CHEMO-MECHANICAL EXTRACTION AND CHARACTERIZATION OF SAYOTE (*SECHIUM EDULE*) FIBERS AT VARYING FIBER MATURITY

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Cellulosic plant fibers are good reinforcing materials for composites because they are cheap, light weight, and exhibit good mechanical properties. The isolation of the crystalline portion exposes the stable hydrogen bond network that can form intermolecular bonding with other matrices, such as starch, polyvinyl alcohol and chitosan, among others. Sun-dried and undried sayote (*Sechium edule*) vines, of varying degrees of maturity, were subjected to uniform chemo-mechanical extraction procedures to obtain crystalline fibers. The chemo-mechanically extracted fibers were characterized using differential scanning calorimetry (DSC), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). DSC thermograms revealed that the sun-dried and mature sample exhibited the narrowest endotherm, indicating the presence of fewer amorphous structures. FTIR spectra showed that the numbers of functional groups present in the fiber samples decreased with increasing degree of maturity. SEM micrographs reveal that the mature portion of the sayote vine had more fibrous and orderly features, compared to the samples extracted from the younger and intermediate portions. Further, chemo-mechanical extraction and X-ray diffraction (XRD) analysis of fibers from mature, sun-dried sayote vines also revealed a relative crystallinity index of the extracted fiber of 65%. The fiber yield from the mature portion of the vine was 9%. Sayote (*Sechium edule*) vine can be a promising source of crystalline fibers for composite fabrication.

Keywords: chemo-mechanical, sayote (*Sechium edule*), fiber maturity, sun-drying, crystalline fiber, cellulose

INTRODUCTION

Cellulosic materials from plants are considered desirable in many industrial applications due to their environmental friendliness, mechanical properties and compatibility with other bio-polymers, such as starch, polyvinyl alcohol, and chitosan. The use of chemical and mechanical methods to isolate plant fibres is an important factor in the production of a functional crystalline cellulosic material. The removal of the amorphous region consisting of hemicelluloses, lignin, and waxes exposes the crystalline cellulosic portion of the plant fibre. The crystalline, cellulosic portion contains networks of hydrogen bonds that can form bonding with other biodegradable polymers, such as starch and polyvinyl alcohol, which makes cellulose a good reinforcing material for biodegradable polymer composites. The numerous surface –OH groups and their associated characteristics, such as ease of surface modification, high strength, potentially low cost, and renewability are some of the characteristics that make cellulose interesting to researchers and industry.[1] Micro- and nano-sized fibres are currently sought for bio-composite fabrication and other industrial applications, such as in the production of membranes, coatings, and functional surfaces, due to their superior mechanical properties. The major driver for using nanocellulose as a reinforcement for polymers is the possibility of exploiting the high tensile strength and stiffness of the cellulose crystals.[2] Due to their stiffness and strength, plant fibers can substitute glass fiber in fiber reinforced composites.[3]

The specific characteristics of plant cellulose vary depending on the geography, climate and soil condition where the plant thrives, as well as on the age of the plant, and a variety of other factors. A number of works on the isolation and characterization of various cellulosic plant fibers have been done by researchers to elucidate on their chemical, mechanical, morphological and thermal properties. The effect of chemical treatment on the properties of hemp, sisal, jute, and kapok fibers for composite reinforcement was investigated and it was reported that the mercerization of plant fibers effectively changed the surface topography and crystallographic structure of fibers.[4] It was observed that an increase in the crystallinity index resulted in an increased order of crystallite packing, and the removal of surface impurities on plant fibers could contribute to better mechanical interlocking and bonding reactions with a matrix. The comparison of the characteristics of cellulose microfibril aggregates extracted from wood, rice straw and potato tubers, using a combination of chemical and mechanical treatments, suggested that the characteristics of cellulose microfibrils are similar, regardless of plant sources and tissue function, but other factors, such as the chemical composition of the polysaccharide and moisture content, might affect the characteristics of the microfibrils.[5]

E. Abraham *et al*.[6] extracted nanocellulose fibrils from lignocellulosic fibers using a simple and low cost method – steam explosion and bleaching of fibers. Steam explosion followed by acid hydrolysis has been found to be successful in obtaining fibers in the nano-dimension from various plants. The thermal stability of the extracted nanocellulose from plant samples was higher than their respective raw fibers. The investigation of the physical, chemical, and mechanical properties of raw and alkali treated borassus fruit fiber revealed that alkali treatment removed the hemicelluloses, lignin and other impurities from the fiber surface, which made the surface smooth, leading to better bonding with matrices.[7] Morphological studies revealed that impurities were removed from the fibers after alkali treatment. Borassus fruit fibers treated with 5% NaOH solution were found to have better mechanical properties than fibers treated with 10% and 15% alkali solutions.

Sundari and Ramesh[8] isolated and characterized cellulose nanofibers from aquatic weed water hyacinth – *Eichornia crassipes* – through chemical and mechanical treatment. The authors stated that pure cellulose fibers were obtained after the chemical treatment. The diameter of the isolated fibers was 25 nm and the length was in the micrometer range. Lignin and hemicelluloses were removed when the fiber was treated with acidified sodium chlorite and sodium hydroxide solutions at different time intervals and temperatures. The characterization of cooked blue agave bagasse fiber from Mexico revealed that fiber cells were regularly arranged and elliptical in shape, with varying lumen size.[9] Nanocrystalline cellulose (NCC) from rice straw was extracted using acid hydrolysis, and characterized. The findings revealed a material with controlled size, high specific surface area, self-assembling nature, rod-like morphology, and good dispersion.[10] The isolation of a high purity nanometer sized cellulose from water hyacinth fiber using a digester and sonication was made possible. Acid hydrolysis was able to remove lignin and hemicelluloses, and sonication reduced the fiber length.[11] Nanometer sized cellulose from pineapple leaf fibers were isolated and characterized. Combining high shear homogenization and ultrasonication after chemical pre-treatment was reported to be effective in producing nanometer sized cellulose.[12] Other methods of extraction have also been reported. The mechanical disc refining pre-treatment of sponge-gourd increased the surface area of fibers, promoted the removal of hemicelluloses, increased fiber devolatilization index, and increased the fiber crystallinity index.[13] Diyana *et al*.[14] used the water retting method in which moisture and enzymatic reactions were utilized to extract fibers from pandan leaves. The method produced fibers with 48.79% cellulose content.

Sayote is an edible plant belonging to the gourd family, the Cucurbitaceae. It is a tropical perennial vine that can reach as high as 40 feet. At present, sayote is the most sustainable vegetable cash crop in the Cordillera Administrative Region, Philippines. It can have a life span of around 20 years with proper care and management. The sayote plant can also grow naturally on empty lots where trees and stonewalls are available for climbing, as seen in Figure 1. The abundance of the sayote vine in the Cordillera Administrative Region (CAR) in the Philippines makes it a good source of fiber for industrial purposes.

Previous reports in the literature have identified differences in fiber structure as a function of plant maturity – namely, more crystalline fiber structure has been found in the mature portion of plants. Specifically, Gorshkova *et al.*[15] investigated the cell wall polysaccharides of developing flax plants and they were able to discover an increase in cellulosic cell wall thickness at maturity. With the use of more advanced spectroscopy methods, the cellulose content of various regions of *Arabidopsis thaliana* inflorescence was analyzed, and some changes in cellulose ordering were found as tissues matured. Thus, older cells, with thickened secondary cell walls, were found at the base and younger primary cell walls were found on top of the inflorescence.[16] As regards sayote fiber, to the authors' knowledge, no studies have been reported focusing on its properties as a function of plant maturity. In our previous study, fibers from the mature portion of the plant were used in composite fabrication.[17] The fabrication of starch/PVOH composite blends, with the incorporation of mature sayote fiber as reinforcement, led to improved chemical, mechanical, and thermal properties of the composites. The present work was performed with the objective to investigate the chemical and mechanical properties of sayote fiber at varying degrees of maturity.

EXPERIMENTAL

Plant sample

Vines were collected from 6-year-old sayote plants during the dry season, in February, from Santo Tomas Proper, Baguio City, in the Philippines. Old, intermediate and younger parts of the vines were considered for fiber extraction and characterization. 'Old' refers to the portion taken near the root of the sayote plant, 'young' refers to the portion taken near the shoot of the sayote plant, and 'intermediate' refers to the portion taken in between the mature and young portions. Sun drying for 5 days before extraction was done prior to treatment. A bunch of sayote composed of 6 vines around 3 meters long were gathered and sorted according to their levels of maturity. Fibers from 100 grams of each old, intermediate and young portions, in their dried state, were extracted by the chemo-mechanical method and then characterized.

Chemo-mechanical extraction

In this study, the sayote vine sample was pre-treated with 17.5% w/w NaOH for 2 hours. It was acid hydrolyzed with 1 M H_2SO_4 at 70-80 °C for 2 hours. The acid hydrolyzed vines were then soaked in 2% w/w NaOH for 2 hours at 70-80 °C, followed by hydrogen peroxide treatment, using a 30% v/v solution, for 12 hours. The chemically treated fibers were subjected to a defibrillation process for 1 minute, using an Oster osterizer, to separate the lignin, hemicelluloses, and the crystalline portion of the fiber. The defibrillated fiber was ultrasonicated in an Elmer Perkin Sonicator at 30 kHz for 5 hours to obtain the fiber. The final product, as seen in Figure 2, was washed 10 times with distilled de-ionized water to neutralize the pH. The extracted sayote fibers were frozen for 1 day, then dried for 2 days, using an SIM International Freeze Dryer at -86.4 °C and 2 mTorr.

Figure 1: Climbing sayote vine – the plant can reach as high as 3 to 6 meters

Figure 2: Suspension of extracted sayote fiber suspended in distilled de-ionized water after ultrasonication

Characterization based on plant maturity and sun-drying

Chemical analysis

Infrared spectra were obtained using a Shimadzu IR Affinity system to determine the functional groups present in the extracted fiber. An amount of 1 mg of the sample was mixed with KBr in a 1:10 ratio, and pressed into a KBr disc. The transmittance was measured at 100 scans from 400-4000 cm^{-1}.

Morphological analysis

SEM micrographs were obtained using a Hitachi TM-1000 SEM to determine the surface morphology of the extracted fibers. The fibers were gold sputtered for one minute before observing at different magnifications.

Thermal analysis

Differential scanning calorimetry (DSC) profiles were obtained using a Shimadzu DSC50 to determine the thermal behavior of the extracted sayote fibers. A 3.00 mg sample was introduced into an aluminum cell and was subjected to a heating rate of 10 °C/min from 30 °C to 280 °C in a liquid nitrogen atmosphere at a flow rate of 20 mL/min.

Crystallinity analysis

The Siemens Kristalloflex 760 X-ray generator, with a copper X-ray tube (wavelength = 1.54056 Å) and a Philips 1080 vertical goniometer at a high voltage of 34 kV and 20 mA current, was used to determine the crystallinity index (CI) of the extracted fibers. The fibers were ground to form a very fine substance. The samples were scanned using Cu Kα radiation at 2θ (°) in the range from 5.0° to 85° at a scanning rate of 0.02°. The crystallinity index was computed using the peak-height method – Segal's method (Eq. 1) – where I(am) is the peak intensity of the amorphous portion and I (002) is the peak intensity of the crystalline portion.[18]

$$Ic = (I(002)-I(am))/(I(002)) X100 \quad (1)$$

RESULTS AND DISCUSSION

Freeze drying was chosen as a drying method, instead of vacuum oven drying, in order to prevent fractures on the fiber surface and in an attempt to achieve a smaller fiber diameter. It has been stated previously that oven drying can cause fractures on the fiber surface, while freeze drying allowed obtaining fiber diameters in the nano-range, though agglomeration occurred.[19] The percentage weight recovery of sayote fibers extracted from young, intermediate and old portions of the vine was 3%, 5%, and 9%, respectively. Figure 3 shows the extracted fiber, revealing variation in fiber color, from a darker to a lighter shade, with increasing maturity. This means that the extracted mature sayote fiber contains higher cellulose amounts. This result is also supported by the findings of Johar *et al.* (2012), who extracted cellulose fibers and nanocrystals from rice husk, by alkaline treatment and bleaching – the authors associated the white color of the final product with the presence of an almost pure cellulosic material.[20]

Infrared spectroscopy of extracted sayote fibers

Figure 4 shows the FTIR spectra of sun-dried fibers of different maturity. The peak at 1720 cm^{-1} is assigned to the presence of a (C=O) bond, characteristic of the acetyl and uronic ester linkage of the carboxylic group to the ferulic and p-coumeric acids of lignin and hemicelluloses, in young, intermediate, and old sayote fibers, respectively, which apparently decreased with fiber maturity. This means that the mature sun-dried sayote sample contained a lower amount of lignin and hemicelluloses. The peak at 2328 cm^{-1}, which is due to the strengthening of hydrogen bonds, appeared in the mature sun-dried sayote sample, but not in the young and intermediate sun-dried samples. This means that the mature sayote sample exhibits the presence of networks of hydrogen bonds in the cellulose structure.

A B C

Figure 3: Photographs of fibers extracted from (A) young, (B) intermediate, and (C) old portions of sayote vines

The results of the FTIR analysis reveal the removal of lignin and hemicelluloses after the NaOH pre-treatment process. This process is known as a major contributory factor in the removal of the amorphous regions from the fiber bundle. The hydroxide ion (OH^-) weakens the hydrogen bonding interaction between the crystalline cellulose and the amorphous hemicelluloses and lignin in the fiber. Similar results have been reported by L. Boopathi *et al.*, who successfully removed lignin and hemicelluloses from 15 percent alkali treated borassus fruit fiber;[7] and by M. T. Sundari and A. Ramesh, who removed lignin through NaOH treatment of *Eichornia crassipes* (water hyacinth).[8] Another contributory factor is bleaching with hydrogen peroxide (H_2O_2). Surface impurities in the cellulosic fiber are decomposed by the release of the hydroxyl ion (HO_2^-) that results in further removal of oxidizable substances – hemicelluloses and lignin. Other authors also reported that steam explosion and bleaching of fibers removed the lignin from the fibers.[6]

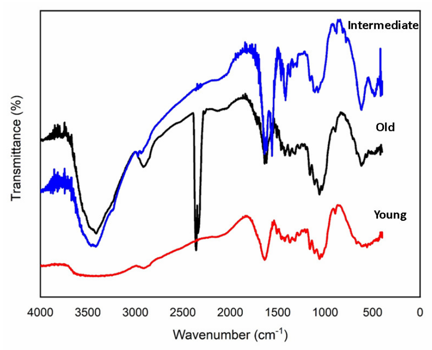

Figure 4: FTIR spectra of sun-dried fibers of different maturity

Thus, the chemo-mechanical process performed in this study, involving NaOH pre-treatment, acid hydrolysis using H_2SO_4, bleaching with H_2O_2, ultrasonication and defibrillation, contributed to the removal of lignin and hemicelluloses, which are not desirable for a fiber's use as reinforcing agent in composites.

Surface morphology

The surface morphology of the extracted fibers was examined through scanning electron microscopy. Figure 5 shows the SEM micrographs of the sayote fibers at 1000x magnification. A more orderly pattern and a smoother surface can be seen with increasing fiber maturity, specifically in the fibers extracted from the intermediate and old portions of the vine. This can be explained by the lesser amount of lignin and hemicelluloses in those fibers.

The orderly and smooth surface indicates the removal of the amorphous regions and impurities, which is favorable to the formation of networks of hydrogen bonds – an important feature for composite fabrication, as well as for other applications, allowing bonding interactions with the matrix material. Other researchers have reported similar results after alkali treatment. Boopathi *et al*. performed 5% alkali treatment of fiber and revealed a smoother fiber surface due to the removal of surface impurities, such as fatty substances.[7] A higher magnification SEM micrograph of the dried mature sayote fiber shown in Figure 6 demonstrates that smaller fiber diameter was successfully obtained.

Thermal behavior of extracted sayote microfibers

Differential scanning calorimetry (DSC) curves show the behavior of sayote microfibers upon heating. Figure 7 shows the endothermic processes occurring in fibers extracted from sun-dried young, intermediate and older portions of the sayote vine. The presence of a large endothermic peak on the curves indicates the evaporation of residual moisture from the fiber. The thermogram corresponding to the younger portion of the vine has the broadest and deepest endotherm, which can be explained by the fact that the young fiber contains a more amorphous structure, as compared to those extracted from the intermediate and older portions of the vine. The intermediate fiber has a broader endotherm than the older one, thus confirming that the older fiber contains the least amount of amorphous structure among the analyzed samples.

The lower amount of amorphous lignin and hemicelluloses in fibers originating from older portions of the plant are explained by the fact that, in plants, secondary cell walls develop with maturity. Specifically, fibers are cells of mechanical tissue called schlerenchyma, which possess extremely long and well developed secondary cell walls. Sun drying also contributed to the strengthening of the fiber due to the natural mechanism of the mesophyll in the plant cell wall, consisting in closing so as protect itself from loss of cellular integrity during drying.[21]

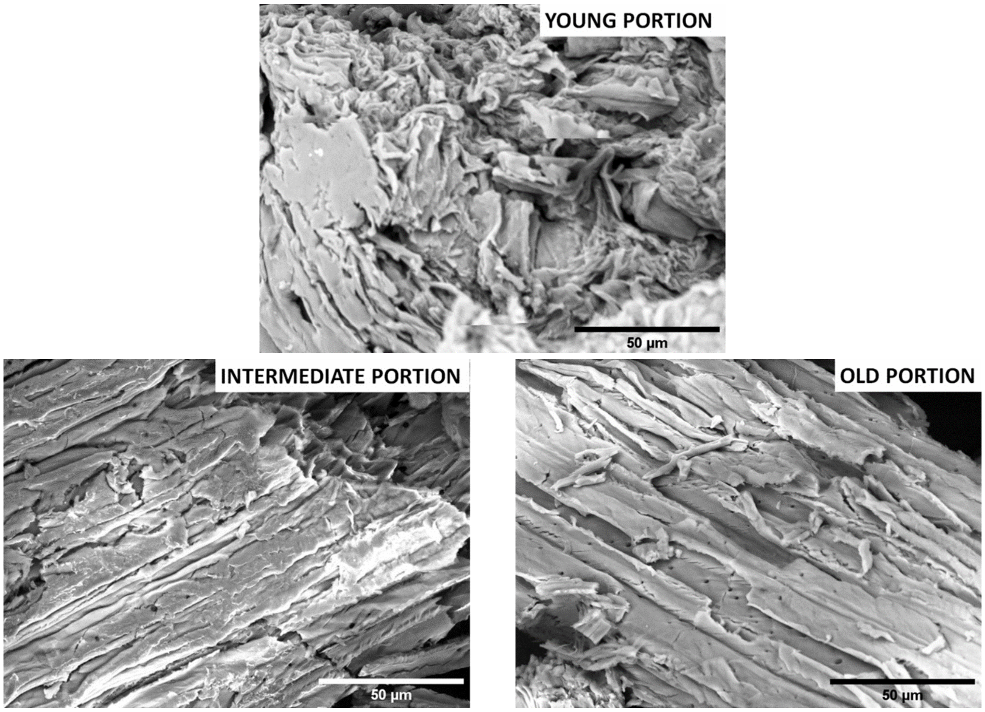

Figure 5: SEM micrographs of sayote nanofibers at 1000X

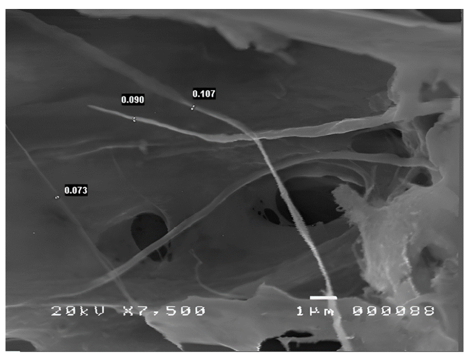

Figure 6: SEM micrograph of mature sun-dried fiber at 7,500X, showing the nano-range diameters of the extracted fiber strands (73, 90 and 107 nanometers)

Fiber crystallinity of extracted sayote fibers

The extracted sayote (*Sechium edule*) fibers, on the basis of maturity, were subjected to X-ray diffraction (XRD) to identify their crystallinity index (CI). Figure 8 shows the XRD diffractograms of the fibers obtained from young, intermediate, and mature portions of the sun-dried sayote vines. In general, XRD diffractograms of most cellulosic materials show a broadened peak and an amorphous region. The XRD patterns recorded for intermediate and mature fibers in this study conform to this observation. However, the pattern corresponding to the young fibers is unusual, showing a high intensity narrow peak, with an unclear amorphous region. We assume that there is an overlap in the peaks of low crystalline materials, which led to an unusual diffractogram. This implies that fibers extracted from the young portion of the vine would not be suitable as reinforcing agent in composites and for other application that require high mechanical strength and hydrogen bonding interactions with other materials.

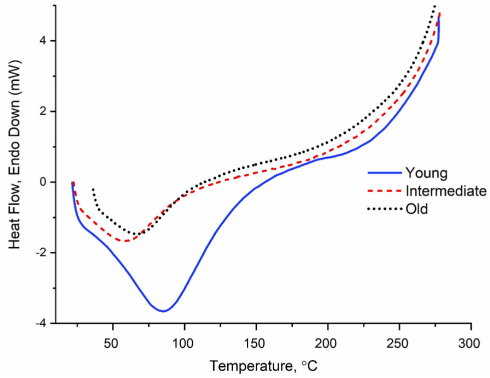

Figure 7: DSC thermograms for sun-dried fibers at different levels of maturity

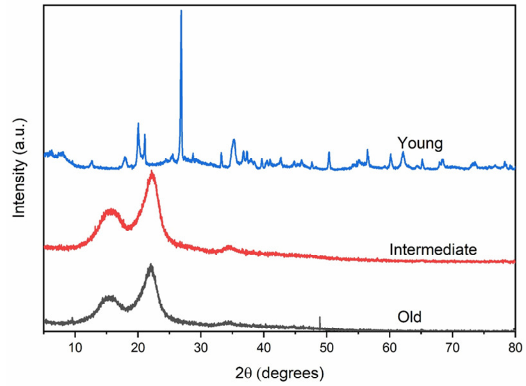

Figure 8: XRD diffractograms of extracted fiber from young, intermediate, and old sayote vine

The crystallinity index (CI) of mature sayote fiber is around 65%, which is higher than the CI of cellulose fibers chemically extracted by other authors in previous works. For example, W. Chen *et al*. reported 60% crystallinity index for wood, bamboo, wheat straw, and flax fibers;[22] N. Johar *et al*. obtained fiber crystallinity of 59% from rice husks;[20] while P. Lu *et al*. – of 54.9% for chardonnay grape skin fibers.[23] However, higher crystallinity that that obtained for mature sayote fiber has also been reported in the literature: R. Sheltami *et al*. extracted fibers from mengkuang leaves, which reached 69.5% crystallinity,[24] while Mattoso *et al*. reported sugarcane bagasse fiber with a crystallinity index of 87.5%.[25] Still, the crystallinity index obtained for the mature sayote fiber can be considered high and it indicates the strength of the fiber after the removal of the amorphous region. The exposure of the more orderly crystalline portion of the fiber enables the formation of hydrogen bonds between the fiber surface and the matrix, which is necessary for composite fabrication.

CONCLUSION

In the light of the findings of the present study, the fibers extracted from the mature sayote vines exhibited lower lignin and hemicellulose content, compared to those from intermediate and young portions of the plant, as confirmed by the DSC, FTIR and SEM analyses performed. Also, XRD diffractograms revealed that the dried mature sayote fiber had relatively high crystallinity, being higher that that reported in the literature for fibers extracted from various other sources, such as wood, bamboo, wheat straw, flax, rice husk, and chardonnay grapes, but lower that others: mengkuang leaves and sugarcane bagasse fibers. SEM analysis demonstrated the nanometer-size of the obtained mature sayote fiber.

To conclude, sun-dried mature sayote (*Sechium edule*) vine can be a promising source of crystalline cellulose fibers, which may be suitable for composite fabrication, when extracted using the chemo-mechanical technique. The treatment applied allowed the removal of the amorphous portion of the fiber and exposed the crystalline region, which, due to its known mechanical strength, is desirable for its application as a reinforcing material in the development of environmentally friendly composite materials.

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