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# Use of steam explosion as a green alternative method to prepare pulp from pineapple leaves

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**Abstract**

The use of agricultural wastes as an alternative form of the cellulosic material to wood has gained considerable attention due to the reduction of deforestation. After cultivation, a large number of pineapple leaves are considered as a waste. Here, the potential steam explosion process was used as a green alternative way to prepare pulp from pineapple leaves without any addition of the chemical treatments. The pineapple leaf fibers were defibrillated from fiber bundles due to the rapid depressurization with the steam pressure between 16 and 20 kgf $cm^{-2}$. The partly removal of hemicellulose and lignin associated with the increase of the cellulose content was found from the fibers after introducing the steam explosion process. With increasing the steam pressure, the decrease of the fiber widths and degree of polymerization were observed, and a higher content of cellulose and less contents of hemicellulose and lignin were obtained. The tensile index of the paper was enhanced when the fibers were steam exploded with the higher steam pressure. The paper prepared from the 20 kgf $cm^{-2}$ steam exploded fibers showed the higher tensile index value of 8.18 Nm $g^{-1}$ in comparison with that of 4.50 Nm $g^{-1}$ obtained from the paper of the untreated fibers. In contrast, the lower tear index of the paper made from the fibers steam exploded with the higher steam pressure was found. Pineapple leaf fibers would be possibly used as an alternative material in pulp and paper applications and as a reinforcing agent in composite applications.

## 1. Introduction

There have been attempts to move toward green materials by using naturally abundant cellulosic materials [1-4]. Among natural and agricultural fibers such as cotton, coir and jute, pineapple leaf fibers present superior mechanical performances because of a higher content of cellulose and lower microfibrillar angle [1,5]. Thailand is one of the largest pineapple producers and exporters [6]. In 2017 the annual fresh pineapple production of 2.3 million tons was reported [7]. After pineapple cultivation, a large amount of pineapple leaves remain as a waste [1,8]. The extraction of fibers from pineapple leaf fibers would be beneficial due to being abundant and inexpensive [1,8]. When the fiber density is taken into account, the specific mechanical properties of the pineapple leaf fibers are similar to those of glass fibers [1]. Due to these reasons, this would broaden the possibility to use the pineapple leaf fibers instead of glass fibers to improve performance of the fossil-based polymers. Many techniques have been proposed to isolate individual fibers from bundles of fibers or cell wall. One of the most efficient methods for defibrillation is steam explosion. This method is considered as a green technique due to removal of hemicellulose and lignin without using chemical reagents and requiring low energy usage [9,10]. This process could not only isolate fibers, but also breakdown lignocellulosic structure, hydrolyze hemicellulose and depolymerize lignin by the sudden depressurization [11]. When fibers are steam exploded, the increase of the cellulose content and reduction of contents of the hemicellulose and lignin within the fibers could be obtained. The aim of this work was to evaluate the less-time and chemical consuming steam explosion process with the steam pressure between 16 and 20 kgf $cm^{-2}$ for defibrillation of pineapple leaf fibers. Fiber morphology and degree of polymerization of the steam exploded fibers were compared with those of the intact pineapple leaf fibers. Mechanical properties (tensile and tear indexes) of paper formed from these steam exploded fibers were investigated to show effect of fiber defibrillation.

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## 2. Experimental methods

### 2.1. Material

Pineapple leaf fibers used as a raw material in this study were extracted from pineapple leaves in the Ratchaburi plantation, Thailand, by decorticated machine.

### 2.2. Preparation of Pineapple Leaf Pulp and Paper

Pineapple leaf pulp was prepared using the method described elsewhere [8,12]. Briefly, pineapple leaf fibers (150 g) were defibrillated using the steam explosion process. The fibers were conditioned at the steam pressure between 16 and 20 kgf $cm^{-2}$ for 5 min, and the pressure was rapidly dropped. During this depressurization, the fibers were disintegrated. The disintegrated fibers were used to form paper with a thickness of ~180 µm and a basis weight of 60 g $m^{-2}$ using a handsheet forming machine according to TAPPI Standard T205 [13].

### 2.3. Characterization

The raw and steam exploded fibers were analyzed to investigate chemical compositions, according to the Technical Association of the Pulp and Paper Industry TAPPI ( test method [13], for instance, cellulose, hemicellulose, and lignin. The surface morphology of the steam exploded and original pineapple leaf fibers was investigated using a scanning electron microscope (JSM-6610LV, JEOL Ltd., Japan) equipped with a secondary electron detector under an accelerating voltage of 5 kV. All samples were gold-coated before investigation. The fiber widths were measured using the *ImageJ* program, and the mean width of each fiber type was calculated from 100 measurements.

Intrinsic viscosity of the intact and steam exploded fibers was determined according to ASTM D1795-13. Subsequently, the degree of polymerization (DP) was calculated based on the following equation.

$$DP = 95 \times \eta \times (c/w) \quad (1)$$

where w is the dried weight of the fiber taken (g), c is the fiber concentration in 0.5 M CED solution (g $ml^{-1}$) and η is the intrinsic viscosity of the cellulose solution (ml $g^{-1}$).

The prepared paper was conditioned at 23°C and 50% of relative humidity, according to ISO 187 before mechanical property testing. Tensile and tear index were tested according to ISO 5270 standard. At least five samples per material were measured to calculate the average and standard deviation values of the tensile and tear strength.

## 3. Results and discussion

Chemical composition measurements of the steam exploded fibers in comparison with raw fibers are presented in Figure 1. The main components in fibers are cellulose, hemicellulose and lignin. Higher proportions of non-cellulosic contents were observed in a raw material with 20.9% of hemicellulose and 4.2% of lignin. These non-cellulosic materials could be partially removed due to the breakdown of the hemicellulose and lignin structure caused by the thermo-mechanicochemical process during the steam explosion process [8,14-16]. This resulted in a higher portion of cellulose in the treated fibers. With increasing steam pressure, the steam exploded fibers with a higher content of cellulose and less contents of hemicellulose and lignin could be obtained. Values of 81.3, 9.5 and 0.4% were presented for contents of cellulose, hemicellulose and lignin, respectively, in the fibers steam exploded with 20 kgf $cm^{-2}$ while the fibers steam exploded with 16 kgf $cm^{-2}$ had only 76.6% of cellulose, 12.6% of hemicellulose and 0.6% of lignin. This could be due to the higher defibrillation at the higher steam pressure, leading to more degradation and depolymerization of hemicellulose and lignin [10,17].

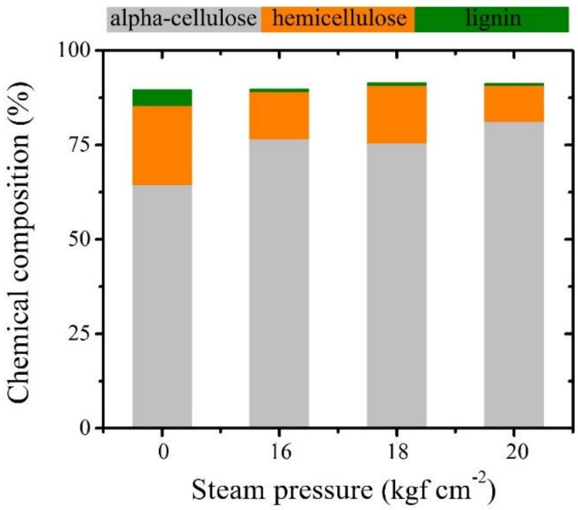


**Figure 1.** Chemical compositions of intact and steam exploded pineapple leaf fibers.

Figure 2 shows fiber morphology before and after the steam explosion method. Due to the rapid depressurization, the isolation of the microfibers from bundles was observed, and the fibers became smaller with increasing the steam pressure from 16 to 20 kgf·$cm^{-2}$. The defibrillation occurred due to the degradation of hemicellulose and lignin caused by the rapid decompression [18].

Figure 3 shows width distribution of the pineapple leaf fibers after the steam explosion process. It was found that only 29% of the fibers steam exploded with the pressure of 16 kgf $cm^{-2}$ with widths of less than 3 µm was found while 44 and 50% of the treated fibers with the pressure of 18 and 20 kgf $cm^{-2}$, respectively, had diameters of less than 3 µm. It would be noted that pineapple leaf fibers used in this study had a low lignin content of 4.2%. Defibrillation would be easily obtained with only steam-explosion process without any further addition of the chemical treatments as lignin acts as a glue to adhere fibers altogether. However, the additional step of the chemical treatment such as alkaline or bleaching might be required for fibers with a high lignin content to eliminate lignin and hemicellulose before microfiber disintegration [11,18].

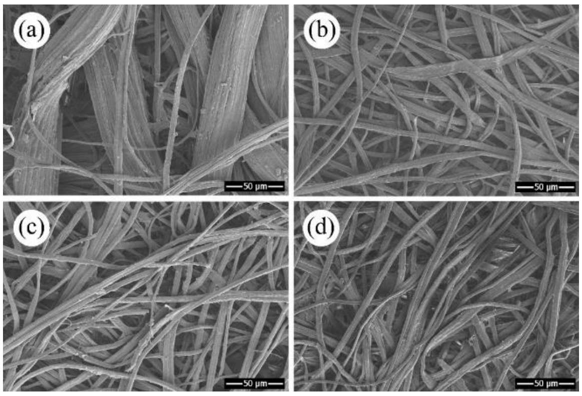


**Figure 2.** Fiber morphology of the steam exploded pineapple leaf fibers with the steam pressure of (a) 0, (b) 16, (c) 18 and (d) 20 kgf $cm^{-2}$, respectively.

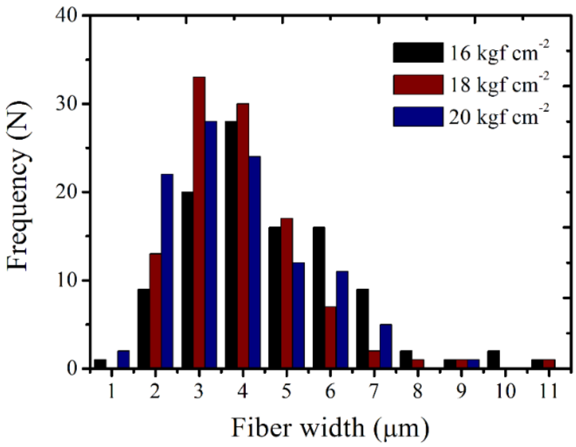


**Figure 3.** Width distribution of the steam exploded pineapple leaf fibers.

Figure 4 shows the average width of the steam exploded fibers in comparison with the original pineapple leaf fibers and the degree of polymerization of the untreated and steam exploded fibers. The average width of the pineapple leaf fibers treated at the pressure of 16 kgf $cm^{-2}$ was 4.65 μm while the mean of the fiber widths of the 20 kgf $cm^{-2}$ steam exploded fibers was 3.78 μm. When the higher steam pressure was introduced, more fibers were disintegrated. It has been previously reported that more homogenous fiber-like materials were observed when the higher steam temperature and longer treatment time were applied [19]. Phinichka et al [17] found that the fibril structure was highly destroyed, and higher amounts of lignin were depolymerized while hemicellulose was more separated when the fibers were steam exploded at higher temperature.

Moreover, the degree of polymerization of the steam exploded fibers was lower with the increase of the steam pressure due to the presence of the heat and high pressure during the treatment [10], as shown in Figure 4. The degree of polymerization of 1,998.5 was measured from the intact pineapple leaf fibers. The significant reduction of the degree of polymerization to 1,482.2 was found with the introduction of the steam explosion method with the pressure of 16 kgf $cm^{-2}$. When the steam pressure increased to 20 kgf $cm^{-2}$, the degree of polymerization of the treated fibers was gradually reduced to 1,115.9. The reduction of the degree of polymerization was caused by breaking inter and intra molecular backbone of the cellulose molecules [10]. The higher the steam pressure, the lower the degree of polymerization of the steam exploded fibers. The similar reduction of the degree of polymerization of the wheat straw after the steam explosion has been also reported [20].

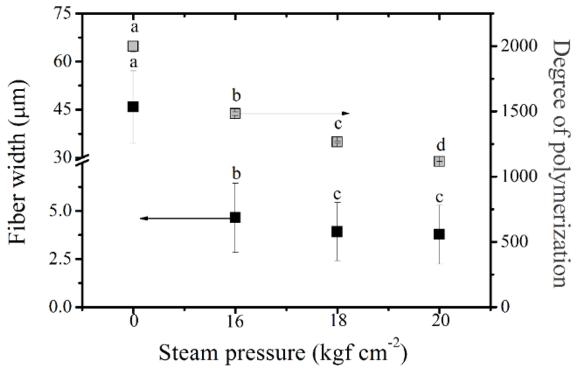


**Figure 4.** Fiber width and degree of polymerization of the pineapple leaf fibers steam exploded with the steam pressure between 16 and 20 kgf $cm^{-2}$ in comparison with intact pineapple leaf fibers. Standard deviations were reported in terms of the error bars, and the significant difference of the data with a P value of less than 0.05 was provided by a different alphabet (a-d), according to a T test analysis.

**Table 1.** Tensile and tear index of paper made from the untreated and steam exploded fibers.

| Paper | Tensile Index (Nm $g^{-1}$) | Tear Index (Nm $m^2$ $g^{-1}$) |
|---|---|---|
| Untreated fibers | 4.50 ± 0.38 | 15.87 ± 0.85 |
| 16 kgf $cm^{-2}$ steam exploded fibers | 4.60 ± 0.19 | 13.16 ± 0.77 |
| 18 kgf $cm^{-2}$ steam exploded fibers | 4.37 ± 0.04 | 13.69 ± 1.68 |
| 20 kgf $cm^{-2}$ steam exploded fibers | 8.18 ± 0.58 | 10.61 ± 1.70 |

Tensile and tear indexes of the paper prepared from the steam exploded fibers and untreated fibers are shown in Table 1. The paper of the untreated fibers had the tensile index of 4.50 Nm $g^{-1}$. When the fibers were steam exploded, defibrillation occurred. Therefore, when paper was prepared with the steam exploded fibers, more hydrogen bonding between adjacent fibers were formed due to the higher surface area of the fibers. This resulted in higher mechanical properties. The tensile index of the paper of the 16 kgf $cm^{-2}$ steam exploded fibers was 4.60 Nm $g^{-1}$, and a value of 8.18 Nm $g^{-1}$ for the tensile index was obtained from the paper made from the fibers steam exploded with the pressure of 20 kgf $cm^{-2}$. On the other hand, the contrast trend of the tear index occurred with increasing the steam pressure to isolate fibers. The tear index of the paper of the untreated fibers was 15.87 Nm $m^2$ $g^{-1}$. The paper of the fibers which passed through the steam explosion with the pressure of 16 kgf $cm^{-2}$ had the tear index of 13.16 Nm $m^2$ $g^{-1}$. With increasing the steam pressure to 20 kgf $cm^{-2}$ the tear index of the paper was reduced to10.61 Nm $m^2$ $g^{-1}$. This could be attributed to the easier tear propagation through the paper made from the steam exploded fibers prepared with the higher steam pressure [21]. During the tearing, there are two mechanisms involved: fiber pulling and fiber rupture. In the paper of the untreated fibers more energy was required to destroy fiber bundles and pulling the large fibers out of the paper in comparison with the paper made from the steam exploded fibers with smaller widths [21]. Similar trends of the tensile and tear index have been reported [22]. Bamboo pulps were treated using a PFI beater from 1,000 to 24,000 beating revolutions. With increasing a number of the beating revolutions, the tensile index increased significantly from 41.23 Nm $g^{-1}$ for the paper prepared from the 1,000 revolution-beaten pulp to 83.90 Nm $g^{-1}$ for the paper made from the 24,000 revolution-beaten pulp. This was due to higher amounts of the hydrogen bonding formed between fibers. However, the increase of the beating revolutions decreased the tear index from 23.03 to 16.64 Nm $m^2$ $g^{-1}$ when the pulp was beaten with 1,000 and 24,000 beating revolutions, respectively. [23-25]. Notably, hydrogen bonding plays a vital role to control mechanical properties of paper [26, 27]. Higher tensile strength could be obtained from paper with higher hydrogen bonding [21]. Therefore, low values of tensile strength of the paper prepared in this work could be attributed to less hydrogen bonding formed within paper due to a low paper weight of 60 g $m^{-2}$. Moreover, the availability of lignin in fibers might be another factor to hinder the formation of hydrogen bonding between fibers. Recently, cellulose nanofibers have been used to improve mechanical properties of the paper [21, 28]. With increasing a content of cellulose nanofibers, the tensile index of the paper increased due to the formation of the strong hydrogen bonding between cellulose nanofibers and bagasse fibers. However, the lower tear index was obtained when the cellulose nanofiber content in the paper was higher due to a shorter length of cellulose nanofibers.

## 4. Conclusion

The steam explosion process is an efficient green method to fibrillate fibers with smaller widths from bundles for a short period. When the steam pressure was higher, the mean width of the steam exploded fibers was found to be lower due to the removal of hemicellulose and lignin. With the introduction of the pressure of 16 kgf $cm^{-2}$, fibers with widths of 4.65 µm were observed, and the widths of the treated fibers were reduced to 3.78 µm when the steam pressure of 20 kgf $cm^{-2}$ was introduced to the pineapple leaf fibers. Moreover, during the treatment the steam pressure destroyed cellulose molecular chains, resulting in the low degree of polymerization. With the higher steam pressure, the lower degree of polymerization of the steam exploded fibers was obtained. The increase of the cellulose content and the reduction of the hemicellulose and lignin contents were also observed at the higher steam pressure. Mechanical properties of the paper were also affected by fiber morphology. After the steam explosion, individual fibers were separated from fiber bundles. This led to the improvement of the tensile index of the paper. This was attributed to more formation of the hydrogen bonding generated between high surface area fibers. Therefore, fibers treated at the pressure of 20 kgf $cm^{-2}$ could prepare paper with the highest tensile index of 8.18 Nm $g^{-1}$. On the other hand, the decrease of the tear index was observed when the paper was formed from the fibers steam exploded with the higher steam pressure. The paper of the 20 kgf $cm^{-2}$ steam exploded fibers had the tear index of 10.61 Nm $m^2$ $g^{-1}$ while a value of the tear index of 15.87 Nm $m^2$ $g^{-1}$ was found from the paper made from the untreated fibers. The steam exploded fibers prepared in this work would be possibly used in pulp and paper production, and could be useful as a reinforcing phase in the composite industry.

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