Pineapple Leaf Fibers (PALF)/ Polyethylene Terephthalate (PET) Electrospun Nanofibers: Effect of Ratio on Chemical & Morphological Properties

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Abstract: Nanofibers capabilities in produced small materials up to nanoscale dimension, making them the perfect fundamental materials that can help improving the effectiveness of many applications. In this study, the properties of PALF/PET electrospun mats were studied. Different ratio of PALF/PET were electrospin to determine the optimum parameters for fabricating electrospun PALF/PET with minimum defect. The sample obtained were then characterized for its morphology and chemical properties. FTIR studies were done to understand the interaction occurred between PALF and PET with increasing PALF ratio. From the FTIR result, increasing PALF showed similarities spectra with raw PALF peak at 1100cm^{-1} indicating the influence of PALF in the fibers. This peak did not appear at low PALF content. The obtaining electrospun mats were observed under FESEM to characterize their morphological properties. Increasing in PALF ratio also attributes to the decreasing of size diameter and diameter range. From the result, PALF$_{PET4}$ shows the smoother morphology with linear properties and smaller average size diameter.

Index Terms: Chemical Properties, Electrospinning, Morphology Properties, Natural Fiber, PALF, PET

I. INTRODUCTION

Natural fibers that come with biodegradable, renewable and friendly environment properties have become promising materials for synthetic fibers substitution [1]. Numerous research has been done to explore natural fibers potential such as the study on ramie, jute fiber sisal fiber and pineapple plant for commercial purposed [2].

Among them, pineapple leaf which comes from the secondary part of plant shows remarkable mechanical properties and easy processing [3]. Pineapple Leaf Fiber (PALF) which contain high cellulose and low lignin content was suitable for producing good properties of nanofibers [4]. Besides, PALF with high cellulose content made it finer than other natural fibers thus made it easier for processing. Seeing it promising opportunities in the future, researchers who studied natural fibers end up proceed for electrospinning the natural fibers. However, up to now, PALF was not yet electrospun and study on its nanofibers size.

Thus, this study focused on producing nanofibers from pineapple leaf via electrospinning method. However, the low molecular weight of natural fibers has become the main drawback for electrospinning process [5]. Combining PALF with other synthetic polymers were expected to enhance its physical and mechanical properties [6].

Nanofibers are one of the sub-study of nanotechnology that widely regarded as great potential applications. Featuring thin web-like structure mats with high surface area per volume, high porosity nanofibers successfully penetrate through the large industrial market. Remarkable interconnectivity between its pores useful in small scale application such as filtration, tissue engineering and drug delivery [7]. [24] in his paper discussed the properties of sisal/PET electrospun and proposed that a smaller size of nanofibers was attributed to the natural fiber due to its low dielectric constant. The dielectric constant is one of the parameters that affect the nanofibers formation due to the solubility of the solvent and the sample [25].

Various established nanofibers technique has been developed such as self-assembly, drawing, phase separation, and template synthesis, however, these techniques usually restricted for laboratory scale only. In 1990, first electrospinning research was patent. Although in the beginning, the electrospinning process only occurs in the laboratory, obtaining optimum parameters for this process make it potential for industrial processing [8].

Known as a simple and adaptable method with less supervised for polymer solutions processing, electrospinning present materials with desired diameters ranging micrometers narrowed to nanometers size [9]. As reported in previous research, electrospinning consists of four main parts which are power supply, syringe pump (syringe), needle and metallic collector [10]. Power supply provided enough voltage to move the electric charge into the solution through the metallic needle. High charge in solution causes solution instability thus force the solution to move to different charge emitted from the metallic collector.

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As the voltage increased, the solution was forced to emit from the metallic needle thus form spherical droplet at the tip of the needle known as Taylor Cone. The surface tension of the solution restricts direct solution dispersion and holds the surface of the solution. At one point after Taylor cone shape was stabilized, ultrafine thread like nanofibers was pulled from the solution and collected at the metallic collector [10].

During the ejection, the solvent will be evaporated to the surrounding left behind pure fibers. During the process, the forces from surrounding cause particular motion on the fiber called whipping motion that move along to the collector. This whipping motion triggered the vibration of the polymer chain solution allows it to moving forward and backward slide past each other which resulting in the formation of fibers with diameters small enough to be called nanofibers [10]. However, studies conducted by [11] stated that the elimination of the whipping motion decrease 50% of the average diameters of PEO fibers.

Water and air pollution have caused great concerns due to the waste produced by industries especially in the chemical industry. This has urged the researcher to find solutions to reduce the risk. Fibers with small diameter show the best filtration capacity [12] Small diameter with compact threads like structure was stated help in improving the forward osmosis process thus enhance effectiveness on filtration wastewater treatment [13]. This is supported by [14] in his study that stated electrospun nanofiber mat of PCL/PEO were successfully increase efficiency in water filtration up to 80.01%.

Research by Cui et al. (2015) improves the use of PP as oil trapper using PVC/PS. By implying absorbant technique, improvement in the smaller fibers diameter, high surface area, high porosity and increasing void numbers of electrospun mats has provided large amounts of storage volume for sorbed oils. Nevertheless, high porosity electrospun mats also help to filter smaller materials such as Pb as suggested by [16].

In this study, electrospun mats of PALF/PET nanofibers were fabricated via electrospinning method using TFA: DCM as the solvent. Optimum PALF/PET ratio was studied to produce electrospun nanofibers with excellent chemical and wetting properties that have the potential for water filtration application.

II. METHOD

A. *Materials*

Local Pineapple Leaf fibers (PALF) were supplied from Bumipadu Solution (Local Malaysian Company) in fibers form. Polyethylene Terephthalate which was received in granular form was supplied from Aldrich. Chemical used in this study which Trifluoroacetic acid (TFA), Dichloromethane (DCM), cyclohexane and ethanol all provided by Aldrich Merck.

B. *Soxhlet method (Dewaxing)*

PALF undergo dewaxing using the soxhlet method to separated unwanted content (wax and terpenes) from the PALF hence provide higher surface area for chemical interaction. 10 g of PALF were put into the thimble and rinse repeatedly with 200ml of ethanol:cyclohexane with 1:1 ratio for 6 hours. The trapped evaporated solution were recycled until the brownish PALF colour decolorized indicating removal of wax, terpenes and a small percentage of hemicellulose and lignin. Then, the remaining sample in the thimble were taken out and rinse using distilled water. The sample were let dry in the oven with a temperature 40°C for 12 hours.

C. *Dissolution of PALF and PET*

Dried PALF and PET were weight accordingly to table 1. Five samples with different ratio of PALF/PET were made to find out the best ratio for the electrospinning process. Pure PET act as the constant sample used for sample comparison. All samples were diluted in 70:30 ratios of Trifluoroacetic acid (TFA) and DCM (Dichloromethane) solvent as tabulated in table 1 to obtained desired concentration and stirred for 6 hours until homogenous solution were obtained.

Sample	PALF (g)	PET (g)	Solvent (TFA/DCM)/(70:30)	PALF/PET Percentage Ratio
PurePET (PET)	-	0.45	5 mL	0 : 100
Ratio1/10 ($PALF_{PET1}$)	0.06	0.60	5 mL	10 : 90
Ratio1/7.5 ($PALF_{PET2}$)	0.06	0.45	5 mL	12 : 88
Ratio1/5 ($PALF_{PET3}$)	0.06	0.30	5 mL	17 : 83
Ratio1/1 ($PALF_{PET4}$)	0.10	0.10	6.5 mL	50 : 50
Ratio1.3/1 ($PALF_{PET5}$)	0.20	0.15	6.5 mL	57 : 43

Table 1: Weight of PALF and PET for different ratio

D. *Characterization*

I. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis was carried out to determine the interaction that may occur between PALF and PET and also to confirm that PALF and PET were electrospun together. The interaction change can be observed through the change in the band intensity, any frequency shifts and also change in the shape of the FTIR spectra. The result was recorded using Attenuated Total Reflectance (ATR) of FTIR (Thermo Fisher Scientific Nicolet iS 10) with frequency range 500-4000 cm^{-1} and 2 cm^{-1} resolutions. The spectra were studied in transmittance using origin and omnic software.

II. Field Emission Scanning Electron Microscopy (FESEM)

Electrospun mat samples were observed under FESEM (SUPRA 40VP 31-31) at accelerating volt of 5.0 kV. Two magnification were captured at 5 K and 20 K to observe the nanofibers diameter and surface morphology. All samples were spattered with a thin layer of gold to provide conductive surface and reduce charging. The diameters were calculated using ImageJ software.

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III. RESULTS & DISCUSSIONS

A. *Dissolution of PET/PALF solution*

Fig 1 (a) and (b) shows PALF/PET solution with highest PET ratio and highest PALF ratio respectively. High PET ratio shows light brown colours of solution however started to turn to dark brown with increasing of PALF ratio. This is suggested due to the presence of lignin that contributes to brown colour of PALF [6]. High PALF ratio was easily lead to a high concentration solution hence producing small amount of solution as it will easily be evaporated to the surrounding [5].

Electrospun sample for each of the solution also shows significant different as shown in fig 1 (c) and (d). Sample with high PET ratio producing deep white surface nanofibers electrospun mats compared to high PALF ratio that showed clear white surface after peeling off. Besides, increasing ratio of PALF also lead to smoother surface compared to high PET ratio that is more brittle during peel off.

This sample were expected to change properties from hydrophobic to more hydrophilic nanofibers due to the high O-H bonding of PALF. Generally, hydrophilic sample owing clear surface to allowed substances to move easily through it [17]. Besides, increasing in PALF ratio attributed to smaller size of electrospun sample as can be seen in FESEM result.

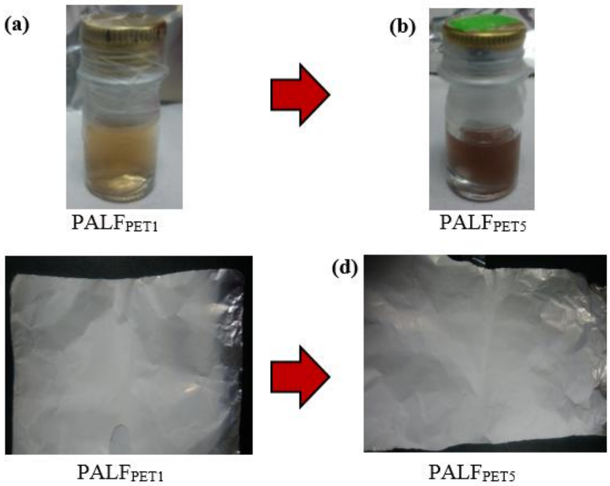
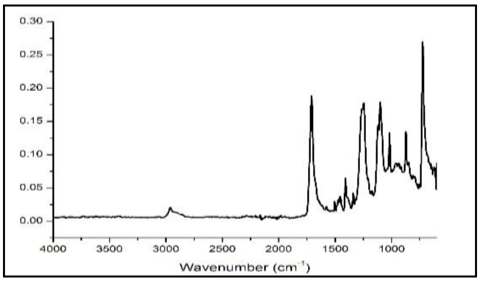


Fig 1: (a) (b) solution of PET/PALF (c) (d) Electrospun sample of PET/PALF

B. *Fourier Transform Infrared Spectroscopy (FTIR)*

The interactions occurred between PET and PALF with different ratio were determined using FTIR analyses. Fig 2 displays the FTIR graph obtained for PET granule and raw PALF.

(a)



(b)

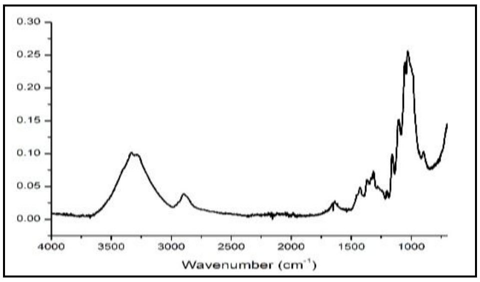


Fig 2: FTIR spectra of (a) PET granule (b) Raw PALF

PET granule as in fig 2(a) is known for its hydrophobic properties hence shows first peak at spectra 2900 cm^{-1} and followed by several significant peak at 1730, 1714cm^{-1}, 1410cm^{-1}, 1250cm^{-1}, 1125cm^{-1} and 722cm^{-1}. Different with PALF in fig 2(b), broad peak was observed at 3400 cm^{-1} indicating the present of big amount of O-H bonding hence lead to hydrophilic properties materials [3]. Sharp and broad peak were observed at 1100 cm^{-1} spectra indicating high amount of cellulose [18].

FTIR analysis was carried out in order to determine the interaction that may occurred between PALF and PET and also to confirm that PALF were electrospun together with PET. Fig 3 shows the FTIR result obtain from pure PET electrospun with different PET/PALF ratio electrospun.

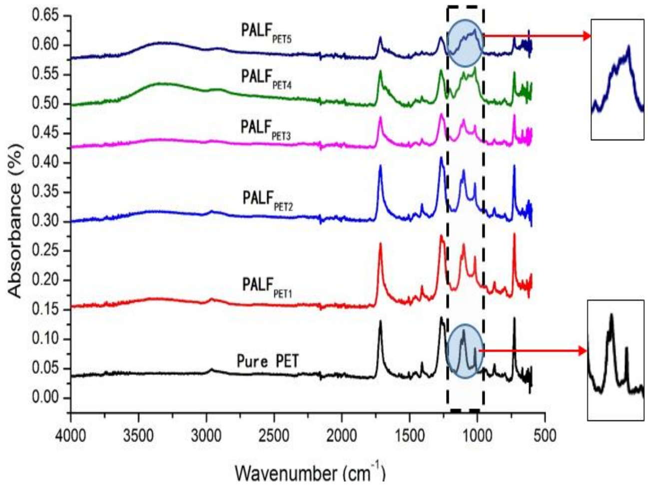


Fig 3: FTIR spectra for different Pure PET and PALF/PET electrospun mats

As can be seen at approximately 3400 cm-1, peak was observed for PET sample with PALF and the peak become broader with increasing of PALF content. Peak at 3400 cm^{-1} represent the wetting properties of the samples. Increasing in PALF ratio produce broader peak that can be seen clearly at ratio 1/1 and 1.3/1 where PALF ratio is higher than PET. Thus, this result shows the addition of PALF will help to change the hydrophobic properties of PET becoming more hydrophilic. A few significant peaks were detected at 1714cm^{-1}, 1410cm^{-1}, 1240cm^{-1} and 722$cm^{-1.}$ All this peak that mostly attributes from PET structure were reduced with increasing of PALF ratio. Small peak indicated present of terephthalic acid was spotted at region 1410 with decreasing intensity after PALF ratio increase [3].

When PALF ratio was precede PET, new peaks representing PALF were detected around 1000cm^{-1} to 1250cm^{-1} indicating that the PALF has been successfully incorporated into the PET. This peak was belonging to the cellulose [19] and become more pronounced upon increasing amount of PALF indicating more PALF cellulose were electrospun together. PALF$_{PET5}$ followed peak similarity for raw PALF peak compared to PALF$_{PET1}$ that consist peak similar to PET granule.

Table II shows the characteristics of FTIR peaks for electrospun PET/PALF.

Table II: The characteristics FTIR peaks for electrospun PET/PALF

Wavenumber (cm^{-1})	Functional Group	References
3450	Hydroxyl group with O-H bonding	Neto et al., 2013 Roque et al., 2016
2900	C-H axial deformation (PET/PALF)	Santos et al., 2015 Neto et al., 2013
1630-1730	C=O present in conjugated ester group/hemicellulose (PET/PALF)	Santos et al., 2015 Neto et al., 2013
1410	C-O-H carboxylic group presence of terephthalic acid (PET)	Santos et al., 2015
1240/1092	C-C-O and O-C-C stretching (PET)	Strain et al., 2015
1170-1150/ 1050-1030	C-O stretching bond present in either cellulose and hemicellulose	Neto et al., 2015 Roque et al., 2016
722	C-H wagging vibration (PET)	Strain et al., 2015

C. *Morphology Properties*

Electrospun samples of PALF/PET were then observed under FESEM to understand more on its surface morphology and the fiber size. Fig 4 (a) and (b) shows the FESEM image of Pure PALF electrospun, fig 5 (a-f) shows the average diameter and FESEM mag: 5K for Pure PET and PALF$_{PET\ 1\text{-}5}$ respectively and for fig 6 higher magnification result with mag: 20K were showed.

Based on the FESEM micrographs, the present of PALF improved the fiber forming potency. Fig 4 (a) and (b) shows the morphology of Pure PALF electrospun mats. Rather than producing nanofibers, an incomplete droplet cell formed-like was deposited due to the solution was too dilute to form continuous jets and the the jet broke up into droplets [20].

Natural fibers were known for its low density hence provide with low molecular weight. Generally, low molecular weight lead to low chain entanglement for the polymer. During the electrospinning, the fiber jet from the needle will stretch into thin fibers and only hold by the chain entanglement. Low chain entanglement will cause break of jet fibers thus produce agglomerate fibers at the collector [26]

Low molecular weight with different chemical structures of different fibers has become the main drawback for lignocellulosic fibers processing [21]. Thus, electrospun raw PALF were not taken for further characterization. Similar result was found by [22] for high ratio of chitosan.

FESEM results as shown in figure 5 indicated that the present of increasing ratio PALF in sample PALF$_{PET4\text{-}5}$ led to a tendency of lower average fiber diameter however with a random diameter distribution. Two different fiber networks with intersecting fibers were observed in electrospun PALF/PET. It is suggested that one networks with bigger diameter probably corresponds to PET and the others with smaller diameter correspond to PALF.

(a)

(b)

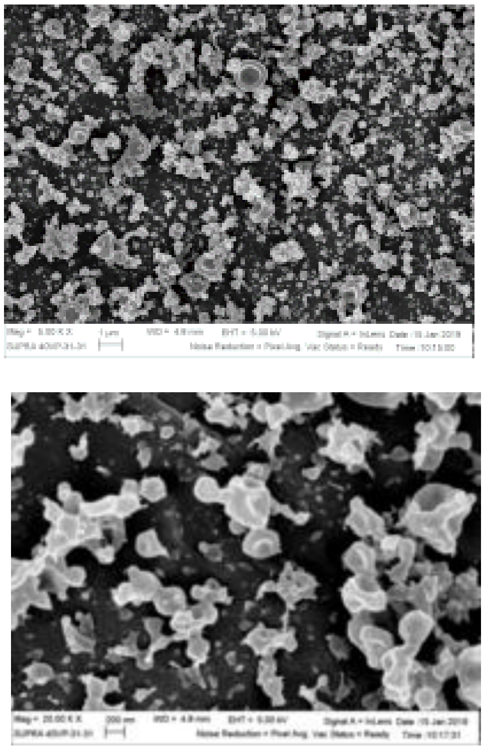


Fig 4: FESEM morphology of Electrospun Pure PALF (a) 5K (b) 20 K

Initially, the first two samples with lower PALF which is sample PALF $_{PET1\text{-}2}$ shows thicker average diameter. This was probably due to the different solubility and the dielectric constant of the PET and PALF [23]. Increasing in PALF ratio (PALF $_{PET3\text{-}5}$) had narrowed the fiber diameter range thus decrease the average diameter at once.

Sample PALF$_{PET5}$ shows the lowest average diameter with an average size of 67.6nm ± 10 approximately however lead to uneven and beaded surface. A thin fiber has coexisted from single fiber indicated that new fibers were ejected however the bond were collapsed during ejection thus did not produce a complete single fiber. This is probably due to the difference in dielectric value [23]. Similar result was observed by [5] which is for electrospun of PCL and chitosan.

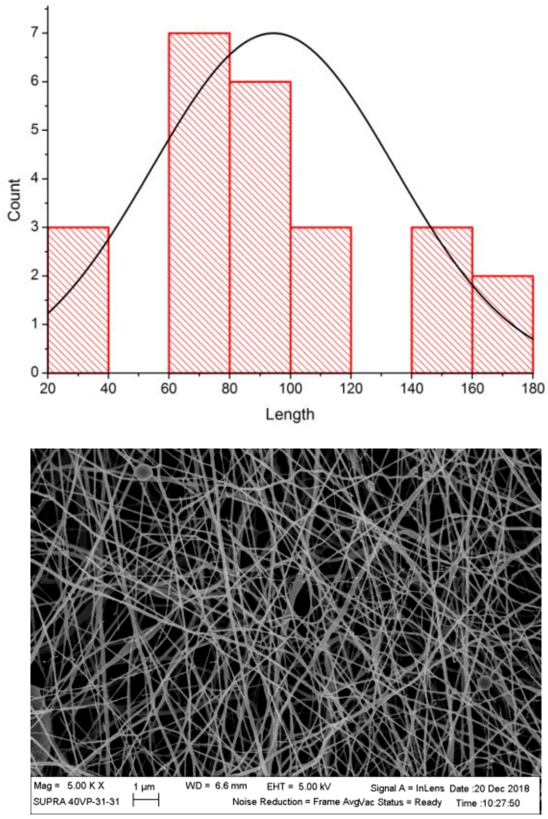


Fig 5 (a) Diameter range and FESEM mag 5 K for Pure PET

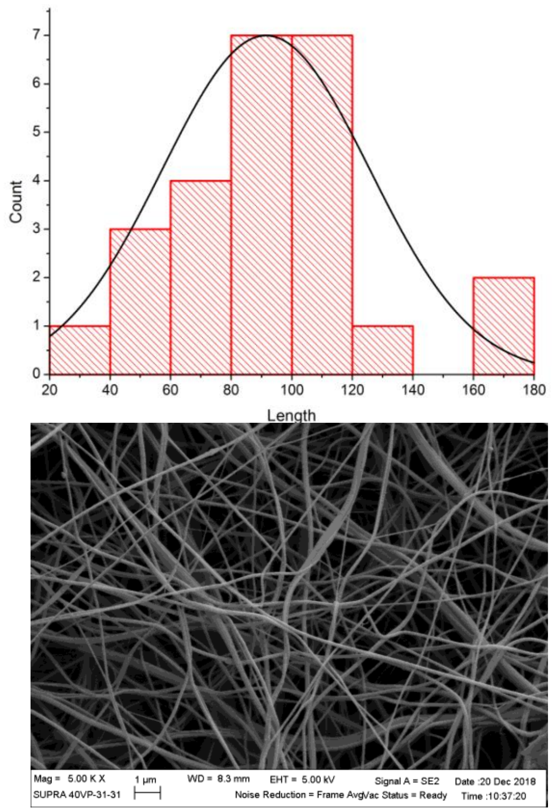


Fig 5 (b) Diameter range and FESEM mag 5 K for $PALF_{PET1}$

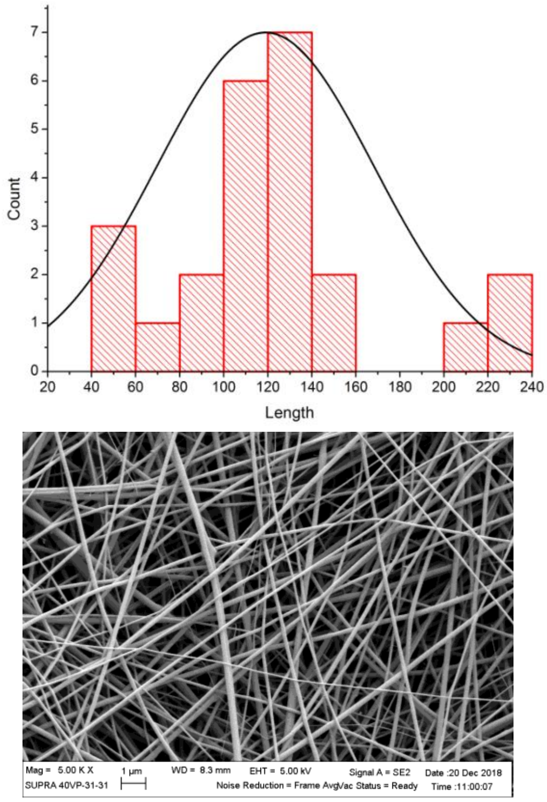


Fig 5 (c) Diameter range and FESEM mag 5 K for $PALF_{PET2}$

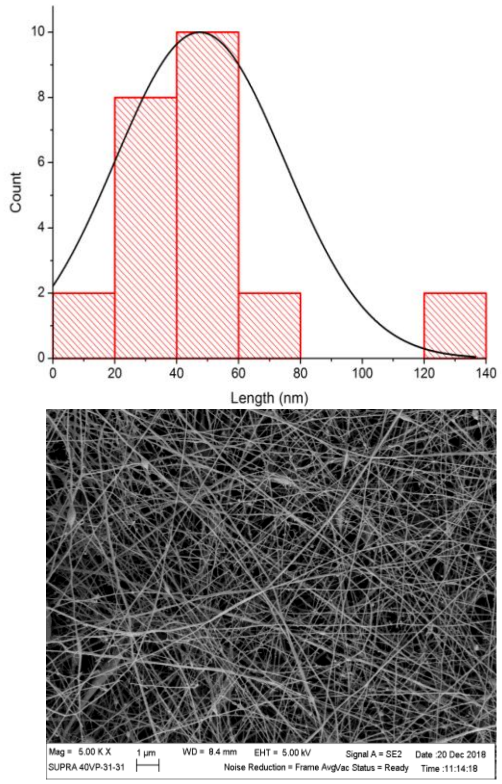


Fig 5 (d) Diameter range and FESEM mag 5 K for $PALF_{PET3}$

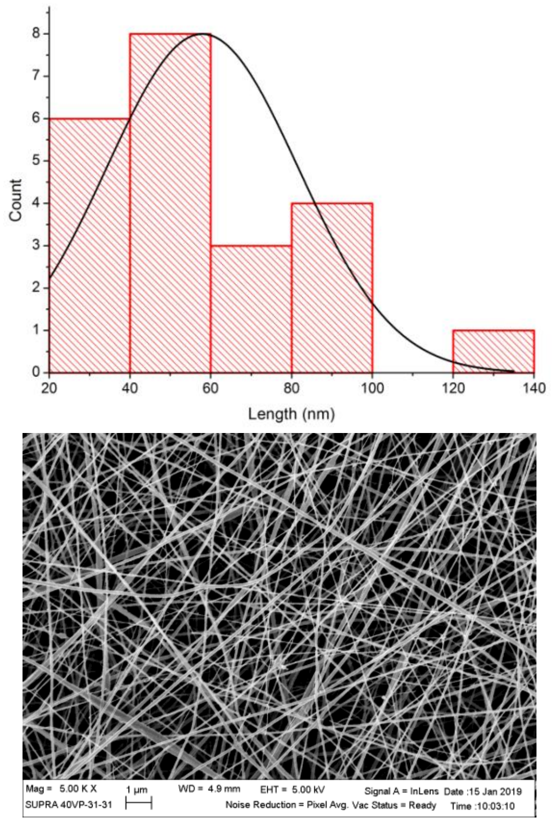


Fig 5 (e) Diameter range and FESEM mag 5 K for $PALF_{PET4}$

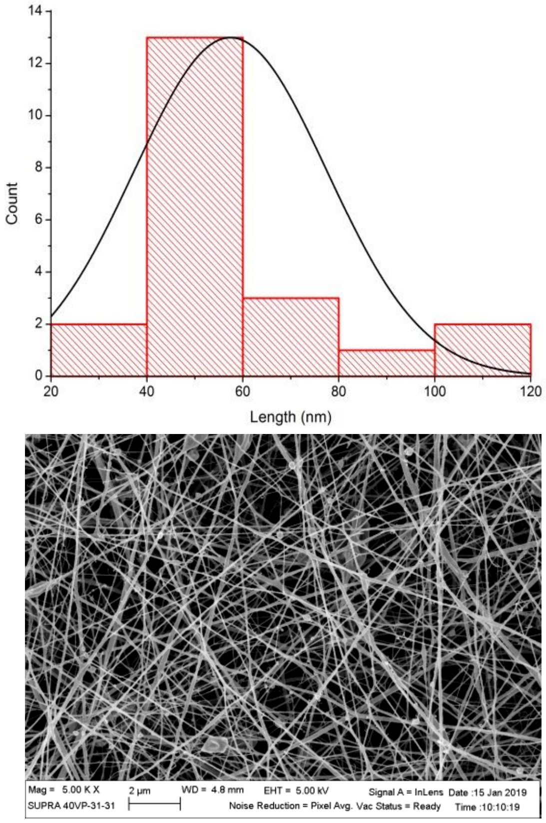


Fig 5 (f) Diameter range and FESEM mag 5 K for $PALF_{PET5}$

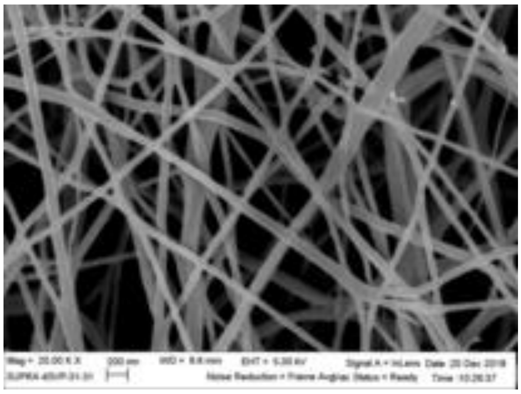


Fig 6 (a) FESEM Mag 20 K Pure PET

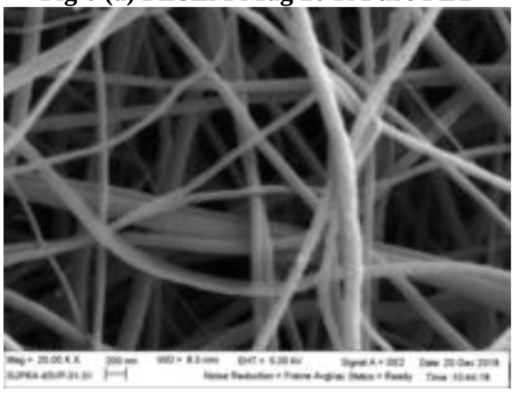


Fig 6 (b) FESEM Mag 20 K $PALF_{PET1}$

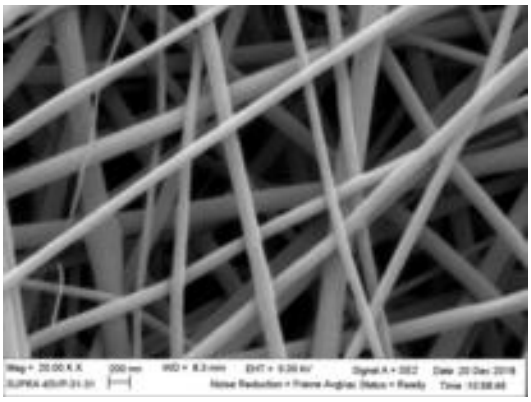


Fig 6 (c) FESEM Mag 20 K $PALF_{PET2}$

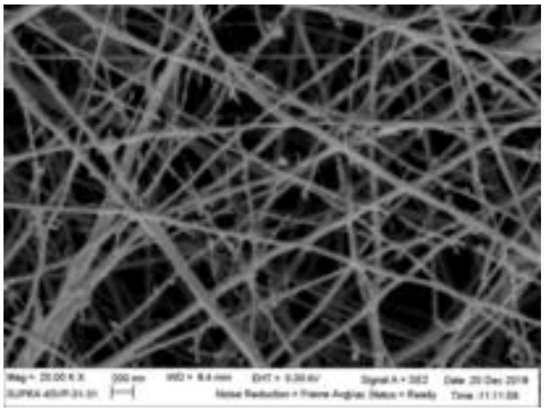


Fig 6 (d) FESEM Mag 20 K $PALF_{PET3}$

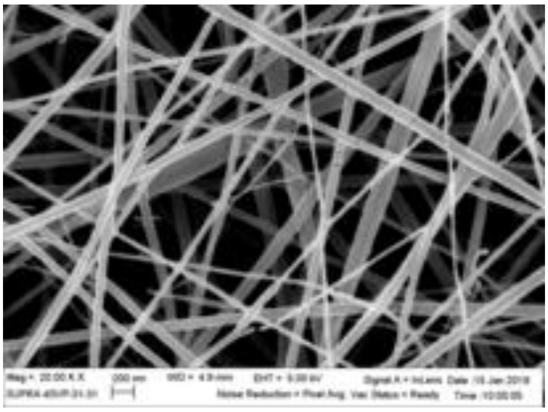


Fig 6 (e) FESEM Mag 20 K $PALF_{PET4}$

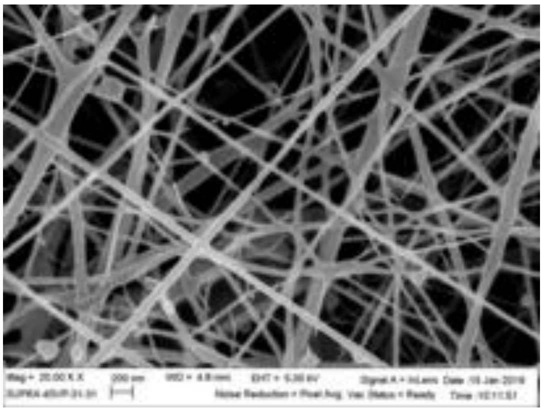


Fig 6 (f) FESEM Mag 20 K $PALF_{PET5}$

From the result, $PALF_{PET4}$ shows smaller average diameter with small diameter range and smooth surface morphology compared to others as shown in fig 6. From this result, it can be seen most of the sample shows bending and nonlinear fibers compared to $PALF_{PET4.}$ Thus, it can be proposed that $PALF_{PET4}$ with 1:1 ratio attributed to the smoother with smaller size of nanofibers.

Table III shows the average diameter measured for Pure PET and PALF/PET electrospun. $PALF_{PET5}$ shows the smaller diameter with small diameter range. On the other hand, small PALF ratio increase the average diameter however decrease as much as 108% with further increasing of PALF ratio. The diameter recorded smaller than pure PET. Hence, it can be proposed that high PALF ratio lead to smaller nanofibers diameter.

Table III: Diameter and Size distribution of electrospun nanofibers

Sample	Average Diameter	Diameter Range
Pure PET	96.379 nm ± 34	20 ~ 180
$PALF_{PET1}$	93.214 nm ± 28	20 ~ 180
$PALF_{PET2}$	120.915 nm ± 41	20 ~ 240
$PALF_{PET3}$	46.263 nm ± 16	0 ~ 140
$PALF_{PET4}$	57.31 nm ± 17	20 ~ 140
$PALF_{PET5}$	43.629 nm ± 10	20 ~120

IV. RECOMMENDATION

The findings of this study can become an establishment for future research on electrospun mat PALF nanofibers which has high chances for various applications especially in filtration. As nowadays, industry encountered problems with the non- renewable resources materials, this finding can help to produce natural fibers that have the opportunity to replace current synthetic materials. Increasing of PALF ratio in blend with PET is possible to be considered for further studies in water filtration application.

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