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SUSTAINABLE PAPER FROM AGRICULTURAL WASTE: A STUDY ON PINEAPPLE LEAF FIBRE USING ORGANSOLOV PULPING

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ABSTRACT. *The use of non-wood fiber sourced from agricultural waste for papermaking has attracted the interest of many researchers. In this study, paper made from pineapple leaf fiber (PALF) was developed using environmentally friendly approaches. This study aimed to prepare a PALF paper using organosolv pulping with acetic acid (AcOH) and 0.1% hydrochloric acid as a catalyst. The PALF was treated with varying AcOH concentrations (16%, 20%, 24%, 28%, and 32%) for 9 hours to study the effectiveness of delignification in producing high-quality pulp. Additionally, the morphological and mechanical properties of the PALF paper were characterized to analyze its potential as a quality paper. The study discovered that organosolv pulping could produce fibers that can be made into paper with properties comparable to those produced by other conventional methods. Based on Fourier Transform Infrared (FTIR) analysis, the emergence of cellulose-associated peaks and the reduced intensities of peaks attributed to lignin and hemicellulose suggest effective delignification. The Scanning Electron Microscope (SEM) analysis revealed that the treated PALF consists of well-separated cellulosic microfibrils. Meanwhile, mechanical analysis using the Universal Testing Machine (UTM) showed that the tensile strength of the papers (0.25, 0.23, 0.27, 0.20, and 0.19 MPa) varied, while the tearing resistance showed an increasing trend (2.07, 5.15, 6.86, 10.03, and 11.1 mN·m²/g) with increasing AcOH concentration. These findings suggest that PALF is a viable alternative for sustainable paper production.*

INTRODUCTION

It is undeniable that paper is an indispensable part of our lives. However, the increasing demand for paper has led to higher production rates, which in turn require more trees to be cut down for wood pulp. This has significant environmental consequences. Wood remains the primary raw material for producing various types and qualities of paper. However, the use of wood fibers for pulping has become increasingly problematic due to their scarcity in recent years. This has prompted interest in alternative sources, such as non-wood lignocellulosic fibers. Currently, non-wood fibers account for only about 10% of the global papermaking industry (Sibaly & Khadoo-Jeetah, 2017). In response to these challenges, researchers have explored various non-wood fibers for paper production. Among these, pineapple leaf fiber (PALF) has emerged as a promising candidate. Malaysia, with its substantial pineapple cultivation, offers an abundant supply of pineapple leaves, which are often discarded as agricultural waste. According to the Malaysian Pineapple Industry Board (MPIB), the country produced about 416,000 metric tons of pineapples in 2022, with Sabah contributing around 468 hectares of cultivated area and production of 7,587 metric tons (MPIB, 2022). Hence, the leaves produced from this pineapple cultivation can be developed for use as fiber in the pulp and paper-making industry.

In this research, the potential of pineapple leaf fiber (PALF) as a substitute for wood in paper production was explored. The use of PALF in the paper industry is still relatively new (Sibaly & Khadoo-Jeetah, 2017). The abundant availability and lower cost of lignocellulosic fibers like PALF in Malaysia provide a promising alternative source for pulp and papermaking (Dhanasekar *et al.*, 2023). Pineapple, scientifically known as *Ananas comosus*, belongs to the Bromeliaceae family. Pineapple leaf is primarily composed of cellulose (70 - 80%), lignin (4.8%), and hemicellulose (19%) (Amirul Azan *et al*., 2020; Asim *et al*., 2015). The high cellulose content of pineapple leaf makes it a viable raw material for producing quality paper, as cellulose is critical to paper strength and durability (Laftah & Wan Abdul Rahman, 2016). PALF also boasts consistent fiber quality, favorable chemical composition, and good mechanical strength (Mahatme *et al*., 2018). Furthermore, PALF has a lower lignin content compared to other non-wood cellulosic fibers (Kumar, 2020).

This study focused on the characterization of paper produced from pineapple leaf fibers (PALF), addressing a critical gap in sustainable material alternatives for the paper industry. While PALF has shown potential as a non-wood lignocellulosic fiber, there is limited research on its application in environmentally friendly pulping processes and the quality performance of the resulting paper (Rodríguez *et al*., 2018). However, there remains a lack of comprehensive evaluation of PALF using greener pulping technologies, such as organosolv pulping, particularly in the context of Malaysian agricultural waste utilization (Saberikhah *et al*., 2011). To address this gap, the present research investigates the use of organosolv pulping, a chemical process that uses organic solvents to remove lignin as a more sustainable alternative. Despite its environmental advantages, organosolv pulping has not been extensively applied to PALF, and its effects on fiber integrity, chemical composition, and paper mechanical properties remain underexplored. Therefore, this study aimed to fill this knowledge gap by characterizing PALF-based paper through a combination of analytical techniques: Fourier-transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) for chemical and morphological assessment, and a Universal Testing Machine (UTM) for mechanical performance evaluation.

MATERIALS AND METHODS

Sample Collection

The pineapple leaves (*Ananas comosus*) were collected from a local pineapple plantation at Putatan, Sabah, Malaysia. The green fresh leaves were used in this study.

Pulping and Paper Making Process

The pineapple leaves were treated with varying concentrations of acetic acid, AcOH (16%, 20%, 24%, 28%, and 32%) for 9 hours at 100 °C, with 1 mL of concentrated HCl added as a catalyst. Following the delignification process, the treated leaves were filtered and rinsed with distilled water to remove any residual chemicals. The resulting pulps were bleached with 30% hydrogen peroxide (H_2O_2) for 4 hours at 60 °C. The bleached pulps were then used to prepare PALF paper sheets using a mold and deckle. The paper sheets were flattened and dried overnight in an oven set to 60 °C. Finally, the dried paper sheets were stored at room temperature for further characterization. The obtained paper sheet was labeled as shown in Table 1.

Fourier Transform Infrared (FTIR) Analysis

The paper samples were analyzed using Fourier Transform Infrared (FTIR) spectroscopy to identify the presence of cellulose and lignin functional groups in the PALF, both before and after pulp treatment.

The analysis was performed with a Perkin Elmer Spectrum 100 FTIR Spectrometer. The spectra were recorded in the wavelength range of 4000 to 450 cm^{-1}, with each measurement consisting of 4 scans.

Table 1. Pineapple leaf fibre paper (PALF-paper).

PALF-Paper	Acetic Acid Concentration (%)
PALFP16	16%
PALFP20	20%
PALFP24	24%
PALFP28	28%
PALFP32	32%

Surface Morphology Analysis

The paper samples were analyzed using a Scanning Electron Microscope (SEM) to examine the surface morphology of the PALF in each sheet. This analysis was conducted with a Carl Zeiss EVO MA 10 instrument. Images of the fibers were captured at magnifications of 100× and 500×, with an accelerating voltage of 15 kV (Soloi & Hou, 2019). The samples, being non-conductive, were coated with a sputter coating to enhance image quality and resolution.

Mechanical Properties

The tearing resistance, also known as the Elmendorf tear test, was measured to assess the internal tearing resistance of the PALF paper using a ZB-SLY1000B tearing machine. Test pieces were cut to dimensions of 6.5 cm × 7.5 cm. Additionally, the tensile strength of the samples was evaluated using a GOTECH Electronic Mechanical Testing Machine, with test pieces cut to dimensions of 10 cm × 1.5 cm.

RESULTS AND DISCUSSIONS

Fourier Transform Infrared Analysis

The effect of chemical treatment using the organosolv method on the removal of lignin from PALF was examined in this study. Lignin is typically regarded as an undesirable polyphenolic compound with an amorphous structure composed of three types of phenylpropane units: p-coumaryl, coniferyl, and sinapyl alcohols. The quality of paper is defined by its high cellulose and hemicellulose content, along with low lignin content, which contributes to its strength and bleachability. The FTIR spectra of untreated PALF and the PALF paper are shown in Figure 1. As observed in the spectrum in Figure 1, the strong absorption band occurs at 3300 cm^{-1} corresponds to the -OH stretching vibration (Obi Reddy *et al.*, 2014) in both untreated and treated PALF. These peak decreases in intensity and become narrower due to the formation of free -OH groups. Meanwhile, the absorption bands around 2918 cm^{-1}, which correspond to the asymmetric and symmetric stretching vibrations of methylene groups (C-H) in cellulose component, become prominent, reflecting that the removal of the lignin was successful (Reddy *et al.*, 2009). It is also notable that the peak around 1730 cm^{-1}, attributed to carbonyl stretching (C=O), has decreased in intensity, likely due to the removal of a substantial portion of uronic acid, a constituent of hemicellulose xylan, from the PALF (Reddy *et al.*, 2009). Furthermore, the intensity of the peaks around the 1630 cm^{-1} which are attributed to the C-H of aromatic lignin, also decreased and completely disappeared in the PALF treated with the highest concentration (32% AcOH). The absorption band near 1425 cm^{-1} showed a reduction in intensity due to the changes in the environment of the C6 atom, potentially related to the formation or breaking of the hydrogen bond at the O6 position in cellulose (Saha *et al.*, 1991). Furthermore, the band near the 1245 cm^{-1} region has also reduced in intensity, probably due to the disappearance of ester and acid carbonyl stretching vibrations in hemicellulose (Obi Reddy *et al.*, 2014). In addition, the peaks near 890 cm^{-1} are associated with the deformation and stretching of C-O-C, C-C-H, and C-C-O in the β-glycosidic linkage of the glucose ring (Gea *et al.*, 2018). The appearance of the 890 cm^{-1} peak in the treated fibers could be due to an increase in cellulose components (Sasikala & Umapathy, 2018). Overall, the organosolv pulping process resulted in effective lignin removal, as observed from the FTIR spectra, where lignin-related peaks have reduced in intensity as the concentration of AcOH increases.

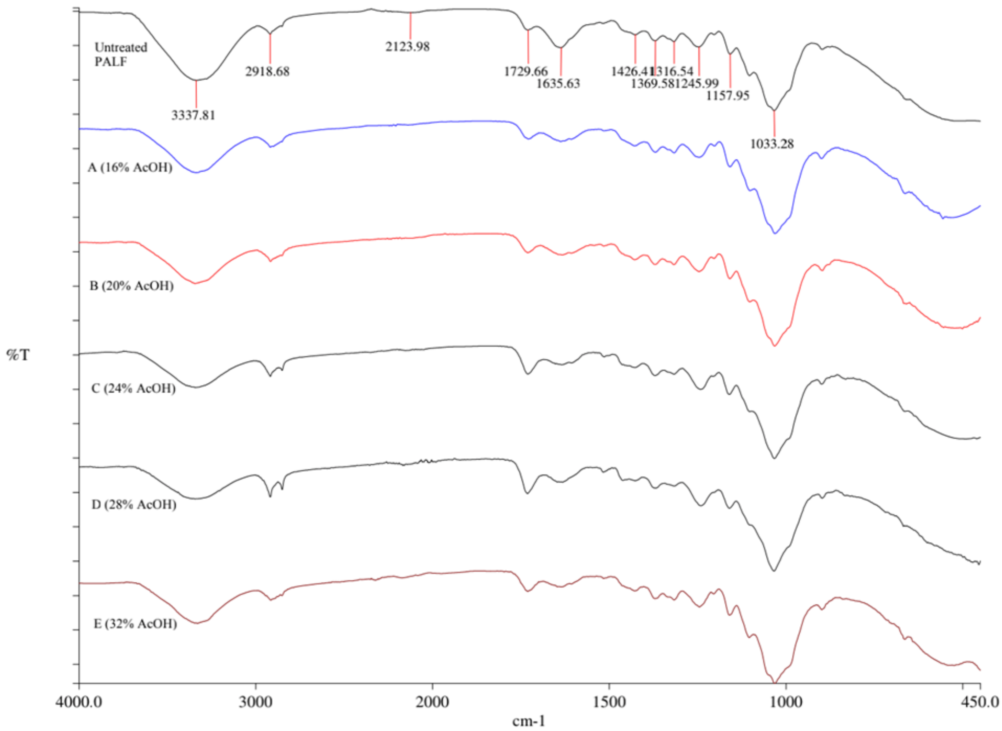


Figure 1. FTIR spectra of untreated PALF and treated PALF using different AcOH concentrations.

In organosolv pulping, the organic acid used principally acts on the impregnation of vegetal tissue and the solubilization of lignin fragments (Xu *et al.*, 2006). The principal chemical reactions involved in this pulping are the cleavage of β-O-4 linkages of lignin, lignin condensation, ester hydrolysis, along with OH groups esterification (Li *et al.*, 2016). At high temperature, several chemical reactions take place during the pulping process, which include the cleavage of β-O-4 and aromatic C-C linkages, new C-O bonds formation which is attributed to the lignin repolymerization (Li *et al.*, 2016). Acidic treatment of fiber also involved the hydrolyzation of hemicellulose and lignin through the breakdown of the polysaccharides to simple sugars, leading to the release of the cellulosic fibers. Meanwhile, the mechanism involved in the bleaching process is lignin oxidation, which results in the dissolution and degradation of lignin (Cherian *et al.*, 2008). The main purpose of the bleaching process is to remove any excess lignin, apart from removing the phenolic compounds or molecules containing chromophore groups after the pulping. This is to increase the brightness of the produced pulp. Bleaching is also essential for the improvement of the physical properties of the fiber, which can be attributed to the better hydration of the pulp during the bleaching process (Jahan *et al.*, 2014). This is because for unbleached organic acid pulp, the hydroxyl groups of the cellulose were either acetylated or formylated, which causes the hydration of the pulp to be reduced (Jahan *et al.*, 2014).

Scanning Electron Microscope (SEM) Analysis

The morphological changes in untreated PALF and pulp fibers treated with five different concentrations of acetic acid, as observed through SEM analysis, are shown in Figure 2. The morphological images of untreated PALF reveal the multicellular nature of the fiber. Generally, most natural cellulosic fibers have a multicellular structure, with cellulose bound together by lignin and hemicelluloses. This is evident in the image, where the fiber's surface is completely covered with lignin and other impurities, making the cellulosic fibers indistinguishable. Meanwhile, in the treated fibers PALF16-PALF32, the emergence of a fibrillar structure, consisting of cellulosic microfibrils, can be observed. Fibrous cell of PALF consists of a vascular bundle system in the form of bunches (Asim *et al.*, 2015), as can be seen at 500× magnification at higher AcOH concentration. Additionally, the impurities on the fiber surface have disappeared. As the concentration of AcOH increases, more fibrils become visible, and the fiber bundles are oriented in various directions within the samples (Soloi & Hou, 2019). With higher concentrations of AcOH, the fiber bundles disintegrate into elementary fibers, which is noticeable through the more widespread distribution of these fibers. The rough surface of the fibers is attributed to increased adhesion between the fiber interfaces and the absorption of water during the papermaking process (Obi Reddy *et al.*, 2014). The SEM image of this PALF paper, compared to hardwood paper for newsprint purposes, shows that the fibre oriented in a similar manner to hardwood paper reflects the potential of PALF as an alternative for pulp and paper production (Chinga-Carrasco, 2009).

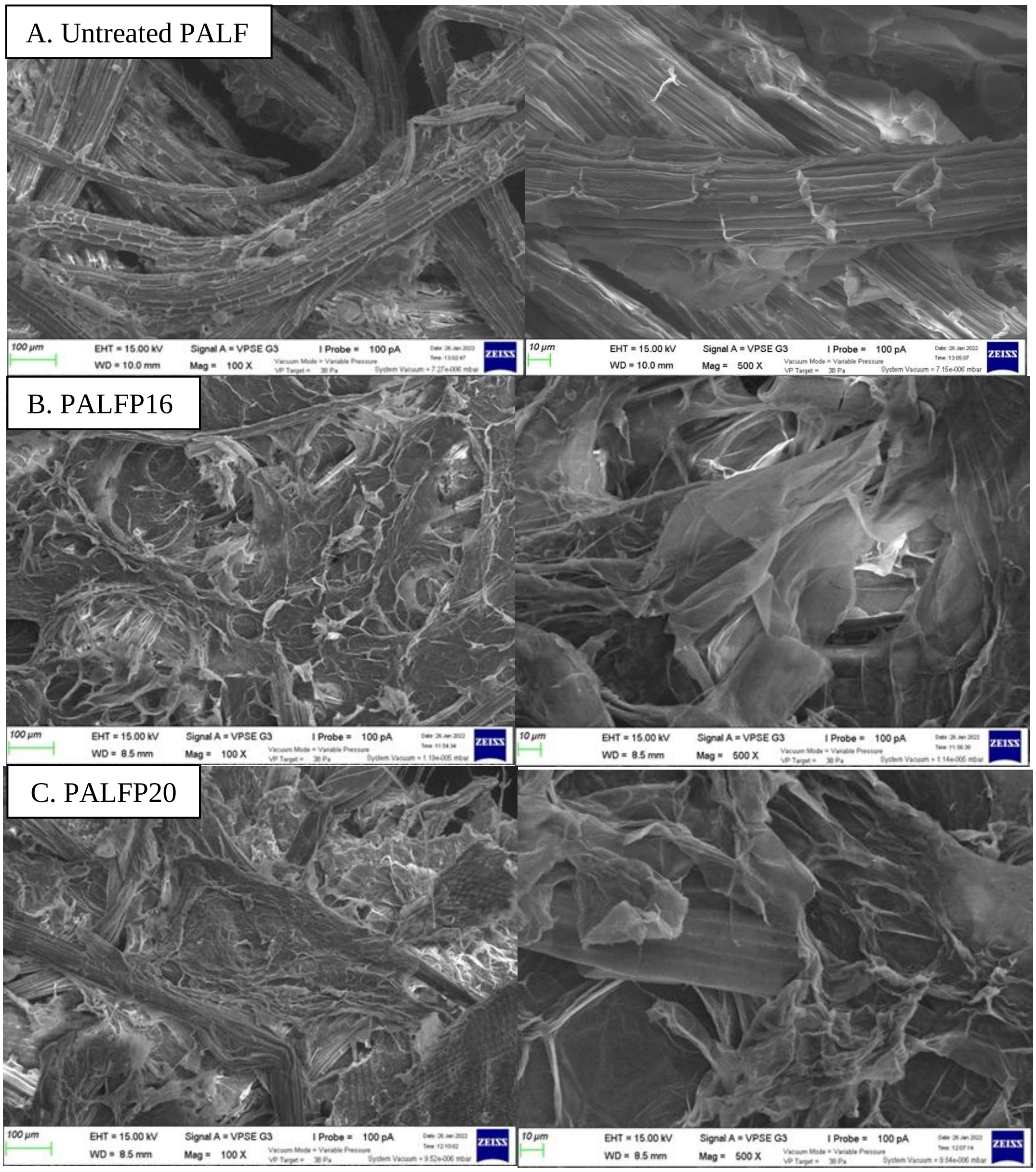
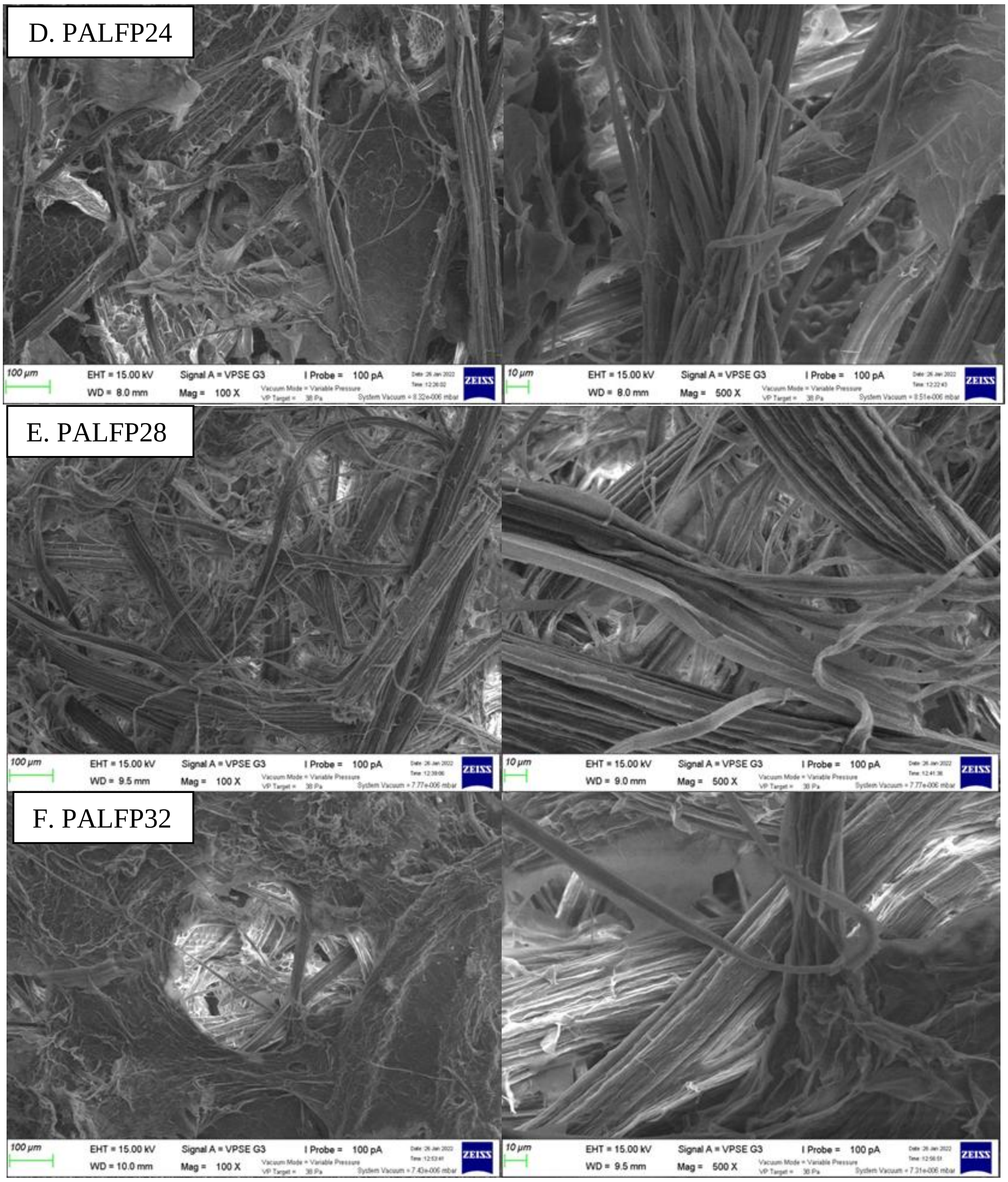


Figure 2. Surface morphology of PALF paper at 100× (left) and 500× (right) magnification: (a) Untreated PALF; (b) PALFP16; (c) PALFP20; (d) PALFP24; (e) PALFP28; and (f) PALFP32.



Continue **Figure 3.** Surface morphology of PALF paper at 100× (left) and 500× (right) magnification: (a) Untreated PALF; (b) PALFP16; (c) PALFP20; (d) PALFP24; (e) PALFP28; and (f) PALFP32.

The physical appearances of the PALF paper sheets are shown in Figure 3. Noticeably, the appearances of the PALF paper sheets are not very different from one another. PALFP16 – PALFP28 are quite alike in terms of their brightness and surface textures. Meanwhile, PALFP30 and PALFP32 have a rougher surface, which is most probably due to the fibrillation of the PALF at higher concentrations of AcOH. Furthermore, the coarseness of the papers' surfaces could also be attributed to the non-fibrous material, such as the outer skin of the leaves, which were caught in the mold along with the fiber during the molding step and not finely ground during the grounding process. But considering the deep green color and hard outer coating of the pineapple leaves, it can be said that the organosolv pulping has effectively delignified the PALF, even though the AcOH concentration used was relatively lower than that of other studies. Besides, the bleaching with H_2O_2 had helped in improving the appearance of the pulp.

Figure 4. Physical appearance of PALF paper using organosolv pulping.

Tensile Strength Analysis

The mean tensile strengths of the PALF paper samples are shown in Figure 4. It was found that the average tensile strength of the PALF paper varies with the varying concentration of AcOH. The tensile strength wane from PALFP16 to PALFP20 but then improved on PALFP24. This could be due to strong inter-bonding of the fiber in PALFP16, but weaker if compared to that of PALFP24. However, the tensile strength then reduced again from PALFP30 to PALFP32. As the cooking solvent's concentration increased, the tensile strength decreased except for PALFP24. The decreasing trend can probably be associated with the cellulosic fiber degradation at higher concentrations of AcOH (Soloi & Mohammad, 2023). This is because, although the lignin removal is enhanced at higher concentration, there is a possible risk of cellulose degradation and fiber rupture (Soloi & Mohammad, 2023). Moreover, the decrease in tensile strength could be related to the hydrophilic nature of the PALF (Asim *et al.*, 2015; Rajeshkumar *et al.*, 2020). The tensile strength of PALF observed in this study was slightly higher than that reported for NaOH-treated PALF in the study by Evelyn *et al.* (2019), suggesting that the current treatment method may offer improved reinforcement properties.

Tearing Resistance Analysis

The tearing resistance analysis of the PALF paper was tested using the Elmendorf tearing tester machine. Figure 5 shows the tearing resistance of the paper sheet. The tearing index of the PALF paper samples produced increases from PALFP16 to PALFP32. This suggests that the increasing concentration of the AcOH has improved the tearing resistance as more of the cellulose bunches were formed during the pulping. As can be seen in the SEM image, PALF has a long fibre bunch. The longer the fibre, the greater the resistance of the paper to tearing (Dwivedi *et al*., 2010). The tearing index of PALFP16 shows the lowest value, which could be due to the poor paper formation of the fibers, which resulted from the high amount of residual lignin and impurities. The tear index of PALF obtained in this study exceeded that of NaOH-treated PALF reported in earlier research (Evelyn *et al*., 2019), suggesting improved fibre integrity and resistance to tearing under the current treatment method.

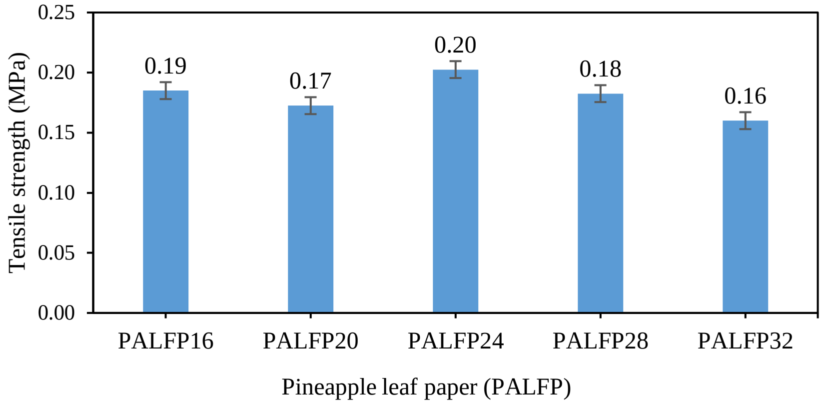


Figure 5. Tensile strength of PALF paper at different concentrations of acetic acid.

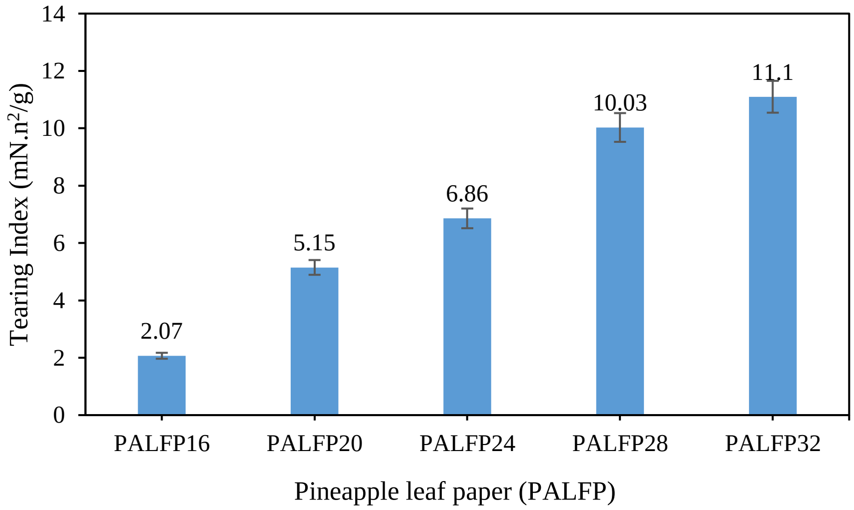


Figure 6. Tearing Index of PALF paper at different acetic acid concentrations.

CONCLUSION

In this study, sustainable paper was successfully produced from pineapple leaf fibre (PALF) without the addition of any external binder, as the resulting pulp was readily mouldable into paper sheets. Moreover, Fourier Transform Infrared Spectroscopy (FTIR) analysis confirmed effective delignification of PALF, as evidenced by the reduced intensity of lignin- and hemicellulose-associated peaks, along with the emergence of cellulose-characteristic peaks in the treated samples. Additionally, Scanning Electron Microscopy (SEM) revealed notable morphological differences between untreated and treated fibres. The treated PALF exhibited the emergence of exposed cellulosic fibrils, as well as the separation of fibre bundles into individual fibres. Furthermore, mechanical testing demonstrated variability in tensile strength across all paper samples, with the highest tensile strength recorded in paper produced from PALF treated with 25% acetic acid (AcOH). On the other hand, the tear resistance of the PALF paper showed a consistent increasing trend with higher concentrations of the pulping solvent, indicating a positive correlation between solvent strength and tearing performance. Above all, the ability to produce mouldable, binder-free paper with adequate mechanical strength suggests potential for small-scale paper production or eco-friendly packaging applications to support the pulp and paper industry in Malaysia. However, further optimisation of treatment conditions, scale-up trials, and durability testing under realuse conditions are recommended to enhance the material's applicability and move toward industrial implementationPlease conclude your work incorporating your most important findings as well as future works.

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