Effects of extraction method on dry pulp yield and morphological properties of pineapple leaf fibre


Biopolymer Research Group, School of Chemical and Energy Engineering, Faculty of Engineering, Universiti Teknologi Malaysia, UTM Johor Bahru, 81310, Johor, Malaysia

Abstract

Pineapple leaves (PALs) are useful agro wastes which have the potentials to be used as an alternative source of non-wood natural fiber. In this study, different extraction methods had been investigated to identify the most feasible pineapple leaf fiber (PALF) extraction method, based on the dry pulp yield and the PALF morphology. The manual retting using a ceramic scraper led to low yield of around 1.8 % (wt.), while water retting for 21 d led to about 6.0 % (wt.) yield of dark greenish dry pulp. Both methods resulted in PALFs which still contained with non-cellulosic residues, as verified by scanning electron microscopic (SEM) imaging. The chemical extraction of PALF using various NaOH solution concentrations (i.e. 1 to 20 % (wt.)) gave different yields. Based on the SEM images, clean and smooth surfaces of fibrils were observed when the NaOH solution concentrations applied at or higher than 6 % (wt.), implying that all impurities including lignin and wax had been completely removed. The mechanical-chemical extraction method produced significantly more dry pulp compared to the chemical extraction method at the same NaOH solution concentration. This was attributed to the fact that the crushed PAL has a larger surface area, thus providing more reaction possibilities with NaOH solution. Finally, it was found that the crushed PAL that cooked at 90 °C needed at least 90 min of cooking time to obtain satisfying whitish dry pulp.

Keywords: Pineapple leaf, fibre extraction, extraction methods, dry pulp yield, fiber morphology

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INTRODUCTION

Increasing global industrialization has led to significant consumption of paper especially for the purpose of packaging, which accounts for over 41 % of paper usage globally (Beckline et al., 2016). According to the pulp and paper capacities survey 2013 – 2018 by the Food and Agriculture Organization of the United Nations (FAO, 2014), the global paper consumption will increase to 500 million tons in year 2025 which corresponds to an annual paper consumption growth of 1.6 %. The intensive paper consumption leads to significant depletion in natural resources especially wood due to rapid deforestation. Therefore, much efforts have been invested to study different non-wood alternatives as potential candidates for paper production, which include palm oil empty fruit bunch (Rodriguez et al., 2014; Kim et al., 2014), bagasse (Rezayati-Charani et al., 2006), wheat-straw (Hedjazi et al., 2009), sugarcane straw (Saad et al., 2008), rice-straw (Rodríguez et al., 2008), napier (Rodriguez et al., 2008; Haameem et al., 2016), kenaf (Low et al., 2018), tea waste/kapok (Majid et al., 2018), tree pruning waste (Low et al., 2019) and reeds (Feng and Alén, 2001). One of the potential replacements of wood in paper pulp production is pineapple leaf fiber (PALF), which is abundantly available in Malaysia (Daud et al., 2013; Yusof et al., 2012). Several researches have been reported to utilize pineapple biomass in Malaysia especially pineapple leaves including PALF/polypropylene (PP) composites (Arrib et al., 2006), PALF/glass vinyl ester (VE) hybrid biocomposites (Zin et al., 2019), PALF/polyester composites (Senthilkumar et al., 2019), silane treated kenaf/PALF phenolic hybrid biocomposites (Asim et al., 2018), scratch resistance epoxy composites from PALF, napier and hemp fibres as filler (Ridzuan et al., 2019), sound adsorption (Putra et al., 2018) and soil cover (Sarah et al., 2018).

Pineapple leaf (PAL) serves as an attractive option to pulp making as pineapple plant can produce up to 1.5 kg of leaves per plant that left in the field after cultivation (Sibaly and Jeetah, 2017). Furthermore, the PALF possesses high cellulose content (66.2 %) and low lignin content (4.2 %) which offer good mechanical properties to handle the potential abrasion and tear as packaging material (Mohamed et al., 2009; Daud et al., 2013). Cellulose is a long branchless glucose polymer that can influence the thermal degradation properties of the fibre (Lai et al., 2013) and the strength of the paper (Laftah and Abdul Rahaman, 2015), resulting PALF to has immense potential as reinforcement and can be utilized as the alternative source of fiber for composites and pulp industry.

With the positive reports from several studies, an effective fiber extraction method must be established to enhance the feasibility of PAL usage as a non-wood alternative for pulp production (Daud et al., 2013; Yusof et al., 2012). The conventional extraction method often falls into three categories, namely mechanical method, chemical method and mechanical-chemical method. Sarah et al. (2018) reported that the fibers extracted using a mechanical-chemical method had low lignin content and high crystallinity when compared to the “roller and bladder” mechanical system and chemical extraction with sodium hydroxide (NaOH) solution and acetic. Sarah et al. (2018) used immersion technique when dealing with chemical extraction method as the PALF was soaked in the solution for 24 h without any addition of heat during the process.
This paper was reported on the comparison between the effectiveness of the mechanical, chemical and chemical-mechanical methods in extracting the PALF from the PAL. The effects of heating in speeding up the process were also investigated and discussed.

EXPERIMENTAL

Materials

The pineapple leaves (PALs) of Josapine variant were collected from the Malaysian Pineapple Board (MPIB) plantation in Alor Bukit, Pekan Nanas, Johor, Malaysia. An industrial grade of sodium hydroxide (NaOH) flake was purchased from PT Asahimas Chemical. The distilled water was used in solution preparation.

Extraction Methods

**Combing method (Manual Retting)**

Pineapple leaves (PALs) were cleaned using tap water to remove the dirt and other residues. The cleaned PALs were combed with a ceramic scraper to extract the PALF by removing the non-fibrous components from the leaves. The fibers obtained were washed under running tap water to remove the remaining dirt residues. The extracted fibers were then sun-dried for few days until constant weights were achieved. Weights of the extracted fibers, PALFs were recorded.

**Water retting method**

The clean PALs were crushed using a fabricated sugarcane crusher machine. The crushed PALs were then soaked in water using the solid/liquid ratio of 1:15 for (3, 7, 10, 14, 17 and 21) d. The retted fibers were then cleaned under running tap water and oven-dried at 60 °C for 24 h. Weights of the extracted PALFs from each soaking time were recorded.

**Chemical extraction method**

Sodium hydroxide (NaOH) solution was used as a medium to extract PALF from the fresh PALs. The NaOH solutions were prepared at different concentrations between (1 to 20) % (wt.) by dissolving the NaOH flakes in distilled water. The clean PALs were cut into approximately 5 cm in length prior to soaking in the NaOH solution by using the solid/liquid ratio of 1:15 for 24 h at room temperature (25 °C). After 24 h, the treated PALs were washed under running tap water until the fibers became clear yellowish in colour. The cleaned fibers were then oven-dried overnight at 60 °C. Weight of the dried PALF was weighed and recorded.

**Mechanical–chemical extraction method**

The mechanical–chemical extraction method followed similar routes as in the chemical extraction method. The NaOH solution concentrations of (1, 3, 5, 7, 9 and 11) % (wt.) were used. The PALs were crushed with a crusher prior to undergoing the alkali (NaOH solution) treatment. The crushed PALs were then cooked in the NaOH solutions at 90 °C with different cooking times as described previously in the chemical extraction method.

In cooking time experiments, the clean PALs were added to NaOH solution with different concentrations (i.e. 1, 3, 5, 7, 9 and 11) % (wt.) using the solid/liquid ratio of 1:15. The mixtures were then heated at 90 °C. The cooking times were varied at (30, 60, 90, 120 and 180) min for each experiment. Once completed, the PALs were cleaned and washed under running tap water until they were free of residues. The fibers were oven-dried overnight at 60 °C. Weights of the dried PALFs were recorded.

**Dry pulp yield**

The dry pulp yield was calculated according to Eq. (1)

\[
\text{Dry Pulp Yield (%) = } \frac{A}{B} \times 100
\]

where A is the initial mass of PAL used in the extraction process (g) and B is the mass of the PALF obtained from the extraction process (g).

**PALF morphology**

The PALF morphology and fiber arrangement were determined by using a scanning electron microscope (SEM) model JEOL JSM-IT300LV (USA). Prior to the scanning, the samples were sputter-coated with 10 nm platinum under vacuum. The SEM images of the samples were taken at different magnifications. The visual images were obtained using a conventional digital camera model Samsung N7100 (Korea).

RESULTS AND DISCUSSION

**Combing method (Manual Retting)**

A whitish PALF obtained from the manual retting method is shown in Fig. 1. The average dry pulp yield from this method was calculated to be 1.8 % (wt.). In average, the process to obtain 0.7 g of PALF from a single leaf weighed around 40 g took almost 20 min. The SEM image in Fig. 1(b) shows the residue of non-cellulosic substances on the surface of the fiber. Since the fibers were manually extracted using a scraper, the quality (i.e. trace of impurities) and the amount of fibre yield (loss of fibres) depended on the skill of the workers. Hence, the combing method was is not a plausible fiber extraction method, especially for big scale production.

![Fig. 1](image-url) (a) Visual and (b) SEM (1500X magnification) images of the manually retted PALF.

**Water retting method**

The image of water retted PALF and its SEM images are shown in Fig. 2, while the dry pulp yield of PALs is presented in Fig. 3. It could be observed that the dry pulp yield decreased with increasing number of immersion time, where the dry pulp obtained in 21 d was between (6.0 to 6.6) % (wt.). The trend was due to the fact that more non-cellulosic substances were softened and washed away as the day of immersion increased. These values were significantly higher than that of the manual retting method. As compared to the manual retting, this extraction method did not require manual labour to remove the residues or impurities from the PALF. However, the SEM images show that some residues still remained on the surface of the fibers. The dark greenish colour of the PALF as shown in Fig. 2(a) was thought to correspond to the presence of residues in the fiber (Wang et al., 2016). Hence, the water retting method was also less effective.

![Fig. 2](image-url) (a) Visual and (b) SEM (1500X magnification) images of water retted PALF after 21 d of immersion.
Chemical extraction method

A sample of PALF extracted using NaOH solution is shown in Fig. 4, while the dry pulp yield of the chemically treated PAL is shown in Fig. 5. The extracted PALF was seen to be whitish in colour, signifying a complete removal of lignin. During the process, the NaOH has disrupted the lignin-carbohydrate linkages that subsequently introduced partial removal of hemicellulose and facilitated the lignin removal (Jahan and Pourali, 2019). Fig. 5 clearly shows that the dry pulp yield decreased with increasing NaOH solution concentrations. However, after 7 % (wt.) NaOH solution concentration, the dry pulp yield was constant at around 3 % (wt.), which was consistent with other studies (Mohamed et al., 2009; Daud et al., 2013; Laftah and Abdul Rahaman, 2015).

Mechanical–chemical extraction method

For the mechanical-chemical extraction, the PALs were crushed with a crusher prior to cooking in NaOH solution at 90 °C. The NaOH solution concentrations were varied (i.e. 1, 3, 5, 7, 9 and 11 % (wt.)) as previously described in the chemical extraction method. As shown in Fig. 7, a whitish dried pulp was obtained when the concentration of NaOH solution was set at 3 % (wt.) and above, indicating the complete removal of residues from PALF at these NaOH solution concentrations. The SEM images of extracted PALF by mechanical-chemical method are presented in Fig. 8.
From Fig. 8(a), it can be observed that the PALF was still covered with the cementing substance layer. This indicated that it was not sufficient for the defibrillation to occur at 1 % (wt.) NaOH solution concentration even with the help of heating. The fibril started to appear more prominently at 3 % (wt.) NaOH solution concentration (Fig. 8(b)). This showed that the mechanical-chemical method, with the help of heating, accelerated the defibrillation process by making it to happen at the lower NaOH solution concentration as compared to the chemical method. Fig. 8(c) to 8(f) show that there was no significant change between the PALF extracted from (5, 7, 9 and 11) % (wt.) NaOH solution concentrations. This implied that with the help of heating at 90 °C, for each case, the delignification process was speed up and more fibrils were exposed at and above 3 % (wt.) NaOH solution concentration.

As presented in Fig. 9, the dry pulp yield decreased with increasing NaOH solution concentrations. Interestingly, even at 11 % (wt.) NaOH solution concentration which led to the lowest PALF yield, the quantity of dry pulp obtained was 6.6 % (wt.) which was still significantly higher than that of the chemical extraction (2.8 % (wt.)). Moreover, a larger quantity of crushed PALF could be treated with the same amount and concentration of the NaOH solution comparing to the chemical extraction method. This was because by crushing, volume of the PALF decreased and at the same time, surface area of the fibers increased as they were broken down into smaller pieces. The higher surface area also promoted a faster reaction between NaOH and PAL. Hence, it could be concluded that the mechanical-chemical extraction was the most favourable among all extraction methods. This result was in agreement with the finding by Sarah et al. (2018) who reported that the semi-mechanical extraction method yielded PALF with high crystallinity and low lignin content.
The visual image was used to determine the most suitable cooking time, based on the colour and residues left on PALF cooked at 3 % (wt.) NaOH solution concentration. From Figs. 11(a) and 11(b), it can be seen the greenish leaf-like residues were still noticeable on the PALF after 30 min and 60 min of cooking times. This signified that the cooking time less than 60 min was insufficient to remove the residues. However, above 90 min, all PALFs exhibited whitish coloured dry pulps. Hence, in order to ensure good quality PALFs and save more energy, the optimum cooking time should be at least 90 min.

**CONCLUSIONS**

The pineapple leaf fiber (PALF) was successfully extracted through the mechanical, chemical and mechanical–chemical extraction methods. Based on the dry pulp yield and morphological appearance, the manual retting using a ceramic scraper was the most inefficient method since it was highly labour intensive and yet, produced the lowest dry pulp yield at about 1.8 % (wt.). The water retting for 21 d led to about 6.0 % (wt.) dry pulp yields. However, the pulps were dark greenish in colour, indicating the presence of unwanted residues, as verified by SEM image. The chemical extraction of PALF using (6 to 20) % (wt.) NaOH solution concentrations was found to successfully extract good quality PALF, as lower NaOH solution concentrations of 1 % (wt.) and 3 % (wt.) failed to completely remove the residues. As compared to the chemical extraction, the mechanical–chemical extraction promised a dry pulp yield of more than 6.0 % (wt.) even at higher NaOH solution concentrations. Moreover, the same given amount and concentration of NaOH solution was able to treat a larger quantity of crushed PAL. It was also found that the extraction process could speed up by cooking the solution at 90 °C for 90 min. The cooking time lesser than 90 min resulted in leaf-like PALF, which is an indication that the leaf structure was not properly broken down. Finally, it could be concluded that the mechanical–chemical method using 3 % (wt.) NaOH solution concentration, solid/liquid ratio of 1:15 and 90 min cooking time was the best method to extract the PALF from the PAL.

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