Extraction and Characterization of Agave gigantea Fibers with Alkali Treatment as Reinforcement for Composites

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Extraction and Characterization of *Agave gigantea* Fibers with Alkali Treatment as Reinforcement for Composites

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ABSTRACT

The new natural fiber was extracted from *Agave gigantea* leaf as a reinforcement in an environmentally friendly composite material. In this study, the leaves were chemically extracted using alkali treatment with NaOH concentrations of 0%, 2.5%, and 5% to obtain cellulose fibers. The alkali treated fibers were measured using the standard ASTM procedure and observations were carried out through scanning electron microscopy (SEM), Fourier transform infrared (FTIR) characterization was conducted, and the crystallinity index and thermal stability of the fibers were tested. The results showed that there was an increase in cellulose content after the alkali treatment. Furthermore, the SEM observations indicated the presence of hemicellulose and ash within the natural raw fiber bundle, which later broke down into individual cellulose after alkalization. The FTIR characterization showed the removed aromatic group at wavenumber 1245 cm^{-1} after alkali treatment. In addition, the crystallinity of the 5% NaOH alkali-treated fiber was 28% higher than that of the raw fiber. Finally, the thermogravimetric analysis showed that the superior thermal stability of the fiber was 345–363°C. From the results, it is evident that these novel fibers are suitable as reinforcement materials to prepare composites for various applications.

摘要

这种新的天然纤维是从龙舌兰叶中提取出来的，作为一种环保复合材料的增强材料. 在这项研究中，采用碱处理，NaOH浓度分别为0%，2.5%和5%，对叶片进行化学提取，以获得纤维素纤维. 使用标准ASTM程序测量碱处理纤维，并通过扫描电子显微镜 (SEM)进行观察，进行傅立叶变换红外 (FTIR)表征，测试纤维的结晶指数和热稳定性. 结果表明，碱处理后纤维素含量增加. 此外，SEM观察表明，天然粗纤维束中存在半纤维素和灰分，这些纤维在碱化后分解为单独的纤维素. FTIR表征表明，碱处理后在波数1245cm-1处去除了芳香基. 另外，5%NaOH碱处理纤维的结晶度比原纤维高28%. 最后，热重分析表明，该纤维优异的热稳定性为345-363°C. 结果表明，这些新型纤维适合作为增强材料制备各种用途的复合材料.

KEYWORDS

Agave gigantae; alkalization; chemical composition; crystallinity; thermal stability

Keywords

关键词; Gigantae; 巨龙舌兰; Alkalization; 碱化; Chemical composition; 化学成分; Crystallinity; 结晶度; Thermal stability; 热稳定性

Introduction

Currently, the management of non-biodegradable wastes is a crucial issue. The majority of countries in the world have already passed laws banned the use of non-biodegradable materials (Sanjay et al. 2018). Production of biodegradable materials, bio-composites, and biofilms could be a solution to this problem (Asrofi et al. 2020; Ilyas et al. 2019; Mahardika et al. 2019). One of the environmentally friendly materials being developed is a natural fiber. In general, natural fibers can be classified into two

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main sources: protein-based animal fibers and other lignocellulose-based plant fibers. The advantages of plant fibers are abundant availability, low cost, and biodegradable compared to synthetic fibers (Abral et al. 2020a; Madhu et al. 2018). The plant fiber has mainly chemical components of cellulose, hemicellulose, and lignin (Mahardika et al. 2018). However, plant fiber had also other fractions such as pectin, wax, and inorganic salts (Gopinath et al. 2016). Plant fiber can be obtained from leaves, seeds, fruit, roots, stems, and bark of plant trees. The rapid development of the biocomposite industry has caused the need for natural fibers to enhanced dramatically, such as the use of sisal, hemp, cotton, and nettle fibers. Therefore, it is difficult to satisfy the demand for the use of fibers available in the market only. So, finding new sources of plant fiber for reinforcement of bio-composites is also an important responsibility for researchers.

Recently, many researchers investigated the properties of fibers/fabrics extracted from plant bark such as *Acacia arabica, Acacia planifron, Acacia leucophloea, Albiziaamara, Azadirachta indica, Carica papaya, Ceiba pentandra, Cordia dichotoma, Grewia tilifolia, Grewia optiva, Prosopis juliflora, and Pithecellobium dulce* (Manimaran et al. 2018; Senthamaraikannan and Kathiresan 2018; Senthamaraikannan et al. 2018). The *Agave gigantea* (AG) plant which belongs to the Pandanaceae family is the one most suitable candidate materials for reinforced polymer composites. This plant is very abundant in nature, widely available in tropical forests such as in West Sumatra, Indonesia. However, the use of AG fiber for reinforcement of polymer composites is still limited. The unique characteristics of AG fiber were long leaves and yellowish-white leaves and greenish sides with many spines. AG plants can be grown without special treatment such as fertilization. AG fibers have excellent properties in traditional product applications such as decorations, mats, baskets (including for women and for storing valuables), hats, fans, pillows, other woven items. The traditional process of AG fiber was to boil it for several hours and dry it in the sun to increase the toughness of the fiber. After that, the brown AG leaves are ready to be applied to produce traditional products. AG fiber has a lot of cellulose content in the fiber, which can be used as a reinforcement for biocomposite (Khan et al. 2020; Madhu et al. 2020; Narayanasamy et al. 2020; Vijay et al. 2019).

There are various methods for preparing cellulose from natural fibers by chemical treatment and thermal steam explosion (Boonterm et al. 2016). Wang et al. (2016), Neto et al. (2019) and Herlina Sari et al. (2018) prepared cellulose from rice husk cellulosic fibers, jute fabric, sisal fibers, ramie fibers, and corn husk fibers, respectively, by alkalization, silanization, and acid hydrolysis (Herlina Sari et al. 2018; Mariano, Cercená, and Soldi 2016; Neto et al. 2019; Wang et al. 2016). Each method has deficiencies and advantages in terms of the composition and properties of the cellulose fibers (Sanjay et al. 2019).

When alkali treatment is used, the structure and properties, and particularly the size of the cellulose, depend on the source of the pure cellulose and on the preparation process: its concentration, time, and temperature (Herlina Sari et al. 2018; Wang et al. 2016). Cellulose from rice straws prepared by 1, 5, 10, and 15 wt% NaOH solution for 30 min using a fiber to solution ratio of 1:20 by weight, leading to pure cellulose (Boonterm et al. 2016). Although there have been many previous studies reporting the production of pure cellulose for various cellulose sources and methods (Asrofi et al. 2018; Mahardika et al. 2018). Alkali treatment can remove non-cellulose content in fiber (Borchani, Carrot, and Jaziri 2015; Ganapathy et al. 2019). Thus, it can improve the interfacial adhesion between fiber/matrix which will enhance the mechanical properties, physical properties, and thermal stability when used as reinforcement (Mahardika et al. 2019; Neto et al. 2019). The alkali treatment with sodium hydroxide can improve the compatibility of these natural fibers (Boonterm et al. 2016; Ganapathy et al. 2019).

The extraction and characterization (physical and thermal properties) of new natural fibers from *Agave gigantea* leaves have not been previously reported, and the present study was isolated cellulose with various alkali (NaOH) treatments (0%, 2.5%, and 5%). All samples were characterized by chemical analysis, scanning electron microscopy, Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction method, thermogravimetric analysis (TGA), and the results were compared with other natural fibers. Initial data obtained from these characteristics can be used more widely for the manufacture of natural fiber-reinforced composite products.

Materials and method

Materials

The materials used in this study were *Agave gigantea* leaves, sodium hydroxide (NaOH), distilled water, aluminum foil, and plastic clips. *Agave gigantea* (AG) leaves are obtained from the Agricultural Polytechnic, Payakumbuh, West Sumatra, Indonesia. The chemical reagents were purchased from PT Brataco Padang. This work is carried out at the Laboratory of Agricultural Product Technology and Engineering, Mechanical Metallurgy Laboratory, Andalas University, and the Laboratory of Material Physics, Padang State University.

Extraction of cellulose fibers

AG plants were harvested and washed in tap water followed by several times washes. After that, the leaves are soaked in a container with 100°C boiling water for 4 h. The AG fiber was extracted using a stainless plate by scraping the soft part of the leaf surface. Immediately after extraction, the fibers were washed with distilled water to remove unwanted material from the fiber. The AG plants and the extracted fibers are shown in Figure 1.

AG fibers were hydrolyzed with sodium hydroxide in various concentrations of NaOH solution (0%, 2.5%, and 5%) at 80°C for 1 h as shown in Figure 1b. Labeling of samples with various concentrations of NaOH can be seen in Table 1. After the alkali treatment, the cellulose fibers were washed three times until a pH 7 with distilled water to remove any residual alkalization on the AG fibers. The clean fibers were dried by adjusting low air humidity (RH 40%) until a constant weight. The AG fiber was dried in a drying oven for 24 h at 60°C.

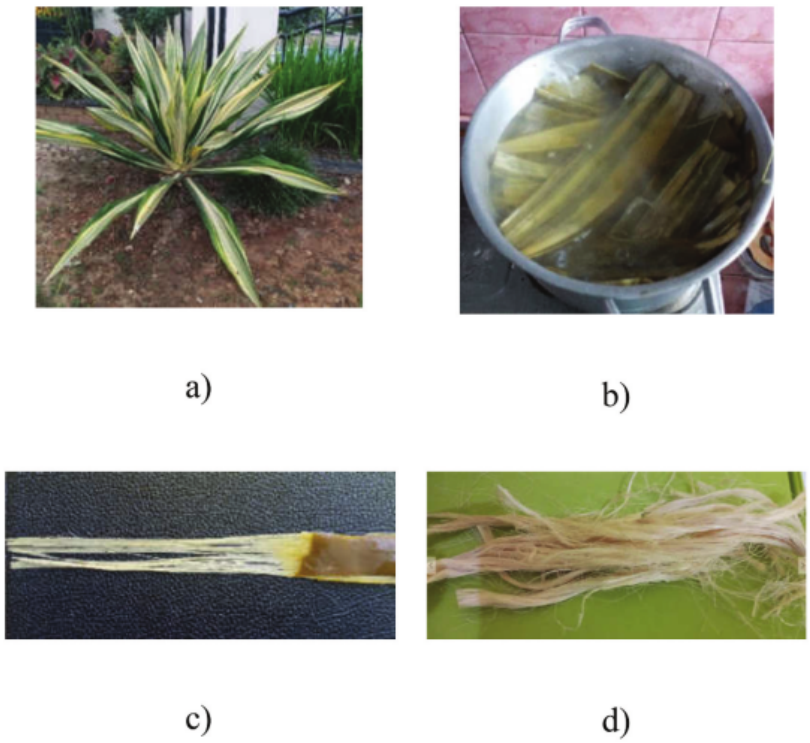

Figure 1. (a) *Agave Ggigantea*, (b) *Agave Ggigantea* leaves, (c) leaves in the retting process, and (d) extracted fibers.

Table 1. Various concentrations of NaOH used in the study.

Sample	Samples code	NaOH (%)
AG fiber	AG-0	0
AG fiber with 2.5% alkali treatment	AG-2,5	2.5
AG fiber with 5% alkali treatment	AG-5	5

Characterization

The effects of various concentrations of NaOH on the chemical composition of the AG fibers before and after treatment were observed. Chemical composition testing was composed of cellulose, and hemicellulose content in AG fibers. American Standard Testing Materials (ASTM) 1104-56 was used to determine the holocellulose content. Holocellulose consists of cellulose and hemicellulose after lignin is removed from the fiber.

The surface morphology of the AG fibers was observed by using scanning electron microscopy (SEM) (JEOL JSM-6510LA). The SEM figure was produced at magnifications between 200× and 500× with 20kV operating voltage. The SEM image was used to measure AG fiber dimensions before and after alkali treament.

ATR-FTIR characterization was used to analyze functional groups in AG fibers before and after chemical treatment. The PerkinElmer FTIR spectrometer (Frontier instrument, USA) were recorded within the wavenumber of 4000-600 cm^{-1} at 4 cm^{-1} resolution. Before characterization, the samples were dried in an oven at temperature 60°C until their weight was constant.

X-ray diffraction (XRD) testing of all the samples was carried out using X'Pert PRO PANalytical instrument (Philips Analytical, Almelo, the Netherlands) to determine the index of crystallinity of AG fiber before and after alkali treatment. XRD testing used CuKα radiation (λ = 0.154) with a 40 kV and 30 mA. The 2θ angle range was tested in 5-60°. The crystallinity index (Icr) was measured using the Segal method (1) (Nam et al. 2016).

$$Icr(\%)=[(I_{200}-I_{am})/I_{200}]x100 \quad (1)$$

where I_{200} is the main peak intensity representing the crystal region at 2θ = 22.2° and I_{am} (2θ = 18.8°) is the intensity for the amorphous content.

Thermogravimetric analysis was performed using the DTG-60 SHIMADZU serial no. C30565000570 (Kyoto, Japan) with a heating rate of 20°C/min. Samples were tested with a nitrogen flow rate of 80 mL/min with a heating rate of 30-550°C.

Result and discussion

Figure 1a shows the physical characteristics of the AG plant, specifically the not stemmed with a short rod around 1 m. The leaves are sword-shaped with 10–15 cm long and 10–14 cm wide. Figure 1b shows the leaves were boiled with distilled water at 100°C for 4 h. Thus, the aims of this process were separated from fiber and leaf flesh using a stainless plate as shown in Figure 1c. Figure 1d shows the clean of AG fibers was obtained. The AG fiber was hydrolyzed with various NaOH (0%, 2.5% and 5% to obtain pure cellulose content). The chemical composition of AG fibers before and after alkali treatment is provided in Table 2.

Table 2. Main chemical composition of AG fibers and crystallinity index (Icr).

Sample	Cellulose (%)	Hemicellulose (%)	Icr (%)
AG-0	71.2	17.5	46.5
AG-2,5	74.6	9.2	62.4
AG-5	76.7	8.8	64.6

The cellulose content of raw fiber is 71.2%, this result higher than *Calotropis* bark fiber (63.6%) (Ramasamy, Reddy, and Rajulu 2018), *Furcraea foetida* fibers (68.4%) (Manimaran et al. 2018), *Albizia amara* fibers (64.5%) (Senthamaraikannan et al. 2018), and *Coccinia grandis* L. fibers (62.4%) (Senthamaraikannan and Kathiresan 2018). After alkali treatment with 2.5% and 5% NaOH, the cellulose content increased by 4.8% and 7.7% compared raw fibers, respectively. Increased cellulose followed by reduced hemicellulose content in the amount of 49.7% after alkali treatment with 5% NaOH. It is due to the hemicellulose–lignin bonds that had been hydrolyzed during the alkalization process as reported in Table 2. The decrease of lignin is also indicated by the increase in cellulose crystals due to the loss of amorphous content according to the XRD test results. These results are consistent with the FTIR characterization, which shows the degradation of hemicellulose (acetyl groups) at 1733 cm^{-1} (Figure 3). Previous studies reported the alkali treatment was effective to remove non-cellulose content in natural fibers (Mahardika et al. 2018; Ramasamy, Reddy, and Rajulu 2018; Syafri et al. 2019).

Figure 2 shows SEM observations of the surface morphology of the AG fibers before and after the alkali treatment with magnifications of 200x and 500x. In Figure 2a the AG fibers are long fiber-bundles with an average diameter of 153.3 µm. Raw fiber displayed a higher section of agglomerated fibers on the tensile fracture surface compared to alkali-treated fibers (Figure 2b). The surface of AG fibers before alkali treatments was indicated of having wax, lignin, and ash, as shown in the yellow arrow in Figure 2a. This is supported by the XRD and chemical composition results with a hemicellulose content 8.8% (see Table 2). The lignin can affect the interface bond between the fiber and polymer matrix (Borchani, Carrot, and Jaziri 2015; Gopinath et al. 2016). Therefore, the hemicellulose–lignin content must be removed to improve the interface bond between the fiber and matrix in the composite.

Figure 2c and d shows the surface morphology of the AG fiber and the tensile fracture surface after 2.5% NaOH alkali treatment. The AG fibers after this process were found to be micro-sized with average dimensions of 213.5 µm. The blue arrow in Figure 2c shows that AG fibers have begun to break down into individual fiber bundles due to reduced hemicellulose–lignin content. The white arrows in Figure 2e show the single-fiber with a smaller diameter of 147 µm after alkali treatment (5% NaOH). The SEM images of the fracture surface of the alkali-treated AG fibers are shown in Figure 2f indicating a clean and rough surface. Similar results were also reported in previous studies (Ganapathy et al. 2019; Herlina Sari et al. 2018; Mahardika et al. 2018).

The structural change of AG fiber before and after alkali treatment was analyzed by FTIR. The FTIR spectrum of all samples is shown in Figure 3. All FTIR spectra were given in absorbance units that tend to be the same. The shifts of peak intensity correspond to structural changes of the fiber (Mtibe et al. 2015). The spectrum for O–H stretching of the cellulose structure at 3309–3332 cm^{-1} (Mtibe et al. 2015). The band at 2900–2907 cm^{-1} indicates the C–H stretching of the cellulose and hemicellulose content (Kassab et al. 2020; Senthamaraikannan et al. 2018). The shift and decreased intensity at 1733 cm^{-1} for carbonyl C–O indicated degradation of hemicellulose (acetyl groups) (Kassab et al. 2020). This result is also consistent with the chemical composition result.

Another band at 1247 cm^{-1} is due to C–O stretching of the characteristic aromatic lignin [9, 18, 25]. Sample AG-0 shows the presence of lignin as shown in Figure 3d. However, after the alkali treatment of 2.5% and 5% NaOH showed the loss of the aromatic group of hemicellulose-lignins. This is a result due to alkaline treatment is effective in hydrolyzing non-cellulose content. These results are consistent with the chemical analysis of fibers and also supported by previous studies (Herlina Sari et al. 2018; Mahardika et al. 2018; Senthamaraikannan and Kathiresan 2018).

XRD curves for AG fiber before and after alkaline treatment with various NaOH samples are shown in Figure 4. The main peak at $2\theta = 22.2°$ shows the characteristic cellulose crystalline region and $2\theta = 18.8°$ indicates amorphous regions (Nam et al. 2016). The crystallinity index of all samples is shown in Table 2. The crystallinity index of AG fibers after 5% NaOH alkali treatment increased by 28% compared to raw fiber. This is due to reduced non-crystalline components such as amorphous, lignin, wax, and other dust content, leading to an increase in the semicrystalline cellulose (Mahardika

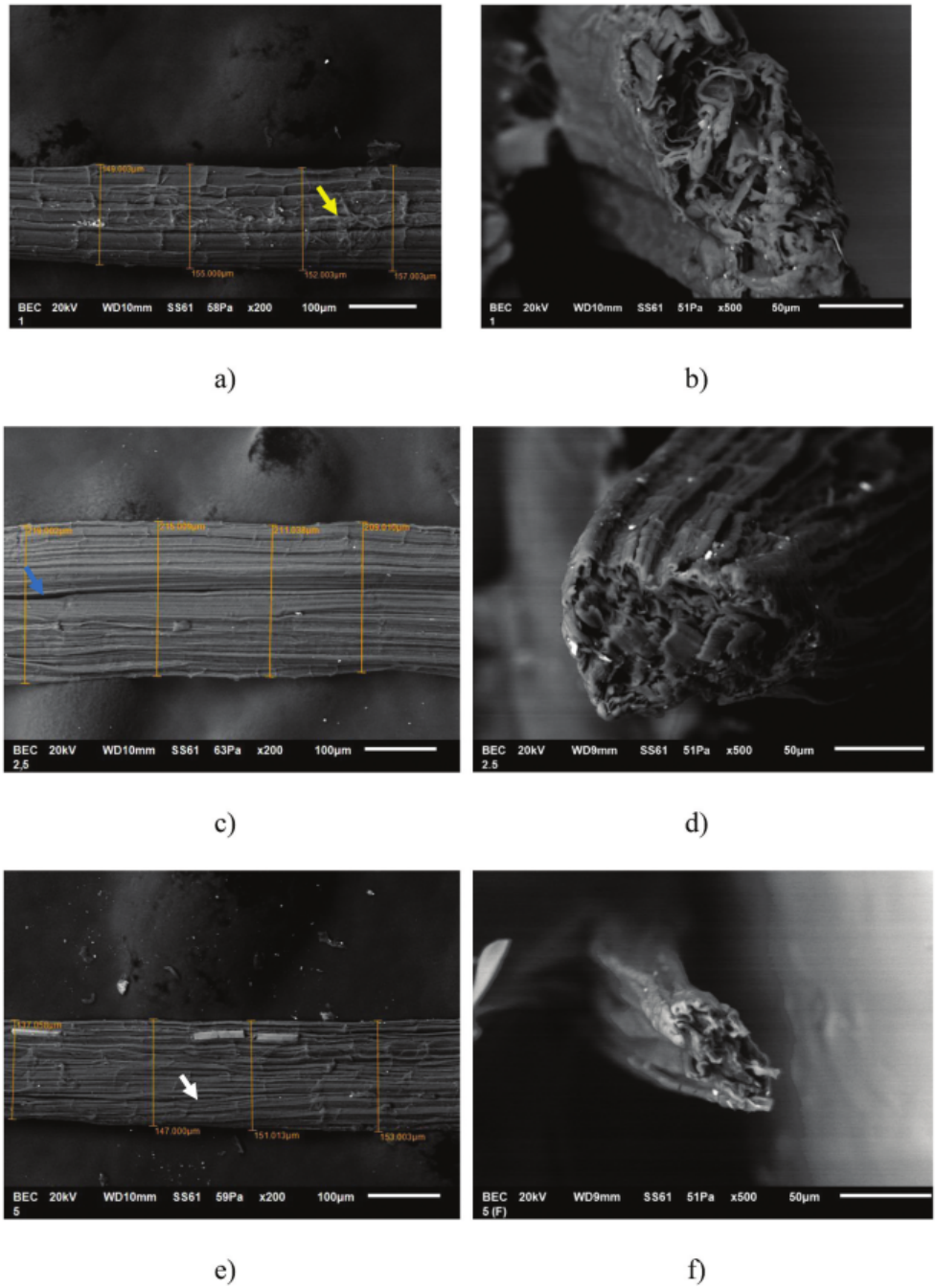

Figure 2. SEM images of AG fiber surface morphology; (a, b) Before alkaline treatment (200× and 500× magnification), (c, d) After 2.5% NaOH alkali treatmented (200× and 500× magnification) and (e, f) After 5% NaOH alkali treatment (200× and 500× magnification).

et al. 2018). The Icr value of this AG fiber is higher than other fibers such as *rhombifolia sida* (56.6%) (Gopinath et al. 2016), *Furcraea foetida* (52.6%) (Manimaran et al. 2018), and *Coccinia grandis* L. (57.6%) (Senthamaraikannan and Kathiresan 2018).

The thermogravimetric analysis curve (TGA) and the derivative of the thermogravimetric (DTG) curves of each tested sample shown in Figure 5a and b. There are three stages of weight loss of the

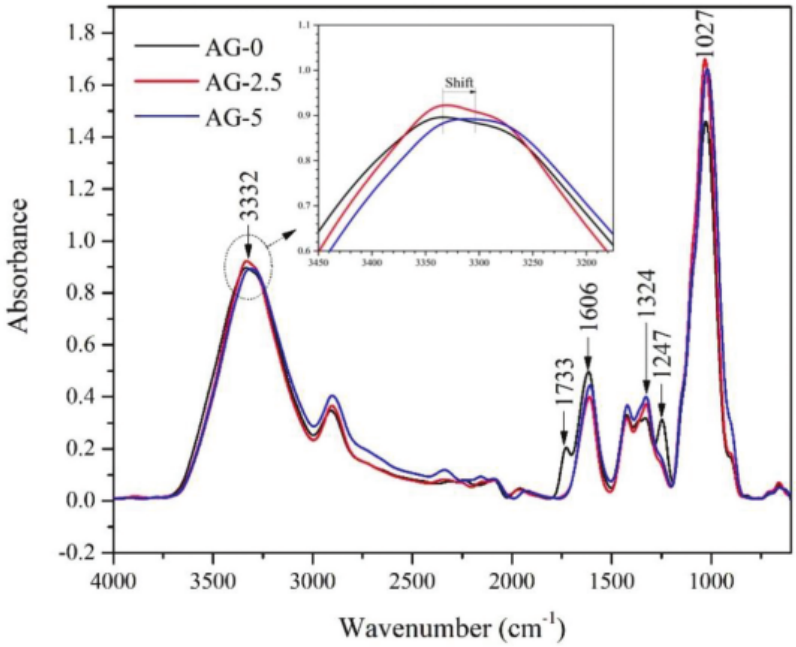

Figure 3. FTIR spectrum of all samples.

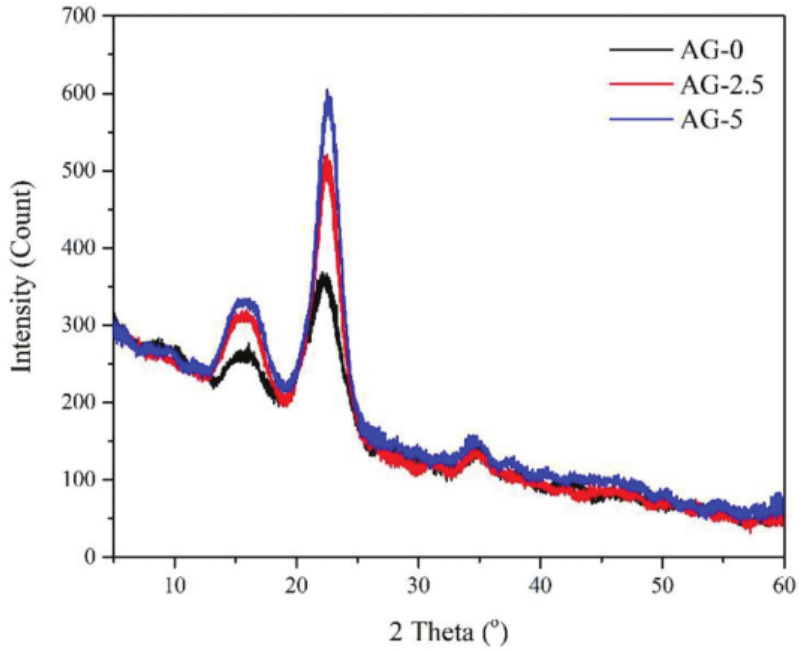

Figure 4. XRD pattern of all samples.

sample shown in a TGA curve. The order of the stages is around 100°C, 250–450°C, and 450–550°C (Abral et al. 2020b). The weight loss of samples at below 100°C due to water evaporation in the fiber (Abraham et al. 2011). These results are also shown on the DTG curve with a peak at that temperature.

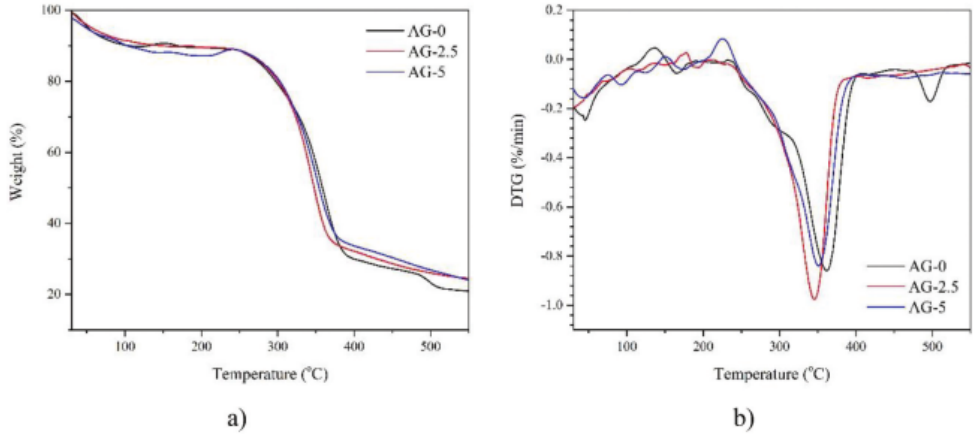

Figure 5. (a) TGA curves and (b) DTG for all samples.

All the samples showed the greatest second weight loss at 250–450°C. This results due to depolymerization and then damage of hemicellulose and cellulose content (Abral et al. 2020b). The depolymerization temperature range of the AG fiber was 345–363°C, as shown in the peak in the DTG curve (Figure 5b). The peak at 363°C had a weight loss rate of 0.9%/min for depolymerization of hemicellulose shown in Figure 5b. In a temperature around 497.2°C, a third weight loss was observed due to final depolymerization to wax, ash, and other substances of AG fibers. Similar results have been observed in previous studies (Abraham et al. 2011; Asrofi et al. 2018; Syafri et al. 2018). The TGA results showed the superior thermal stability of AG fibers with a maximum degradation of 363°C, this result higher than *Furcraea foetida* (320°C) (Manimaran et al. 2018), *Albizia amara* (330.6°C) (Senthamaraikannan et al. 2018) and pineapple fibers (310°C) (Abraham et al. 2011).

Conclusion

In this study, it has been successful in extracting cellulose fiber from AG leaves using alkali treatment for the various concentrations of NaOH. The experimental results showed that the alkali treatment with 5% NaOH produces pure cellulose, namely 76.7%, much higher than other fiber sources. The average diameter of fibers was within the range of 147 μm. The concentration of sodium hydroxide is an important factor due to the change in the properties of cellulose fibers. The high crystalline cellulose includes contributions from alkali treatment with 5% NaOH. These results are consistent with the superior thermal stability of AG fibers. Therefore, we can say that 5% NaOH is the best condition to isolate cellulose from AG fibers using alkali treatment. These results indicate that cellulose from AG fibers has the great potential to be a reinforcement in the biocomposites.

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