGA) using Perkin-Elmer model 4000. About 8-12 mg of kapok fiber was heated at rate of 10 ^{o}C/min in nitrogen atmosphere and temperature for each samples were set from 30 - 700 ^{o}C. Derivative Thermogravimetry (DTG) analysis was carried out in the same way.

RESULTS AND DISCUSSION

1. Sample Selection

The composition of ATKF biocomposite shown in Table 1. Selection of best biocomposite for further characterizations was based on mechanical test results.

Table 1: The composition of ATKF biocomposite.

Sample	1	2	3	4	5
Polyvinyl alcohol (g)	4	4	4	4	4
Kapok (%)	0	10	20	30	40

2. Mechanical Properties

Figure 1 indicate tensile strength and elongation at break results while figure 2 show result of elastic modulus for pure PVA and ATKF biocomposite thin film composite. It reveals that pure PVA shows the highest percentage of elongation to break (48.05%) compared other samples. It was found that the tensile strength of ATKF biocomposite increases as kapok content increased to 30% (17.11 MPa). The highest tensile strength with the value of 17.11 MPa was obtained when 30% kapok is added. The increase of tensile strength probably because of strong interfacial interaction between kapok fiber and matrix which is PVA thus inhibit crack formation. The ability of fibers to restrict matrix mobility also contributed to tensile strength improvement (Zainuddin *et al.*, 2013).

However, the tensile strength of ATKF biocomposite started to decrease when it reaches 40% kapok loading. This observation might be due the possible aggregation of kapok fibre that could reduce tensile strength of the ATKF biocomposite as described by Lu *et al.*, (2008). In other words, kapok cellulose was not dispersed homogeneously in the PVA matrix at 40% content that lead the decline of tensile strength (Cho & Park, 2011). Increasing kapok loading resulted in increased elastic modulus of ATKF biocomposite until reached 30% (177.89 MPa) but declined as kapok loading increase further until 40%. Pure PVA show the lowest elastic modulus with the value 15.24 MPa which supported from previous research by Puttaswamy *et al.*, (2017), also have the lowest elastic modulus results among the samples for pure PVA.

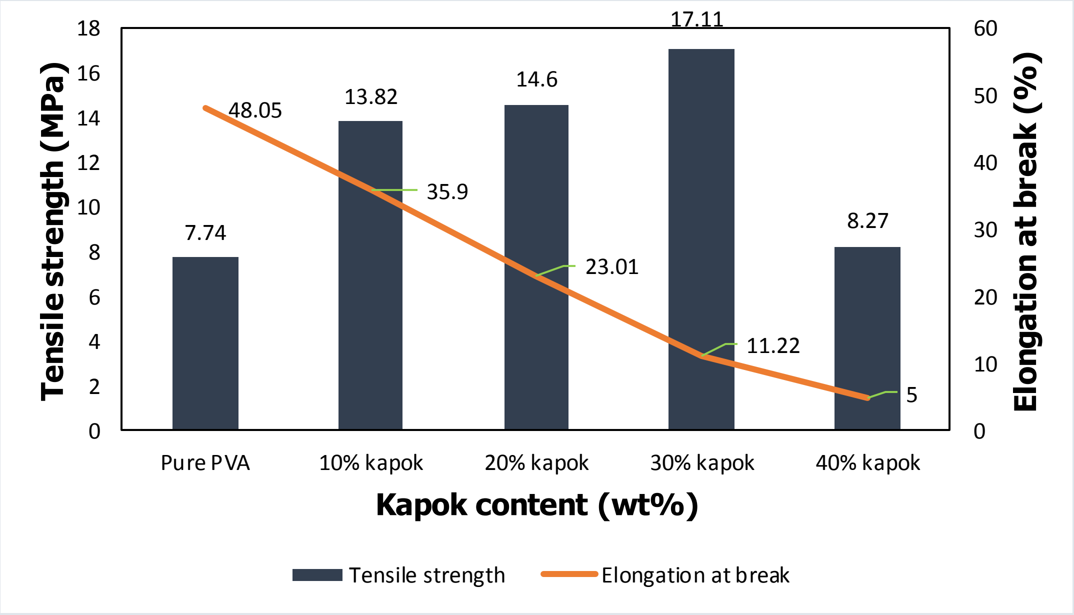

Figure 1: Tensile strength and elongation at break of ATKF biocomposite thin film.

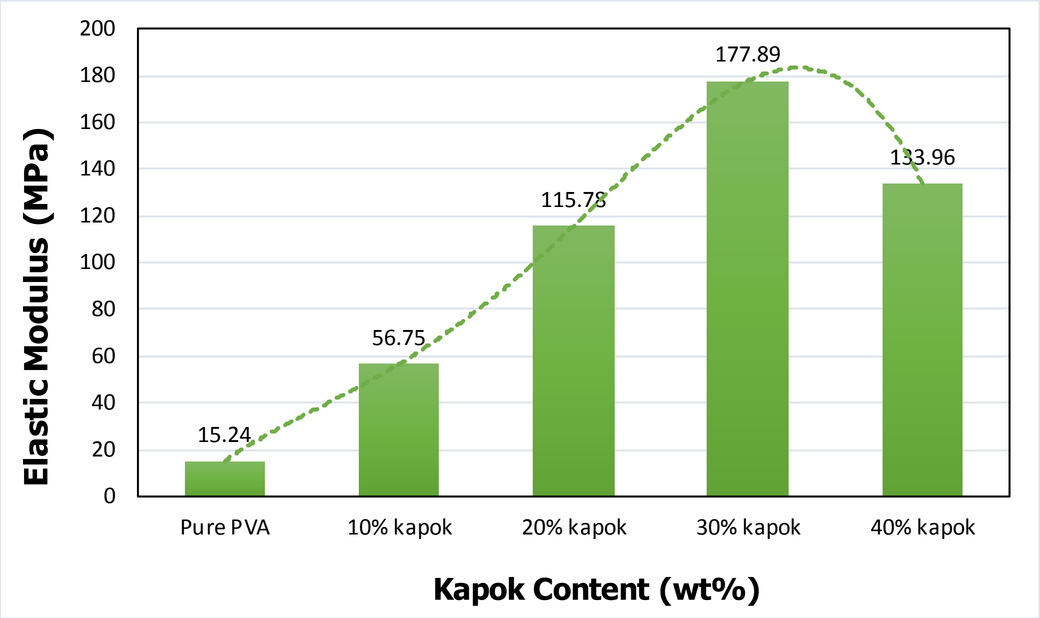

Figure 2: Elastic modulus of ATKF biocomposite thin film.

3. Fourier Transform Infrared (FT-IR) Spectroscopy

FTIR spectra of ATKF, ATKF – PVA biocomposite (30%), pure PVA and raw kapok were shown in figure 3. Strong and broad peak at 3341 cm^{-1} showed hydrogen bonded O – H stretching vibration due to existence of water. The intramolecular hydrogen bonds inside the PVA cause O – H stretching vibration while the formation of this peak are responsible by intermolecular hydrogen bonding between hydroxyl groups of PVA and kapok fiber (Jahan *et al*., 2018). Weak peak at 2900 and 2901 cm^{-1} was assigned C – H stretching vibrations of alkyl group. Absorption band at 1368 cm^{-1} attributed to C – H bending and peak 1737 cm^{-1} reveal the C = O group and esters in lignin and acetyl ester groups in xylan which can be seen at raw kapok spectre (Matuana *et al*., 2001). Theoretically, carbonyl group (C=O) should not exist in pure PVA spectrum at peak 1716 cm^{-1} since PVA chemical formula only consists hydroxyl group (OH). However, there are maybe some remaining of acetate group which contain C=O group exist in the PVA batch since PVA is prepared from PVAc. This statement can be proved by the presence of carbonyl group (C=O) at band 1723 cm^{-1} (Campos *et al*., 2013). The absorption band at 896 cm^{-1} is showing the typical structure β-glycoside linkage at raw kapok spectrum and ATKF wavenumber of C–O–C stretching at peak 1159 cm^{-1}.

ATKF – PVA biocomposite film (30%) bands reveal the interaction between O – H bonding kapok/PVA composite at peak of 1422 cm^{-1} (Prachayawarakorn *et al*., 2013). ATKF band show O-H bending of adsorbed water is observed at peak 1629 cm^{-1}. Even ATKF cellulose undergoes drying process, however the peak of adsorbed water still presence. This is due to the cellulose-water interaction make the water in the cellulose molecules difficult to be extracted (Moran *et al*., 2008). As studies by Tiwari *et al*. (2013), absorption peaks in raw kapok at 1595 cm^{-1} and 1426 cm^{-1} shows the presence of lignin compound corresponding to the aromatic skeletal vibration. Based on the FTIR spectrum of ATKF, hemicellulose and lignin peaks were found to completely vanish. The lignin removal from kapok fibre was encouraged by loss of peaks at 1426 cm^{-1} in ATKF (Bono *et al*., 2009; Draman *et al*., 2013).

The FT-IR composites material interaction between matrix and filler can be identified by band position shifting to a lower wavenumber in the spectra which indicates the interaction of hydrogen bonding between the composites (Wu *et al.*, 2009). From the FT-IR spectre below, the hydrogen bonded hydroxyl peak at 3350 cm^{-1} for raw kapok and 3301 cm^{-1} for pure PVA has shifted to lower wavenumber at 3290 cm^{-1} for ATKF – PVA biocomposite (30%). This results lead to the new hydrogen bond formation between kapok and PVA during composite preparation (Jumaidin *et al.*, 2017). The peak shifting revealed the good compatibility between matrix filler interaction while hydrogen bonding formation in composite materials probably supported with mechanical properties enhancement of the materials (Jumaidin *et al.*, 2017).

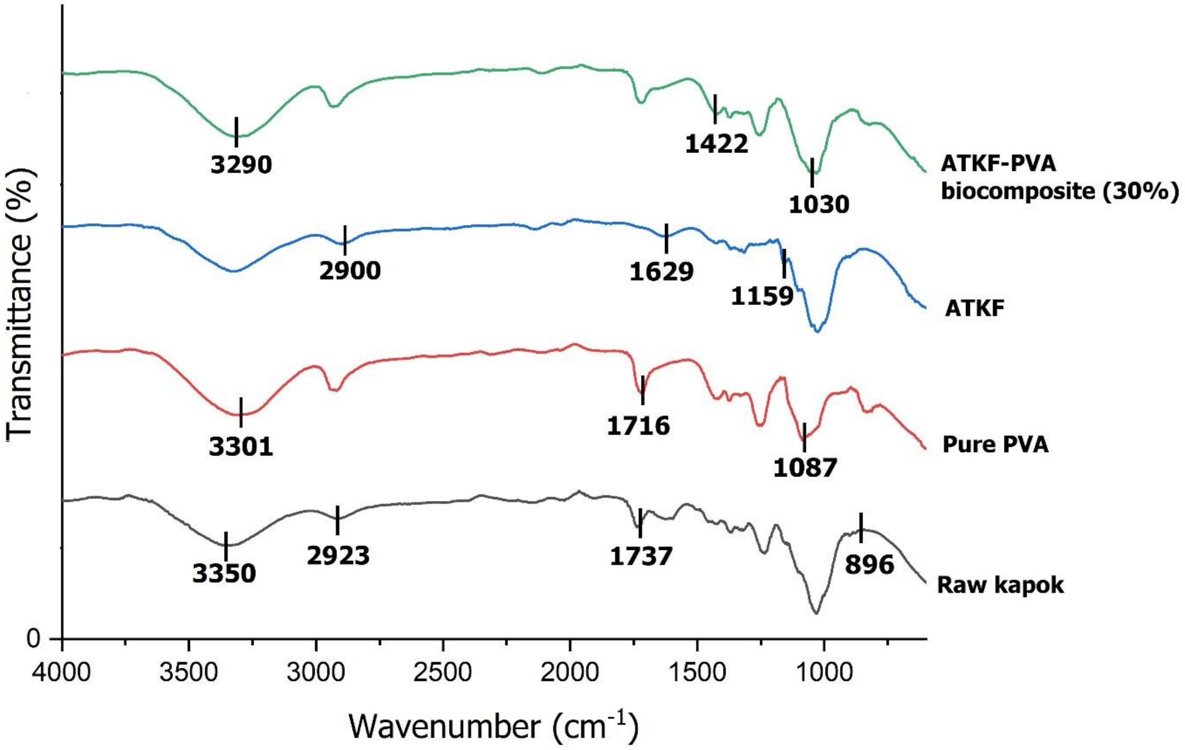

Figure 3: FT-IR spectre of ATKF – PVA biocomposite (30%), ATKF, pure PVA and raw kapok.

4. X – Ray Diffraction (XRD) Analysis

Figure 4 demonstrated x-ray diffractogram of ATKF, ATKF – PVA biocomposite (30%), pure PVA and raw kapok. From the results, pure PVA show typical a peak with high intensity at range $2\theta = 19.89^{o}$ which are same from the previous researches by Hajeeassa *et al.*, (2018) and Jahan *et al.*, (2018). The crystallinity index obtained for pure PVA was 53.45% similar to Jahan *et al.*, (2018) which the crystallinity index for their PVA membrane around 53%. On the other hand, it can be interpreted PVA reveal a semicrystalline structure because of the high affinity to form hydrogen bond (Hajeeassa *et al.*, 2018). Meanwhile, raw kapok shows small peak at 17.37^{o} and broad peak at $2\theta = 23^{o}$. The crystallinity index (CI) of raw kapok fibre was 32% which is slightly lower as reported by Purnawati *et al.*, (2018**)** which the kapok fibre crystallinity index was 35.65%. The peak 17.37^{o} represents the presence amorphous parts of kapok such as lignin, hemicellulose, wax and other impurities (Ma *et al.*, 2015).

When raw kapok fibre was treated with 17.5 NaOH solution, there is change occur in the result. The peak intensity incline compares to raw kapok due to the removal of lignin and hemicellulose. The sharp peak at region $2\theta = 22.1^{o}$ and weak peak show in region $2\theta = 16.8$ and 12.3^{o} in diffractrogram represent typical crystalline cellulose peak (Bondsen *et al.*, 2006). The crystallinity index of ATKF is expected to be increased up to 64% which also similar result reported by Liu & Wang (2009) where the dewaxed fiber crystallinity index was 63%. On the other hand, the diffractogram pattern of ATKF -PVA composite (30%) indicated 2 overlap peak at region 20° and 23°. No new peak produced from the ATKF -PVA composite (30%). It can be concluded that ATKF -PVA composite (30%) is a heterogeneous composite phase.

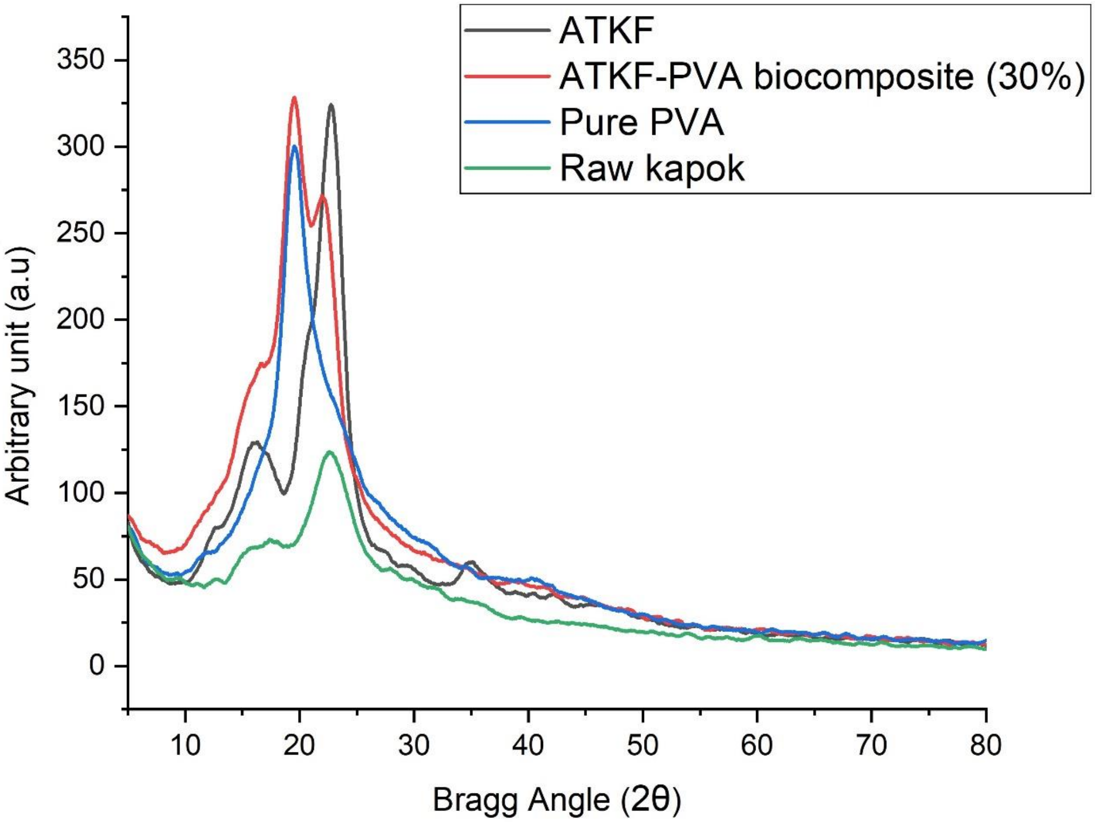

Figure 4: XRD diffractogram of ATKF, ATKF – PVA biocomposite (30%), pure PVA and raw kapok.

5. Scanning Electron Microscopy (SEM)

Scanning electron micrographs of raw kapok fibres, cross section of kapok fibre, ATKF, pure PVA, surface of ATKF – PVA biocomposite (30%) and cross section ATKF – PVA biocomposite (30%). A silky appearance, smooth surface (Draman *et al.* 2013) and tangled like structure can be seen in raw kapok fiber as shown in Figure 5a. The average diameter for raw kapok is 28.7 µm which slightly larger than reported by Purnawati *et al.*, (2018) in the range 17.59 µm. Meanwhile, kapok fibre cross section on Figure 5b shows a large lumen with hollow tube shape of and thin fibre wall of kapok fibre cross section. The cell wall thickness of kapok fiber obtained an average of 1.16 µm which similar to Purnawati *et al.*, (2018) meanwhile the average lumen diameter obtained was 14.51 µm which larger from a previous finding by Mwaikambo and Bisanda, (1999) and Kang *et al.*, (2007).

ATKF in Figure 5c indicate the kapok fiber look a rougher than raw kapok fiber and also the structure turned out to be totally flattened with strip like shape (Draman *et al.* 2013). ATKF image also show the different length from raw kapok fibers which the length was drastically reduced after chemical treatment (Sonia & Dasan, 2013). The average width estimated is around 19.5 µm which is in the range as reported by Draman *et al.*, (2013) were 18.5 to 23.1 µm the width obtained from kapok treated with NaOH. As for pure PVA image (Figure 5d), the plain image shows as expected which alike from previous research from El-Kheir *et al.*, (2015). The situation in Figure 5e was a difference where the ATKF and PVA matrix form a uniform layer composite. It can be seen the ATKF merge into PVA matrix and form a good adhesion interaction. However, data from XRD reveal that the layer formed was heterogenous. The alkali treatment attests that the hydrogen bonding interaction between PVA and ATKF was improved as indicated in FTIR analysis.

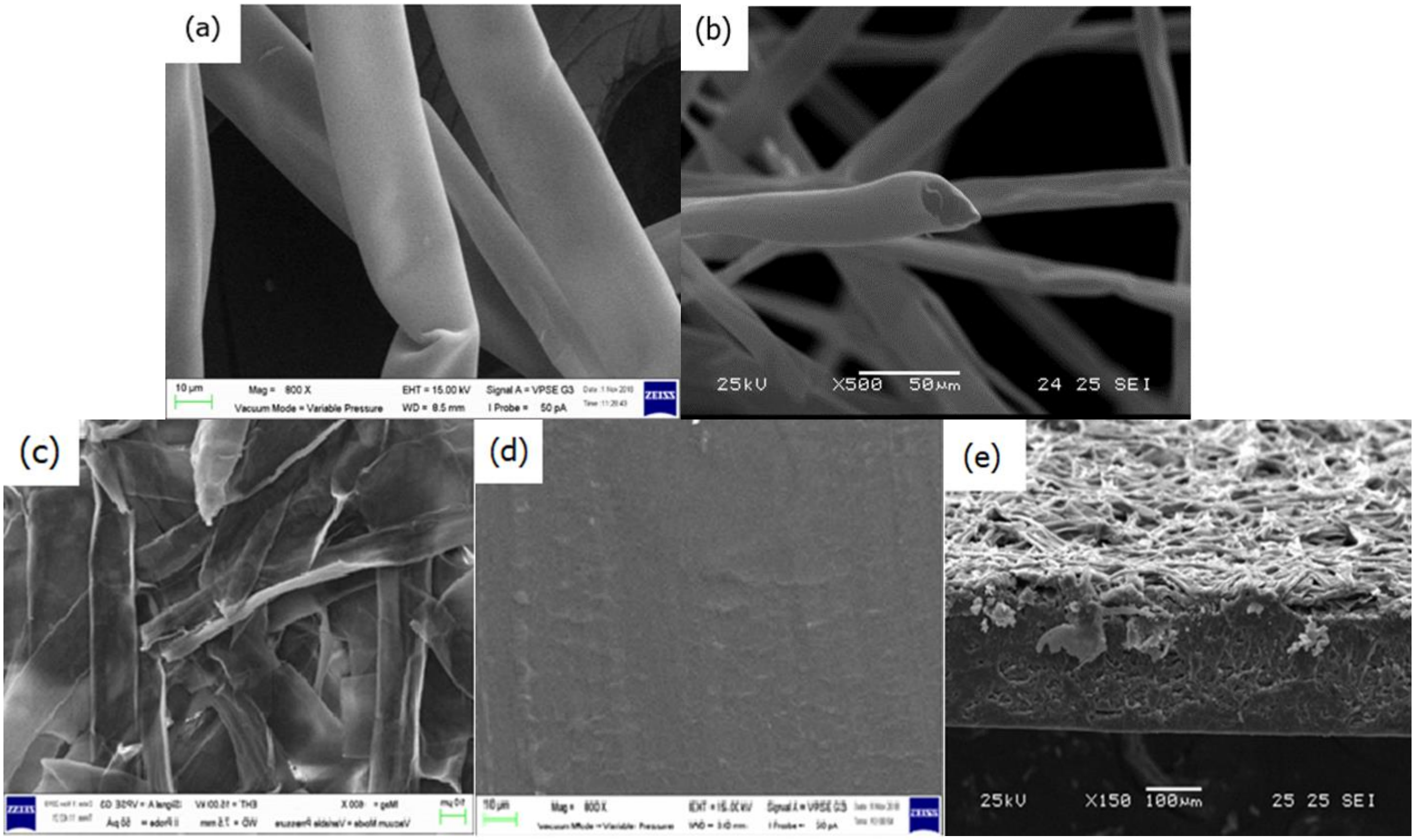

Figure 5: SEM images of (a) raw kapok fibre; (b) cross section of kapok fibre; (c) ATKF; (d) pure PVA; (e) cross section ATKF – PVA biocomposite (30%).

6. Thermogravimetric Analysis

Figure 6 and 7 below shows the thermograms of TGA and DTG respectively. The starting degradation temperature was observed to be 350 °C, 359 °C, 394 °C and 360 °C for raw kapok, pure PVA, ATKF and ATKF – PVA biocomposite film (30%) respectively. The initial weight loss for all samples begins from 50 to 150 °C due to removal of moisture content in the samples by evaporation

Raw kapok exhibits two major degradation stages. The raw kapok degradation temperature started at 350 ^{o}C. At this stage, polysaccharides, i.e. cellulose and hemicelluloses were started to be degraded (Draman *et al.*, 2014) and proceed until it reaches the completion decomposition temperature at 550 ^{o}C (Brebu and Vasile, 2010). Meanwhile, for ATKF, the moisture evaporation occurs at 56 ^{o}C and decomposition temperature started at 352 ^{o}C. During this stage, cellulose in ATKF was almost burnt because of oxidative thermal degradation and crystallite structure deterioration (Yeng *et al.*, 2015).

Pure PVA also have 2 major degradation stages. The first weight loss occurs at temperature range 103 – 106 °C due to removal of moisture in the samples by evaporation which causes 5% mass loss from the original weight. Kim *et. al.*, (2008) published that the cause of this first weight loss is related to the evaporation of both water and also glycerol. As the temperature increased, the decomposition of PVA started at 360 to 370 ^{o}C. At this stage, the hydroxyl and methyhydroxyl groups were stated to be eliminated. The degradation continues until it reaches the completion decomposition at 480 – 500 ^{o}C where the polymer C – C backbone has been destroyed completely (Ahad *et al.*, 2012).

For ATKF – PVA biocomposite film (30%), it showed an expected result which 2 steps of mass loss. The first weight loss was definitely due to evaporation of moisture content in the sample that occur at ranges of 90 –110 ^{o}C. The drastic mass loss that start at 360 ^{o}C might be due to the disintegration of intra- and intermolecular of hydrogen bonds between PVA and cellulose (El-Kheir *et al*, 2017). The completion decomposition at 500 ^{o}C has indicated that PVA and cellulose main chain have been completely degraded (Othman *et al.*, 2011).

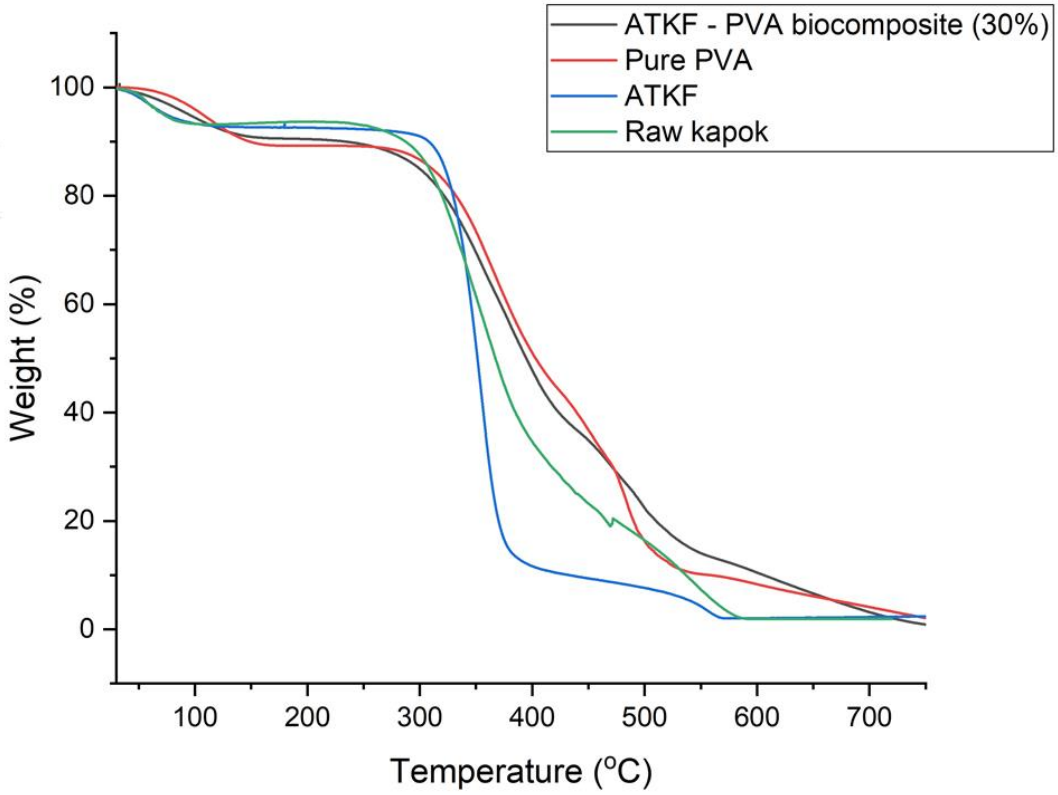

Figure 6: The TGA thermograms obtained from ATKF – PVA biocomposite film (30%), pure PVA, ATKF and raw kapok.

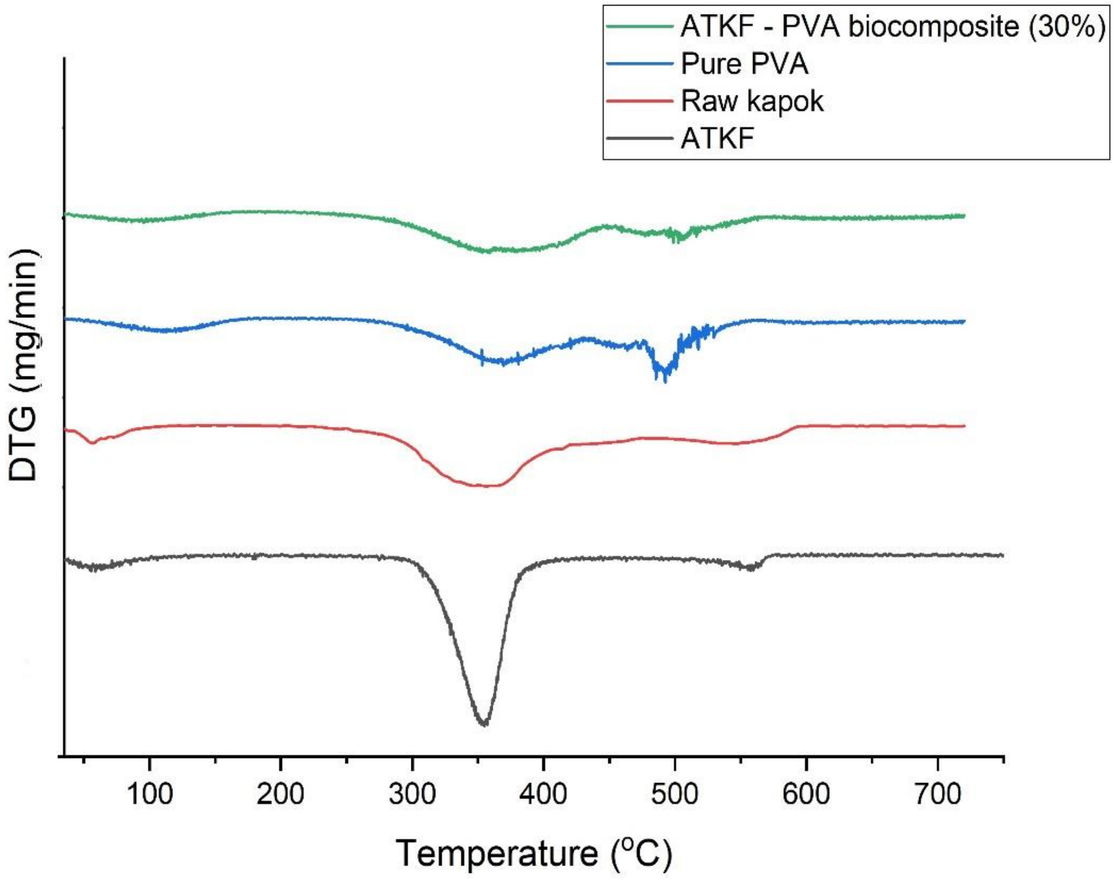

Figure 7: DTG thermogram of ATKF – PVA biocomposite film (30%), pure PVA, ATKF and raw kapok.

CONCLUSION

This study has shown the improvement in tensile strength of ATKF – PVA bicomposite until it reaches 30% kapok loading and decrease when it reaches 40% kapok loading. FTIR spectra indicates interaction of ATKF with PVA matrix produce new hydrogen interaction which helps improve the mechanical properties. As for XRD analysis, the result reveals ATKF – PVA biocomposite film (30%) was heterogeneous which in diffractogram, the peaks of kapok/PVA (30%) overlap each other that supposedly new peak formed for the composite. SEM micrograph shows ATKF – PVA biocomposite (30%) film form a heterogeneous layer. However, ATKF – PVA bicomposite (30%) interaction is more smooth and uniform which give advantage in mechanical properties. From the TGA data, ATKF – PVA bicomposite (30%) was founded less thermally stable than raw kapok and pure PVA is the least thermally stable among other samples.

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