

Physicochemical Properties of Pineapple Plant Waste Fibers from the Leaves and Stems of Different Varieties

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Pineapple agro-waste, the residue produced during harvesting or processing activities, is widely available around the world. After harvesting, most pineapple residue is disposed of and serves as fertilizer, or is burnt in an open field. However, these methods are not only ineffective, but also contribute to air pollution. The main objective of this study is to determine the physicochemical properties (*i.e.*, cellulose, hemicellulose, lignin, proximate composition, dry matter, and nitrogen content), of leaves and stems in different varieties (MD2, Moris, and Josapine) of the pineapple plant waste. The data obtained were analyzed using thermogravimetry analysis and proximate analysis. The results showed that the stems and leaves of different varieties exhibit different percentages in lignocellulosic content (hemicellulose, cellulose, and lignin). Proximate analysis showed that nutrient contents were available in the leaves and stems of pineapple plant of different varieties.

Keywords: Physico-chemical properties; Pineapple plant; Waste

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INTRODUCTION

The pineapple (*Ananas comosus* L.) is one of the most essential fruits in the world and is the leading edible member of the Bromeliaceae family. It is an important food crop that is planted extensively in the tropical and sub-tropical regions (Rosnah Shamsudin *et al.* 2009). Commercially, it is mostly sold as canned fruit and is consumed worldwide. The plant can grow up to a height of 75 to 150 cm with a spread of 90 to 120 cm. It is short, having a stout stump with narrow, fibrous, and spiny leaves. The plant develops into a cone-shaped juicy and fleshy fruit with a crown at the top (Tran 2006). According to the FAO online data base, the area of pineapple plantations in 2012 around the world was almost 996,000 ha with an estimated production of more than 23 million tons of pineapple fruit (FAO 2014).

According to the Agrofood Statistics (2010), one hectare of pineapple field can produce about 17,400 fruits, which is approximately 25 metric tonnes of pineapple fruit. In Brazil, based on a rough estimate made in 2002, there are 40 leaves per plant with each leaf weighing about 0.065 kg and 2% fiber per leaf. The total fiber production based on 1.22 ton/hectare would be about 74,528.16 tons; considering that the fiber price is US\$ 0.36/kg, the market value of fiber is US\$ 434 per hectare (Satyanarayana *et al.* 2007).

There have been very few studies on the fibers of the pineapple plant (leaves and stems). There are many varieties of pineapple plant, and each variety is different in terms of characteristics. The pineapple plant is considered not profitable after the fruit has been

harvested because there is a lack of knowledge about the potential of the plants after the fruit is discarded.

After harvesting activities, most of the pineapple residue is disposed and either serves as fertilizer or is burnt in an open field. However, these methods are not only ineffective, but also contribute to air pollution (Wan and Zainuddin 2013). Thus, one of the possible ways to handle pineapple residue without jeopardizing or sacrificing the quality of the environment is by converting this residue into a value-added product. An innovative approach will not only help generate additional income, but will also create job opportunities (Ahmed *et al.* 2002). The plantation sector around the world has generated large amounts of waste plant, thus creating problems for the environment and affecting the next cultivation of plants.

Plant waste fibers, or agro-wastes, can be described as lignocellulosic materials comprising cellulose, hemicellulose, and lignin. Woods, agricultural wastes, water plants, grasses, and other plant substances are the example of the lignocellulosics materials (Abdul Khalil *et al.* 2006). Properties, composition, and structure of plant waste fibers make them appropriate for uses such as composite, textiles, and pulp and paper manufacture. Furthermore, plant fibers can be used to produce fuel, chemicals, enzymes, and food. Biomass, including agricultural crops and residue, forest resources, animal and municipal wastes, is the largest source of cellulose in the world. Organic plant wastes such as palm oil, pineapple, banana, and coconut fiber are annually renewable, available in abundance, and cheap. These lignocellulosic byproducts could be a principal source of fibers, chemicals, and other industrial products (Reddy and Yang 2005).

After the harvesting period, these waste materials create significant environmental problems. Therefore, the economic utilization of these fiber wastes would be beneficial. Although a lot of work and research in agriculture has been undertaken, attention to agricultural technology in fiber applications has been limited and inadequate. Researchers should conduct more studies to assess the potential of agricultural wastes (Reddy and Yang 2005).

The main objective of this study was to determine the physicochemical properties of pineapple plant waste (leaves and stems) from various plant varieties such as MD2, Moris, and Josapine. The properties to be studied should include cellulose, hemicellulose, lignin, crude protein, crude fiber, ash, fat, moisture, carbohydrate, dry matter, and total nitrogen content. These research findings can lead to a better understanding and knowledge of the physico-chemical compositions of the fibers themselves. Information is important in order to minimize the environmental and health risk associated with disposal of pineapple plant in the field. Hence, findings from this study can be used by the polymer chemist, scientist, and food technologist for further applied research.

EXPERIMENT

Materials

Three different samples from a variety of pineapple crops (Josapine, MD2, and Moris) were collected at Pineapple Plantation, MAEPS, Serdang, Selangor at longitude:2° 58' 39.882" and latitude:101° 41' 27.0132". The pineapple plant was taken right after the fruit was harvested. One kilogram of sample from the leaves and stems of different varieties of pineapple plant were picked randomly and simultaneously. The pineapple leaves and stems were manually chopped and then washed with tap water. According to the method

used by Steyn (1959), after washing, the materials were cut down to 2 to 3 cm and dried at 60 °C to preserve the nutritional content of the materials. The stems and leaves were separated and the drying process took about 72 h using an oven (OF-G22W, Jeio Tech, Korea). The samples were ground and sieved into 1-mm particle size using a Mill Grinder (Retsch, SM200 Rostfrei, Germany). After grinding, both samples were dried further, for 24 h at 60 °C. The dried samples were kept in a refrigerator for further analysis.

Methods

Determination of hemicellulose, cellulose, and lignin contents

The hemicellulose, cellulose, and lignin contents were determined by using a Thermogravimetric Analyzer (TGA) (Mettler Toledo, TGA/SDTA851^e, USA). Approximately 10 mg of oven-dried sample was placed in an alumina ceramic crucible and weighed. The TGA was programmed starting at 30 to 600 °C at a rate of 10 °C/min under 10 mL/min N₂ purging. The temperature was then further increased to 900 °C at the same rate, but purged with 10 mL/min air (Rozita Omar *et al.* 2011). All data analysis was taken from the graph.

Determination of dry matter content

Dry matter represents everything contained in a sample except water; this includes protein, fiber, fat, and minerals. In practice, it is the total weight of the sample minus the weight of water, expressed as a percentage. In this study, it was determined by drying the sample in an oven (OF-G22W, Jeio Tech; Korea) until the sample reached a stable weight. The sample was weighed and about 1 g placed in a dry crucible. The crucible and sample were oven-dried for 24 h. After that, the crucible and sample were weighed. The percentage of dry matter was calculated and recorded (Nennich and Chase 2007). Equation 1 is as follows:

$$\text{Dry Weight/Total Weight} \times 100 = \% \text{ Dry Matter} \quad (1)$$

Determination of moisture content

Moisture content was determined using the Standard Official Methods of Analysis of the AOAC (1990). This involved drying to a constant weight at 105 °C and calculating moisture as the loss in weight of the dried samples. The crucible was thoroughly washed and dried in an oven at 100 °C for 30 min and allowed to cool inside desiccators. After cooling, they were weighed using a weighing balance and their various weights were recorded as (W1). Then, 2.0 g of the finely-ground samples were put into the crucibles and weighed to determine W2. Thereafter, the sample and crucible were placed inside the oven and dried at 100 °C for 4 h, then cooled and weighed at the same temperature for 30 min until constant weights were obtained to get W3. Then, the moisture content of the samples was calculated from Eq. (2):

$$\frac{(\text{Initial weight of filled crucible}) - (\text{Final weight of filled crucible})}{(\text{Initial weight of filled crucible}) - (\text{Initial weight of empty crucible})} \times 100\% \quad (2)$$

Determination of ash content

Total ash content of the samples was determined using furnace incineration, as described by AOAC (1990), based on the vaporization of water and volatiles with burning organic substances in the presence of oxygen in the air to carbon dioxide at a temperature

of 550 °C (dry ashing). About 1.0 g of finely-ground dried sample was placed in a porcelain crucible and incinerated at 525 °C for 6 h in an ashing muffle furnace (KSL-1700X, MTI Corporation; USA) until ash was obtained. The ash was cooled in a desiccator and weighed. The percentage of ash content in the samples was calculated as:

$$(\text{Weight of ash}) / (\text{Weight of original}) \times 100 = \% \text{ Ash} \quad (3)$$

Determination of crude fiber content

Crude fiber content was determined using the method described by AOAC (1990). The crucible was dried in an oven for about 1 h at 105 °C, then cooled in a desiccator. The weight of the crucible was calculated. About 1.0 g of the samples and 1.0 g of filter agent using Celite 545 diatomaceous earth (Sigma Aldrich, Germany) were dissolved in 200 mL of boiling 0.25 N sulfuric acid using fibertec analysis (Fibertec™ 2010, Foss Analytical; Denmark) and boiled for 30 min. The hydrolyzed mixture was filtered through the crucible and the residue was rinsed with boiled distilled water to remove the acid from the filtrate inside the crucible. Again, 200 mL of boiled 0.313 N sodium hydroxide (NaOH) was added to the crucible and boiled for 30 min. The hydrolyzed samples were filtered again, and the residue was rinsed with boiled distilled water until the crucible was free of alkaline. The residue was rinsed again with a small amount of acetone and then drained. The residue in the crucible was dried in the oven at 105 °C until a constant weight was achieved. The crucible was placed in the muffle furnace at 550 °C and was burnt completely (Meloan and Pomeranz 1980). The crucible was then placed in the desiccator until a constant weight was achieved and calculated as:

$$(\text{Weight of residue without ash}) / (\text{weight Sample}) \times 100\% = \% \text{ Crude Fiber} \quad (4)$$

Determination of crude protein and nitrogen contents

The crude protein content of the samples was determined using the Kjeldahl method of AOAC (1990), which involved protein digestion and distillation. For the protein digestion, about 2.0 g of the sample was weighed into a Kjeldahl flask, and 2 tablets of Kjeldahl Catalyst were added. This was followed by adding 25 mL of concentrated sulfuric acid. The whole mixture was subjected to heat in the fume cupboard. The heating was done gently at first and increased with occasional shaking until the solution acquired a green color. The temperature of the digester remained above 420 °C for about 30 min. The solution was cooled and black particles found at the neck of the flask were washed down with distilled water. The solution was re-heated gently at first until the green color disappeared. Then, it was allowed to cool.

To prepare for protein distillation, the Kjeltac distillation apparatus (Kjeltac™ 2300, Foss Analytical; Denmark) was steamed through for 15 min, after which a 100-mL conical flask containing 5 mL of boric acid/indicator was placed under the condenser so that the condenser tip was under the liquid. About 5.0 mL of the digest was pipetted into the body of the apparatus *via* a small funnel aperture. The digest was washed down with distilled water, followed by the addition of 50 mL of 60% NaOH solution. The digest in the condenser was steamed through for about 1 to 5 min, after which enough ammonium sulfate was collected. The receiving flask was removed and the tip of the condenser was washed down into the flask, after which the condensed water was removed. The solution in the receiving flask was treated with 0.01 M hydrochloric acid. Also, a blank was run

through along with the sample (James 1995). After titration, the percentage of nitrogen was calculated using Eq. 5,

$$\% N_2 = (V_1 - V_2) \times (\text{molarity of acid}) \times 0.01410 \times (W) \times 100\% \quad (5)$$

where V_1 is the volume of acid used in the titration, V_2 is the corresponding amount of acid for the blank titration, and W is the weight of the sample.

On average all biological proteins contain 16% N; therefore protein content is estimated by multiplying N% by 6.25 (6.25 is the reciprocal of 0.16). Thus, crude protein does not differentiate between N in feed samples coming from true protein or other nonprotein nitrogen (NPN) compounds, nor does it differentiate between available and unavailable protein.

$$\% \text{Nitrogen} \times 6.25 = \% \text{Crude Protein} \quad (6)$$

Determination of crude fat content

The total fat in the sample was determined according to the Soxhlet extraction method using Soxtec Extraction (Soxtec™ 2050, Foss Analytical, Denmark). First, a 250-mL clean aluminum cup was dried in an oven at 105 to 110 °C for about 30 min and cooled in a desiccator. Approximately 1.0 g of sample was weighed into labeled thimbles. The aluminum cup was weighed correspondingly and filled with about 80 mL of petroleum ether (boiling point 40 to 60 °C). The extraction thimbles were plugged tightly with cotton wool. The Soxtec apparatus was assembled and allowed to reflux for 75 min. The thimble was removed with care, and petroleum ether was collected from the top container and drained into another container for re-use. After that, the flask was dried at 105 to 110 °C for 1 h, when it was almost free of petroleum ether. After drying, it was cooled in a desiccator and weighed (Pearson 1976). Then, the fat percentage of the samples was computed using Eq. 7:

$$(\text{Weight of fat}) / (\text{Weight of sample}) \times 100 = \% \text{fat} \quad (7)$$

Determination of carbohydrate content

The total percentage of carbohydrate content in the pineapple plant sample was determined using the difference method as reported by Onyeike *et al.* (1995). This method involves adding the total values of crude protein, lipid, crude fiber, moisture, and ash constituents of the sample and subtracting it from 100. The value obtained is the percentage of carbohydrate constituent of the sample. Thus:

$$100 - (\% \text{Moisture} + \% \text{Crude Fiber} + \% \text{Protein} + \% \text{Fat} + \% \text{Ash}) = \% \text{Carbohydrate} \quad (8)$$

Statistical analysis

Microsoft Excel and SAS 9.0 system (SAS Institute Inc., Cary, NC, USA) was used to analyze the data for the mean, standard error, and least significant difference test (LSD) ($P < 0.05$) to compare differences among percentages of the lignocellulosic and proximate analysis of the material.

RESULTS AND DISCUSSION

Determination of Thermal Decomposition Temperature of Hemicellulose, Cellulose, and Lignin

Based on a study by Md. Yunos *et al.* (2012), the thermal degradation of components in the samples occurred in three stages. The first stage is moisture removal from samples, the second stage is hemicelluloses decomposition, and the third stage took place when cellulose and lignin degradation occurred. All the stages occurred at a certain temperature range between minimum 0 °C to maximum temperature, 900 °C. According to Yang *et al.* (2007), hemicellulose is the easiest component to be thermally decomposed, followed by cellulose and hemicellulose. The degradation for hemicellulose and cellulose occurred in the range 220 to 315 °C and 315 to 400 °C, while lignin degradation occurred within a higher range 160 to 900 °C. Figures 1 and 2 show the TGA and DTG curves for various pineapple plant including the leaves and the stem fibers.

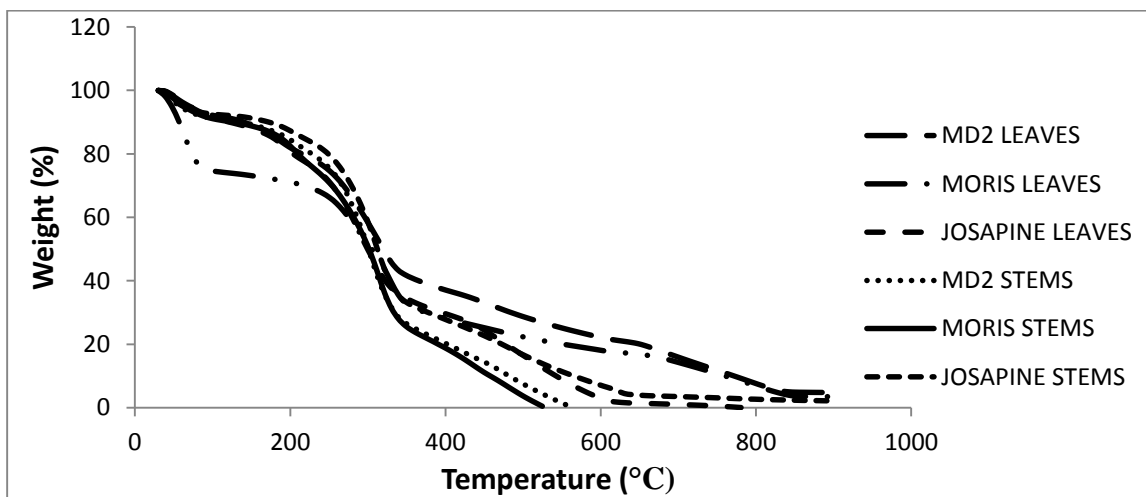


Fig. 1. Thermogravimetric analysis (TGA) curves for pineapple leaves and stems at different cultivars

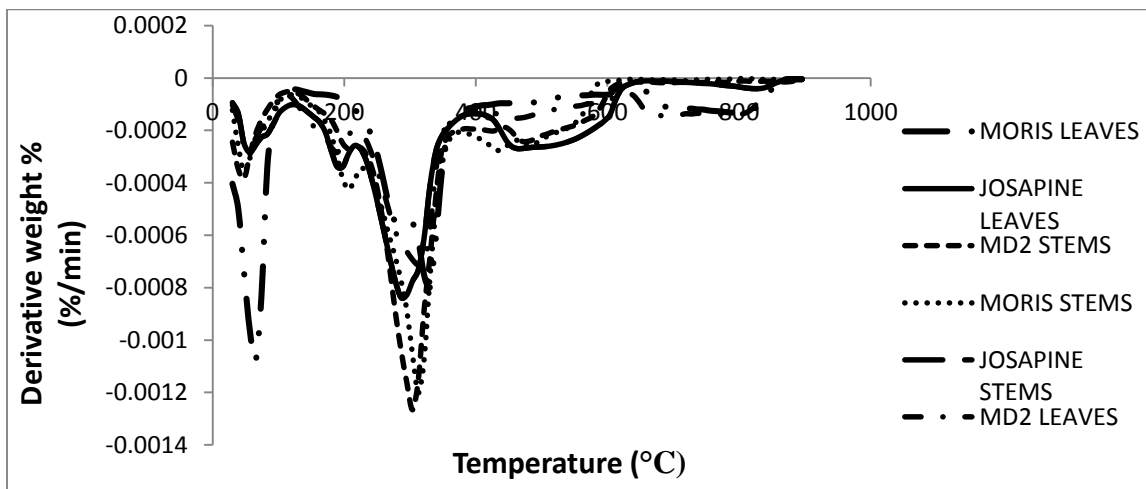


Fig. 2. Derivative thermogravimetric (DTG) curves for pineapple leaves and stems at different cultivars

The percentages of hemicellulose, cellulose, and lignin in the fiber were observed based on the weight loss (%) at these temperatures. The first stage of decomposition was the evaporation of moisture where the weight loss of fibers occurred at temperature ranging from 30 °C to 130 °C. The mass loss varied between 5% to 10% for the leaves and stems at different pineapple cultivar. For the next stage, decomposition on hemicellulose, cellulose and lignin took place within the range of 150 to 900 °C.

Hemicellulose

As noted by Yang *et al.* (2007), hemicellulose decomposes first, followed by cellulose and lignin. The degradation for hemicellulose and cellulose occurred within the ranges of 220 to 315 °C and 315 to 400 °C, while lignin degradation occurred within the range 160 to 900 °C. From the TGA and DTG curve, the second phase consisted of hemicellulose decomposition. For the plant fiber including leaves and stems, hemicelluloses start to decompose at temperature as low as 150 °C, for leaves from Moris cultivar, but, stems from MD2 cultivar, decomposed at the highest temperature, 304 °C. According to Wang *et al.* (2009), hemicelluloses are the easiest components to be thermally broken down and are easily volatilized at relatively low temperature due to their amorphous structure, which is rich in branches and consists of various saccharides that appear in a random organization. From Fig. 1, it was estimated that the leaves from the Josapine variety had the highest percentage of hemicelluloses (14%), followed by the MD2 variety (12%), and Moris (11%). Stems for the MD2 variety showed the highest value compared to the others, which was 24% followed by Moris (16%) and Josapine (12%). The hemicellulose content of the pineapple plant in the stems and leaves of different cultivars is comparable with that of jute (14 to 16%), ramie (13%), and sisal (12%), but is lower than that of barley straw (38%) and corn stover (33%) (Han and Rowell 1996). Moreover, according to Paster *et al.* (2003), higher hemicellulose content would be preferable for producing ethanol and other fermentation products because hemicellulose is relatively easy to hydrolyze into fermentable sugars.

Cellulose

According to Paster *et al.* (2003), cellulose is the main structural component providing strength and stability to the plant cell walls and fiber. The amount of cellulose in a fiber influences its properties, the economics of fiber production, and the utility of the fiber in various applications. Figure 2 shows that cellulose degraded between the temperature at 291 to 328 °C. Compared to hemicelluloses, cellulose is more thermally stable because of its crystalline nature in which it is bonded together by hydrogen bonds to form microfibrils (Alwani *et al.* 2014). From the TGA curve, it was estimated that the fibers with the highest percentage of cellulose were stems from MD2 cultivar (67%), followed by stems from Moris and Josapine cultivar, 51% and 48%, respectively. From a previous study by Abdul Khalil *et al.* (2006), pineapple leaf was found to be high in cellulose content, which was 74.33%. However, from this research, the pineapple stems were found to have high cellulose content compared to the leaves part. This is probably related to the relatively higher weight of the fruit it supports and the fact that it is less perishable than other part of plants (Reddy and Yang 2005). TGA curves showed that the leaves from Moris contain about 46% of cellulose, followed by Josapine and MD2, 45% and 41%, respectively. Each of the cultivars showed different percentage of cellulose, which might be due to variation in age, type, and plantation of the pineapple plant (Rozita *et al.* 2011).

Fibers with higher cellulose content would be preferable for textile, paper, and other fibrous applications. The value of the pineapple plant waste and its potential application is not determined by cellulose only, but it should be noted that the value also depends on the quality of the fibers obtained, both from the pineapple leaves and stems at different cultivar.

Lignin

Lignin is a highly crosslinked molecular complex with an amorphous structure and acts as glue between individual cells and between the fibrils that form the cell wall (Mohanty *et al.* 2000). Lignin provides plant tissue and individual fibers with compressive strength and strengthen the cell wall of the fibers to protect the carbohydrates from chemical and physical damage (Saheb and Jog 1999). Lignin was the most difficult component to decompose compared to other components, where the decomposition occurred slowly, starting from 160 °C and extending to 900 °C (Yang *et al.* 2007; Alwani *et al.* 2014). The DTG curves showed that the decomposition of the lignin occurred in a wider temperature range compared to hemicelluloses and cellulose. However, the peak at DTG curve showed that lignin decomposition occurred especially within the range of temperature between 417 to 816 °C. The lignin content was observed from TGA graph, and the percentage of lignin was estimated to be around 12 to 18% for the pineapple leaves and 20 to 24% of lignin for pineapple stems for different cultivars. According to Abdul Khalil *et al.* (2006), the lignin content in pineapple leaf was found to be 10.41%, which is less, compared to the results from this experiment. However, it was still lower than hardwood and softwood percentages, which was 14 to 34% and 21 to 37%, respectively. From the result obtained, lignin was found to be highest in the stems from all cultivars, compared to the leaves. The stem actually is the rigid part of the pineapple plant, thus it contains the highest percentage of lignin. One of the factors that impacts the lignin concentration and composition of forages is the plant maturation process. During the process, accumulation of stem mass exceeds leaf mass addition. Stems contain a higher proportion of thick-walled tissues (sclerenchyma, xylem fiber, and xylem vessel) and less photosynthetic tissues (mesophyll, chlorenchyma) than found in leaves, resulting in stems having a higher lignin concentration than the leaves (Jung 2012). Lignin is often viewed as a waste product because of problems in its structural diversity and heterogeneity, which pose challenges to deconstruction. Despite these challenges, lignin contains structural units that could serve as a source of fuels and high-value for chemical production (Mendu *et al.* 2011).

Table 1. Proximate Composition of the Leaves of Different Varieties of Pineapple Plant

Constituents	Proximate Analysis (%)		
	MD2	Moris	Josapine
Moisture	7.87 ± 0.71 ^a	7.23 ± 1.12 ^a	9.42 ± 0.02 ^a
Ash	2.35 ± 1.25 ^a	2.08 ± 0.23 ^a	2.11 ± 0.83 ^a
Crude fiber	30.93 ± 0.25 ^a	31.03 ± 0.25 ^a	31.04 ± 0.16 ^a
Crude protein	5.82 ± 1.31 ^a	7.05 ± 1.12 ^a	5.64 ± 0.41 ^a
Crude fat	3.05 ± 0.25 ^a	2.53 ± 0.13 ^a	3.15 ± 0.30 ^a
Carbohydrate	34.57 ± 2.94 ^a	33.44 ± 1.34 ^a	33.31 ± 0.14 ^a
Dry matter	92.50 ± 0.50 ^a	92.77 ± 0.30 ^a	90.38 ± 0.00 ^a
Total nitrogen	0.93 ± 0.21 ^a	1.13 ± 0.18 ^a	0.91 ± 0.07 ^a

*Means (± SD) with the same letter are not significantly different at p > 0.05 for each row

Table 2. Proximate Composition of the Stems of Different Varieties of Pineapple Plant

Constituents	Proximate analysis (%)		
	MD2	Moris	Josapine
Moisture	8.78 ± 1.03 ^a	10.79 ± 0.79 ^a	8.02 ± 0.05 ^a
Ash	1.24 ± 1.04 ^a	3.55 ± 0.03 ^a	4.07 ± 0.90 ^a
Crude fiber	37.63 ± 0.63 ^b	39.88 ± 0.63 ^{ab}	41.75 ± 0.75 ^a
Crude protein	2.30 ± 0.20 ^a	4.00 ± 1.19 ^a	3.20 ± 0.90 ^a
Crude fat	2.71 ± 0.01 ^b	3.80 ± 0.20 ^{ab}	2.55 ± 0.05 ^b
Carbohydrate	47.36 ± 2.49 ^a	38.27 ± 0.58 ^b	40.15 ± 0.57 ^{ab}
Dry matter	89.50 ± 3.50 ^a	89.21 ± 0.53 ^a	91.98 ± 0.30 ^a
Total nitrogen	0.37 ± 0.03 ^a	0.52 ± 0.15 ^a	0.64 ± 0.19 ^a

*Means (± SD) with the same letter are not significantly different at p > 0.05 for each row

Moisture

The results of the proximate analysis of leaves and stems from varieties of pineapple plant are presented in Tables 1 and 2. The moisture content of the leaves (dry basis) for Josapine showed the highest percentage at 9.42 ± 0.02%, followed by MD2 (7.87 ± 0.71%) and Moris (7.23 ± 1.12%). The percentage of moisture content for stems (dry basis) was the lowest at 8.02 ± 0.05%, and the highest was for the Moris cultivar (10.79 ± 0.79%). The result obtained for the moisture content of the leaf fiber and stems fiber were slightly lower than those reported by Mohanty *et al.* (2000), which was 11.8%. The results of moisture content between the leaves and stems of different pineapple cultivar showed no significant difference (p > 0.05), as seen in Tables 3 and 4. Therefore, the average of the moisture content for all the pineapple cultivars did not exceed 13%, thus making this material properly safe for long-term storage (Kaliyan and Morey 2006).

Ash

Ash is a residue that contains the inorganic mineral elements of a feed sample, and its contents are determined in a laboratory by burning the sample at a high temperature (removing the organic matter) and weighing the residue. Ash content for the leaf samples of three different varieties provided almost the same value, MD2 (2.35 ± 1.25%), Josapine (2.11 ± 0.83%), and Moris (2.08 ± 0.23%), but there is a high percentage value for the stems of all the varieties. Stems from the Josapine cultivar showed the highest percentage

($4.07 \pm 0.90\%$) and the lowest were obtained from the MD2 cultivar ($1.24 \pm 1.04\%$). Percentage values from both the leaves and stems of different cultivars showed no significant difference ($p > 0.05$), as shown in Tables 1 and 2. The results obtained for ash are comparable with the findings of Wan Nadirah *et al.* (2012), which was 4.73% for leaf fiber. The stems showed high value for ash because of the soil contamination during the process of harvesting of plant material (Hoffman 2005).

Crude Fiber

Crude fiber can be defined as carbohydrates that are not digestible by mammalian enzymes but that can be digested by rumen microorganisms. Fiber consists of cellulose, hemicellulose, lignin, and other soluble fibers (Parish 2007). Based on the findings shown in Tables 1 and 2, materials from the leaves and stems from different cultivars contain a high percentage of crude fiber since they have a high percentage of cellulose, hemicellulose, and lignin. The results obtained for crude fiber in leaves varied from $30.93 \pm 0.25\%$ to $31.04 \pm 0.16\%$, while for stems, crude fiber content was between $37.63 \pm 0.63\%$ to $41.75 \pm 0.75\%$. The results from Table 1 showed no significant difference ($p > 0.05$), while the pineapple stems in different varieties showed a significant difference ($p < 0.05$). The differences of crude fiber content in each of the variety were varied because the age of maturity for the each of cultivar is different. As the plants became older, the crude fiber tended to increase. The crude fiber content of pineapple stems is comparable with that of oil palm trunks (OPT), oil palm fronds (OPF), and palm press fiber (PPF), which contain about 37.6%, 38.5%, and 41.2% crude fiber, respectively (Alimon and Zahari 2012).

Crude Protein

Crude protein comprises both true protein and non-protein nitrogen. True protein is sometimes called “natural protein.” It is either degradable or undegradable. Non-protein nitrogen compounds can supply nitrogen to the rumen microbes, then build microbial protein in the rumen (Parish 2009). Based on these findings, the crude protein content for the leaves and stems varied between $5.82 \pm 1.31\%$ and $7.05 \pm 1.12\%$ and between $2.30 \pm 0.20\%$ and $4.00 \pm 1.19\%$, respectively. The minimum value was found in MD2 stems, $2.30 \pm 0.28\%$ and the maximum value was found in Moris leaves ($7.05 \pm 1.12\%$). None of the results from Tables 1 and 2 showed significant difference ($p > 0.05$). Crude protein content is very different across feeds, but within a feed, higher protein is usually associated with higher quality. As forages mature, their crude protein is diluted with increasing fiber content. According to Alimon and Zahari (2012), the crude protein from palm oil waste, which is palm kernel cake (PKC), shows a high value of 17.2% crude protein, but the lowest value was found in oil palm trunks (2.8%).

Crude Fat

Crude fat is an estimate of the total fat content of feed and includes true fat (triglycerides) as well as alcohols, waxes, terpenes, steroids, pigments, esters, aldehydes, and other lipids. From this research, the results for pineapple leaves varied between $2.53 \pm 0.13\%$ to $3.15 \pm 0.30\%$ and for pineapple stems varied between $2.55 \pm 0.05\%$ to $3.80 \pm 0.20\%$. Forages are usually low in crude fat, which is less than 5% (DM), and grains may be slightly higher up to 10% (DM), and Palm Kernel Expeller (PKE) is typically 7 to 10% (DM) (Calvert *et al.* 2012). The results from the leaves of different cultivars showed no significant difference ($p < 0.05$), while the results from stems show a significant difference

($p > 0.05$), as seen in Tables 1 and 2. This is because the pineapple cultivar were at different stages of maturity, and this factor affected the biological nature of the pineapple plant.

Carbohydrates

Carbohydrate content is derived from the different percentages of the proximate analysis, including moisture content, crude protein, crude fiber, crude fat, and ash. Plant carbohydrates may be conveniently classified as structural (or cell wall) carbohydrates and non-structural (or cell contents) carbohydrates. The structural carbohydrates are dominated by cellulose and the hemicelluloses, and these polymers form the basis of fibre in all plant tissue. Leaves from the MD2 cultivar showed a high percentage of carbohydrates ($34.57 \pm 2.94\%$), followed by Moris ($33.44 \pm 1.34\%$) and Josapine ($33.31 \pm 0.14\%$). As seen in Table 2, the stems from the MD2 cultivar had the highest percentage of carbohydrates ($47.36 \pm 2.49\%$). The lowest carbohydrate content for pineapple stems were found in the Moris cultivar ($38.27 \pm 0.58\%$), followed by Josapine ($40.15 \pm 0.57\%$). The results from Table 1 show no significant difference ($p < 0.05$), but the pineapple stems at different varieties showed a significant difference ($p > 0.05$). This might be due to the effect of percentage of crude fat and fiber since the calculation involved both items.

Dry Matter

The dry-matter accumulation at harvest differed widely among pineapple cultivars. According to Hanafi *et al.* (2009), dry matter accumulation occurred in both leaves and stems in the pineapple plant, about 48.5% and 21.6% of dry matter. A similar result was also reported by Hanafi and Halimah (2004), where dry matter accumulations were estimated about 45.0% in leaves and 16.0% in stems. However, the results were different in this study, where the greatest value ($92.50 \pm 0.50\%$) was obtained from the MD2 leaves and the smallest value ($90.38 \pm 0.00\%$) was obtained from the Josapine leaves. The results obtained from the stems varied between $89.21 \pm 0.53\%$ to $91.98 \pm 0.30\%$, and it was higher than those reported by Hanafi *et al.* (2009) and Hanafi and Halimah (2004). From Tables 1 and 2, the result from the leaves and stems of pineapple at different cultivar shows no significant difference ($p < 0.05$) among them. The variation in the proportion of dry matter among pineapple parts and cultivars could possibly be due to the environmental conditions or to inherent differences between cultivars. Another factor is the various interactions of weather on crop growth and yield which may affect dry matter content (Hanafi *et al.* 2009). In conclusion, the results show that dry matter is high, thus less moisture is present, and there is high nutrient density in the sample.

Total Nitrogen

The results show that the percentage of total nitrogen of leaves varied between $0.91 \pm 0.07\%$ to $1.13 \pm 0.18\%$. For the stems, the total nitrogen varied between $0.37 \pm 0.03\%$ to $0.64 \pm 0.19\%$. Leaves from the Moris cultivar exhibited the highest percentage ($1.13 \pm 0.18\%$), followed by MD2 ($0.93 \pm 0.21\%$) and Josapine ($0.91 \pm 0.07\%$). Stems from Josapine cultivar had a higher percentage of total nitrogen ($0.64 \pm 0.19\%$) and the lowest total nitrogen percentages for stems were from MD2 ($0.37 \pm 0.03\%$). There was no significant difference ($p < 0.05$) for pineapple leaves and stems of different cultivar, as shown in Tables 1 and 2. These results were different from a study by Hanafi *et al.* (2009), where stems from Moris contained $1.55 \pm 0.08\%$ total nitrogen and stems from Josapine cultivar contained a high percentage of total nitrogen, $2.27 \pm 0.18\%$. Consequently, total nitrogen contents in previous studies showed a high percentage in leaves for Josapine and

Moris cultivars that were 12.80 ± 0.34 and 10.42 ± 0.26 compared with the findings in this study. The results for total nitrogen indicate that stems have a lower total nitrogen content compared with the leaves of the pineapple plant in different cultivars. According to LaDon *et al.* (1980), nitrogen levels in the soil can affect much of the nitrogen fractions in the plant.

CONCLUSIONS

1. The lignocellulosic content (hemicelluloses, cellulose, and lignin) and proximate analysis (moisture, ash, crude protein, crude fat, crude fiber, carbohydrate, dry matter, nitrogen content) in different pineapple cultivars (MD2, Josapine, and Moris) were studied.
2. Based on the results obtained, it can be concluded that the percentage of cellulose is the highest, followed by lignin and hemicelluloses, both in the leaves and the stems of different varieties of the pineapple plant .
3. The results from proximate analysis indicate that the fiber from different varieties of pineapple plant is rich in nutrients, both in the leaves and the stems.

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REFERENCES CITED

- AOAC. (1990). "Official Methods of Analysis," *Association of Official Analytical Chemists (AOAC)*, Washington, DC.
- Abdul Khalil, H. P. S., Siti Alwani, M., and Mohd Omar, A. K. (2006). "Chemical composition, anatomy, lignin distribution, and cell wall structure of Malaysian plant waste fibers," *BioResources* 1(2), 220-232.
- Agrofood Statistics (2010). "Buku Perangkaan Agro Makanan," <http://www.moa.gov.my>, Accessed 23 December 2013.
- Ahmed, O. H., Husni, M. H. A., Awang Noor, A. G., and Hanafi, M. M. (2002). "The removal and burning pineapple residue in pineapple cultivation on tropical peat: An economic viability comparison," *Pertanika J. Trop. Agri. Sci.* 25(1), 47-51.
- Alimon, A. R, and Wan Zahari, W. M. (2012). "Recent advances in the utilization of oil palm by-products as animal feed," *International Conference on Livestock Production and Veterinary Technology (ICARD)*.
- Alwani, M.S., Abdul Khalil, H.P.S., Sulaiman, O., Islam, M.N., and Dungani, R. (2014). "An approach to using agricultural waste fibres in biocomposites application: Thermogravimetric analysis and activation energy study," *BioResources* 9(1), 218-230.

- Calvert, F., Christiansen, J., Ofsoski, N., and Smillie, J. (2012). "Pasture and feed forage quality," Hill Laboratories Technical Notes, pp. 1-4.
- Food and Agriculture Organization of the United Nations. <http://faostat3.fao.org/faostat-gateway/go/to/download/Q/QC/E> (Accessed on June 15, 2014).
- Han, J. S., and Rowell, J. S. (1996). "Chemical composition of fibers," in: Paper and Composites from Agrobased Resources, Rowell, R. M., Young, R. A., and Rowell, J., eds. CRC, London, pp. 85-134.
- Hanafi, M. M., and Halimah, A. (2004). "Nutrient supply and dry-matter partitioning of pineapple cv. Josapine on sandy tin tailings," *Fruits* 59(5), 359-366.
- Hanafi, M. M., Mohammed Selamat, M., Husni, M. H. A., and Adzemi, M. A. (2009). "Dry matter and nutrient partitioning of selected pineapple cultivars grown on mineral and tropical peat soils," *Communications in Soil Science and Plant Analysis* 40(21-22), 3263-3280.
- Hoffman, P. C. (2005). "Ash content of forages," *Focus on Forage* 7(1), 1-2.
- James, C. S. (1995). *Analytical Chemistry of Foods*, Springer, New York.
- Jung, H. J. G. (2012). "Forage digestibility: The intersection of cell wall lignifications and plant tissue anatomy," Department of Agronomy and Plant Genetics. Department of Animal Sciences, University of Minnesota, USA.
- Kaliyan, N., and Morey, R. V. (2006). "Factors affecting strength and durability of densified products," *ASABE Paper No. 066077*.
- LaDon, J. J., William, E. D., and Duane, O. E. (1980). "Nitrogen in animal production," Agricultural Experiment Station, North Dakota State University of Agriculture and Applied Science.
- Meloan, C. E., and Pomeranz, Y. (1980). *Food Analysis: Theory and Practice*, AVI Publishing Company, Westport, Connecticut.
- Mendu, V., Harman-Ware, A. E., Corcker, M., Jae, J., Stork, J., Morton, S., Placido, A., Huber, G., and DeBolt, S. (2011). "Identification and thermochemical analysis of high-lignin feedstocks for biofuel and biochemical production," *Biotechnology for Biofuels*, 4(43), 1-13.
- Md. Yunos, N. S. H., Samsu Baharuddin, A., Md. Yunos, K. F., Naim, M. N., and Nishida, H. (2012). "Physicochemical property changes of oil palm mesocarp fibers treated with high-pressure steam," *BioResources*, 7(4), 5983-5994.
- Mohanty, A. K., Misra, M., and Hinnchsen, G. (2000). "Biofibres, biodegradable polymers and biocomposites: An overview," *Macromol. Mater. Eng.* 276/277, 1-24.
- Nennich, T., and Chase, L. (2007). "Dry matter determination," Feed Management Education Project, United States Department of Agriculture Natural Resources Conservation Service.
- Onyeike, E. N., Olungwe, T., and Uwakwe, A. A. (1995). "Effect of heat treatment and defatting on the proximate composition of some Nigerian local soup thickeners," *Food Chemistry* 53(0308-8146), 173-175.
- Parish, J. (2007). "Effective fiber in beef cattle diets," *Cattle Business in Mississippi*, www.msucare.com/livestock/beef/mca_mar2007.pdf, Accessed 20 April 2014.
- Parish, J. (2009). "Protein requirements of beef cattle," *Cattle Business in Mississippi*, www.msucare.com/livestock/beef/mca_apr2009.pdf, Accessed 20 April 2014.
- Paster, M., Pellegrino, J. L., and Carole, T. M. (2003). "Industrial bioproducts: Today and tomorrow," *US Department of Energy, Office of Energy Efficiency and Renewable Energy, Office of the Biomass Program*, Washington, DC.

- Pearson, D. (1976). *General Methods in the Chemical Analysis of Foods*, Longman Group, London.
- Reddy, N., and Yang, Y. (2005). "Biofibers from agriculture by products for industrial applications," *TRENDS in Biotechnology* 23(1), 22-27.
- Rosnah Shamsudin, Wan Ramli, W.D., Mohd Sobri, T., and Osman Hassan. (2009). "Physico-Mechanical Properties of the Jospine Pineapple Fruits," *Pertanika Journal of Science and Technology* 17(1), 117-123.
- Rozita Omar, Idris, A., Yunus, R., Khalid, K., Aida Isma, M. I. (2011). "Characterization of empty fruit bunch for microwave-assisted pyrolysis," *Fuel* 90, 1536-1544.
- Satyanarayana, K.G., Guimaraes, J.L., and Wypych, F. (2007). "Studies on lignocellulosic fibers of Brazil Part I. Source production, morphology, properties and applications," *Composites, Part A* 38, 1694-1709.
- Saheb, N. D., and Jog, J. P. (1999). "Natural fiber polymer composites: A review," *Adv. Polym. Tech* 18(4), 351-363.
- Steyn, W. J. A. (1959). "Leaf analysis: Errors involved in the preparative phase," *Agricultural and Food Chemistry* 7(5), 344-348.
- Tran, A. V. (2006). "Chemical analysis and pulping study of pineapple crown leaves," *Industrial Crops and Products* 24(1), 66-74.
- Wan Mohd, A., and Zainuddin, Z. (2013). "Pineapple leaf fibre (PALF): From Western to wealth," *JURUTERA Buletin*, 18-20.
- Wang, K., Jiang, J.X., Xu, F., and Sun, R.C. (2009). "Influence on steaming pressure on steam explosion pretreatment of *Lespedza stalks (Lespedzacryobotrya)*: Part 1. Characteristics of degraded cellulose," *Polym. Degrad. Stab.* 94(9), 1379-1388.
- Yang, H., Yan, R., Chen, H., Lee, D.H., and Zheng C. (2007). "Characteristics of hemicelluloses, cellulose and lignin pyrolysis," *Fuel*. 86(12-13). 1781-1788.

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