
Characterization of the Physical Properties of *Bacillus thuringiensis* Corn husk Fibers through Alkalization

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Abstract –*Bacillus thuringiensis* (Bt) corn husk bers were characterized for possible usage in textile manufacture. The ber yield, diameter and length were determined and compared to that of native sweet corn husks bers Bt corn husks were subjected to alkalization for 60 minutes at 5 g/L and 10 g/L NaOH concentrations. Fibers subjected to 5 g/L and 10 g/L NaOH concentration treatment had a ber yield of 14.69% and 16.74% respectively. The ber diameter and the ber length was greater in the 5 g/L (817.7 μ m and 11.7 cm) than in the 10 g/L (723.7 μ m and 5.3 cm) NaOH concentration treatment. Fiber yield was measured using a standard analytical balance. Measurement showed no significant difference between the two corn species. Diameter of bers was also measured using the Laser Diffraction method and also no significant difference can be observed. The ber length, measured using a Vernier Caliper had a significant difference. Bt corn husk bers do not have an observable advantage over native sweet corn.

Introduction. – For the past several years, due to the rapid changes in climate, awareness towards these environmental issues accelerated industries to start utilizing sustainable and renewable resources instead of conventional synthetic materials [1]. For a material to be classified as sustainable, it should be biodegradable or recyclable, made from renewable resources, should in any way not cause any harm to the environment and must be on par with conventional materials in terms of quality and price. Agricultural byproducts are amongst one of the most common renewable resources which are usually secondary or residual products from common crops such as cotton, rice, corn and wheat. Residues such as husks, bagasse, and stalks are usually discarded in most countries. Research regarding these residues are commonly studied in the Philippines since it is an agricultural country and majority of the practices within the land include burning of these residues or turning them into fertilizer. Some are also utilized as alternative fuel however proper knowledge and procedures are yet to be released and may even pose a threat to the environment as well as the people. This however can help farmers since it reduces their expenditures in the aspect of farming. These agricultural byproducts are cheap source of cellulosic bers. The composition and structure makes them suitable to be used as composites, textile, and paper manufacturing [2]. *Bacillus thuringiensis* (Bt) corn is

a genetically modified species of corn which includes the Bt protein capable of giving protection from pests. Bt-corn hybrids are different from nonBt corn not only in the genetic code but also in some additional genetic material (Bessin 2010). The Bt protein comes from a soil bacterium, *Bacillus thuringiensis*, which has the ability to produce protein toxins which are harmful to larvae of the pests [44]. Plant based bers contain cellulosic materials that provides strength and rigidity to the bers. Aside from cellulose, non-cellulosic materials are also present such as pectin which holds the bers together and lignin which acts as a binder for cellulose bers adding strength and stiffness to the cell walls [4].

According to the Department of Agriculture (DA), corn is the second most important crop in the Philippines in par with being the second largest agricultural crop grown in the world according [5]. Approximately 14 million Filipinos regard corn as their main source of food. Besides being food for humans, nearly 50% of livestock feeds contain corn. Besides the corn itself, other parts can also be utilized for biofuel from corn oil and as stated beforehand, forage and silage for livestock feeds. The numerous amounts of agricultural byproducts such as cob, husk and silk produced by the massive production of corn, especially the Bt corn, is under-utilized or considered as waste.

These agricultural byproducts can be used as a source of raw material for ber. Majority of the parts besides the corn itself such as the cob, husk and silk is under-utilized and considered as a waste by many. These byproducts can be used as a source of ber for many other uses [6].

Methods. – The conduct of the research experiment is divided in six phases: first, the collection and Bt corn husks; second, the ber extraction; third, calculating the ber yield; fourth, the calculation of the ber diameter; fifth, the measurement of the ber length and; sixth, analysis of the data using statistical tests.

Materials. Bt corn was acquired from a sweet corn vendor near the Jaro Public Market. These Bt corn were from a plantation San Miguel, Iloilo. The husks were transported to the Philippine Science High School - Western Visayas Research Laboratory for storage prior to ber extraction. Chemicals and laboratory equipment such as glasswares and measuring devices were borrowed from the Chemistry Lab of the school. Other basic materials such as distilled water were bought from the local supermarket.

Procedure. To extract the bers three set-ups containing 50 grams of corn husks were placed in a container with a 5 g/L NaOH concentration and another three set-ups containing 50 grams of corn husks were submerged in a one liter distilled water with 10 g/L NaOH concentration placed in a two-liter beaker. These beakers were placed on a hot plate set to 450 for 90 minutes. After alkalization, the bers were rinsed in a beaker filled with 50 mL distilled water for several times. They were then placed on another beaker containing a 50 mL of a 10% acetic acid solution, and then rinsed again until pH 7 is reached. The pH level was measured using a pH meter. After extraction the bers were air dried for 48 hours, each setup was weighed using a top loading balance. A total of 15 samples were descriptively and inferentially measured. The diameters of the samples were measured using the laser direction method. Each strand was placed on a cardboard with a 4cmx1cm slit. A 532 nm green laser pointer was situated 30cm from the strand. The distance between the strand and the wall was 4m. As the laser pointer hit the strand, the bright bands were measured from the center of the bright central band to the starting edge of the first bright band to the left. For each measured distance, the diameter of the ber was solved using the formula

$$\frac{\lambda}{\frac{D_m}{D_w}} \quad (1)$$

where $\lambda = 532$ nm, $d_{\text{wall}} = 4$ m since it is the distance between the wall and the strand and d is the distance between the center of the bright band of the laser and the starting edge of the first bright band to the left. The samples were measured from end to end using a Vernier caliper to get its length. The data acquired were analyzed using the IBM Statistical Package for the Social Science (SPSS) Software.

The mean and standard deviation were determined for descriptive analysis. The t-test was used to determine significant differences between two independent samples in the study.

Results. – This study specifically aimed to extract bers from Bt corn husks and determine and compare the ber yield, diameter, and length of extracted Bt corn husk bers when subjected to alkalization treatment with NaOH concentration of 5 g/L and 10 g/L. Bt corn husks were boiled per each concentration and 150 g of the husks were divided into three setups with 50 g each. The three setups were boiled in a 2 L solution of water and NaOH. The solution of NaOH and water followed a concentration of 5 g/L and 10 g/L. The three set-ups were boiled for one hour. Each setup was then rinsed with 100 mL 10% acetic acid solution. After rinsing, the bers were again rinsed with distilled water until pH 7 was reached. The pH was measured using a digital standard pH meter. Fifteen samples that were randomly picked per setup were measured for ber diameter and length.

Fiber Length, Fiber Diameter, Fiber Yield. The ber yield after alkalization is higher in the 10 g/L (16.74%) than in the 5 g/L (14.69%) NaOH concentration. Table 1 shows the data.

NaOH Conc	Mean Fiber Yield	Std. Dvt.
5g/L	14.69	±5.36859
10g/L	16.74	±2.09001

The fiber diameter is greater in the 5 g/L (817.7 μm) than in the 10 g/L (723.7 μm) NaOH concentration treatment. See table

NaOH Conc	Mean Fiber Diameter(cm)	Std. Dvt.
5g/L	817.7	±156.34684
10g/L	723.7	±83.76536

The ber length is greater in the 5 g/L (11.8067 cm) than in the 10 g/L (5.3333 cm) NaOH concentration treatment.

NaOH Conc	Mean Fiber Diameter(cm)	Std. Dvt.
5g/L	11.8	±2.93873
10g/L	5.3	±1.85831

t-test Results on Fiber Yield, Diameter, and Length. The t-test results shows that there is no significant difference between the ber yield of Bt corn husks treated with 5 g/L and 10 g/L NaOH concentration as reflected by the

$t(4) = -0.547$, $p=0.613$. Additionally, there is no significant difference between the ber diameter of Bt corn husks treated with 5 g/L and 10 g/L NaOH concentration as reflected by the $t(4) = 0.918$, $p=0.411$. However, there is a significant difference between the ber length of Bt corn husks treated with 5 g/L and 10 g/L NaOH concentration as reflected by the $t(4) = 3.225$, $p=0.032$. See table.

Table 4: T-test Results

Fiber Property	df	t-value	p-value
Yield	4	-0.547	0.613
Diameter	4	0.918	0.411
Length	4	3.225	0.032

Discussion. – Fibers subjected to 5 g/L and 10 g/L NaOH concentration treatment had a ber yield of 14.69% and 16.74 % respectively. The ber diameter in the 5 g/L is 817.7 μ m and 723.7 μ m in the 10 g/L NaOH concentration treatment. The ber length in the 5 g/L is 11.7 cm and 5.3 cm in the 10 g/L NaOH concentration treatment. There is very little difference with the ber yield of Bt corn husks treated with 5 g/L and 10 g/L NaOH concentration. With the difference of only 2%, there is no substantial effect in the ber yield of Bt corn husks with 5 g/L and 10 g/L NaOH concentration alkalization treatment. The results of Yilmaz in his study in 2013 regarding the chemical extraction parameters of corn husks showed that corn husks treated with 5 g/L NaOH concentration has higher yield (6%) than the husks treated with 10 g/L NaOH concentration (3%). The results obtained by Yilmaz were lower than the results acquired by the researchers which are 14.69% for the 5 g/L and 16.74% for the 10 g/L NaOH concentration treatment. Similar to the ber yield, there is no significant difference in the diameter of bers extracted from Bt corn husks. In the study of Ekhuemelo and Tor in 2013 regarding the assessment of corn stalk and husk characteristics, the corn husk ber diameter is 30.19 μ m. This value is lower than the ber diameter value acquired by the researchers. The difference in the results acquired compared to the other studies mentioned above is due to the difference in the components of Bt and non-Bt corn husks; Bt corn husks have higher content of lignin than non-Bt corn hybrids [7]. Lignin, together with cellulose, is the most important structural component of natural bers. Lignin assists in the water transportation and rigidity of plants by adding strength and stiffness to the cell walls [8]. The basic components of bers such as cellulose, hemicelluloses and lignin govern the physical properties of bers [9]. The 5 g/L and 10 g/L NaOH concentrations of alkalization treatments have substantial effect on the ber length. The harshness or higher concentrations of alkalization treatments have affected the extracted ber length negatively. NaOH binds to cellulose molecules into aqueous solution to a certain extent, and prevents macromolecules from associating, so greater amount of NaOH has harsher effect to the

bers [10]. The length of corn husk bers extracted by Yilmaz in his aforementioned study is 15.5 cm for the bers treated with 5 g/L NaOH concentration and 9.3 cm for the bers treated with 10 g/L NaOH concentration. This is consistent with the results acquired by the researchers in which bers treated with higher NaOH concentrations have shorter length. Physical properties of natural bers are important in determining the suitability of bers in different industrial materials [9]. The length and diameter of Bt corn husk bers extracted from both 5 g/L and 10 g/L NaOH concentrations are within the range of bers extracted from date palm. The similar physical properties shared by date palm and Bt corn husk bers have potential for a wide range of applications [11,12]. These applications include natural ber composites (NFC) and modern industrial applications such as materials for the automotive industry [11]. Factors that may affect the data acquired by the researchers are the following: different models of hot plates used by each setup, the rinsing done separately by the three researchers, and the other factors such as climate, weather conditions and soil quality [13]. Furthermore, various other physical properties were not characterized by the researchers due to the time constraint and lack of budget and expertise. Compared to the industrial properties of ber, Bt corn husk bers are on par in terms of basic properties such as ber length. In terms of ber length distribution, Bt corn bers are not equal to that of industrial ber. This is due to the processes done in order to retrieve the ber. Fiber length distribution indicates the strength, length and yield of a ber. The higher the value, the higher its potential in the industry. Bt corn husk bers are not equal to industrial bers however it still has potential in the industry due to the fact that it comes from agricultural byproducts. In summary, there is no significant difference in the ber yield and diameter of Bt corn husks bers, while there is a significant difference in ber length. Bt corn husk bers differ in native corn husk bers due to the abundance of lignin in Bt corn husk bers. Lignin is one of the most important structural components of natural bers. These physical properties of Bt corn husks are within the range of date palm bers. Consequently, Bt corn husk bers have a potential wide range of applications.

Conclusion. – The ber yield and diameter of bers treated with 5 g/L and 10 g/L NaOH concentration have no significant difference while there is significance in ber length. As compared to industrial ber, Bt corn husk bers are comparable only in terms of mechanical properties such as length and cannot stand alone as enough evidence for it to be in the same level as industrial bers.

Recommendations. – It is recommended to conduct the boiling of Bt corn husks in 5 g/L and 10 g/L concentration at the same time since factors such as air temperature and humidity has an effect in the boiling time and cooking of Bt corn husks [14]. The use of fully-functional hot plates with similar models is also recommended as this, too, can affect the boiling time and cooking. It is also recommended to use Bt corn husks with the same moisture content, age,

and condition. Using different NaOH concentrations and cooking time is also recommended since this can affect the physical properties of Bt corn husk fibers [15]. Expansion or additional parameters to be acquired is also advised not only to gain more supporting data but also to expand the range of uses of the fiber and not limit it to textile industries only.

REFERENCES

- [1] Di Bella G, Fiore V, Valenza A. The effect of alkaline treatment on mechanical properties of kenaf fibers and their epoxy composites. *Composites. Part B* (68): 14-21.
- [2] Reddy N, Yang Y. 2005 Biobers from agricultural byproducts for industrial applications.
- [3] Gewin V. 2003. Genetically Modified Corn Environmental Benefits and Risks. *PLoS Biology* 1 (1): e8. Available from: <http://journals.plos.org/plosbiology/articles?id=10371/journal.pbio.0000008>. Gu L, Jin H, Zha C. 2007. Direct dissolution of cellulose in NaOH/thiourea/urea aqueous solution. *Carbohydrate Research*. 342 (6). 851-858.
- [4] Ashan J. 4 July 2012. Different types of fiber and their uses [Internet]. *Apparelbrief*; [4 July 2012, cited 17 March 2016]. Available from: <https://apparelbrief.wordpress.com/2012/07/04/different-types-fiber-their-uses/>
- [5] Majeed K, Jawaid M, Hassan A, Abu Bakar A, Abdul Khalil HPS, Salema AA, Inuwa I. Potential materials from food packaging from nanoclay/natural fiber based composites. *Materials-Design* 46: 391-410. DOI: 10.1016/j.matdes.2012.10.044.
- [6] Chen B, Wilkinson S, Xia J, editors. 2016. *Sustainable Buildings and Structures*. London (LN): Taylor-Francis Group. 275 p.
- [7] Gu L, Jin H, Zha C. 2007. Direct dissolution of cellulose in NaOH/thiourea/urea aqueous solution. *Carbohydrate Research*. 342 (6). 851-858
- [8] Hakeem KR, Jawaid M, Rashid U, editors. 2014. *Biomass and Bioenergy: Processing and Properties*. Cham (SW): Springer International Publishing.
- [9] Chirayil CJ, Mathew L, Thomas S. 2014. Review of recent research in nanocellulose preparation from different lignocellulosic fibers. *Rev. Adv. Mater. Sci.* 37: 20-28. Available from: <http://www.ipme.ru/ejournals/RAMS/no13714/03-13174-cintil.pdf>
- [10] AL-Oqla FM, Sapuan SM. 2014. Natural fiber reinforced polymer composites in industrial applications: feasibility of date palm fibers for sustainable automotive industry. *Journal of Cleaner Production*. 66 (2014): 347-354.
- [11] Ekhuemelo DO, Tor K. 2013. Assessment of fiber characteristics and suitability of maize husk and stalk for pulp and paper production. *Journal of Research in Forestry, Wildlife and Environment*. 5 (1). 41-49.
- [12] Bledzki AK, Faruk O, Fink HP, Mohini S. November 2012. Biocomposites reinforced with natural fibers: 2000-2010. *Progress in Polymer Science*. 37(11): 1592-1596. Bledzki AK, Mamun AA, Lucka-Gabor M, Gutowski VS. 2008. The effects of acetylation on properties of fiber and its polypropylene composites. *Polymer Letters* [Internet]. [cited 1 December 2017]; 2(6): 413-422. Available from: www.expresspolymlett.com/letolt.php?le=EPL0000609-mi=cc
- [13] Fibre2Fashion [Internet]. c2015. Philippine textile sector getting back on track: Experts. [cited 24 March 2015]. Available from: <http://bre2fashion.com/news/textilenews/newsdetails>
- [14] Delta Farm Press [Internet]. 19 October 2001. Factors affecting fiber quality; [19 October 2001; cited 26 March 2016]. Available from: <http://deltafarmpress.com/factorsaffecting-fiber-quality> Di Bella G, Fiore V, Valenza A. The effect of alkaline treatment on mechanical properties of kenaf fibers and their epoxy composites. *Composites. Part B* (68): 14-21.
- [15] Fibre2Fashion [Internet]. c2015. Philippine textile garment exports cross 2bn in 2014. [cited 13 February 2015]. Available from: <http://bre2fashion.com/news/textilenews/newsdetails>
- [16] Haufe J, Patel MK, Shen L. 2009. Product overview and market projection of emerging bio-based plastics [Internet]. [Cited 09 Sep 2016]; Available from: <http://www.plastice.org/leadadmin/les/PROBIP2009Final-June-2009.pdf>
- [17] ISAA SEAsiaCenter. 2014. *Agricultural Biotechnology: A Lot More than Just GM Crops*. Irri, Los Baos, Laguna (PH): ISAA SEAsiaCenter.
- [18] Li X, Tabil L, Panigrahi S. 2007. Chemical treatment of Natural fiber for use in natural fiber reinforced Composites: a Review. *J Polym Environ*. Article from: *Journal of Polymers and Environment*
- [19] Bessin R. [Internet]. 2010. *Bt-Corn: What it is and How it Works*. Lexington, KY (USA): Department of Entomology, University of Kentucky College of Agriculture [updated 2013 Jan 22; cited 2010]. Available from: <http://www2.ca.uky.edu/entomology/entfacts/ef130.asp>.
- [20] Mercola J. [Internet]. 2013 Sept 10. Monsanto Decimates Their Credibility [cited 2015 Oct 28]. Available from: <http://articles.mercola.com/sites/articles/archive>
- [21] Mojsov K. 2011. Application of Enzymes in the Textile Industry: A review. [Internet]. [cited 2017 October 20] Available from: <http://eprints.ugd.edu.mk/1381/1>
- [22] Nishiyama Y, Langan P, Chanzy H. 2002. Crystal Structure and Hydrogen-Bonding System in Cellulose I from Synchrotron X-ray and Neutron Fiber Diffraction. *American*
- [23] Chemical Society. [cited 23 October 2017]; 124 (31), 90749082. Available from: <http://pubs.acs.org/doi/pdf/10.1021/ja0257319> DOI: 10.1021/ja0257319
- [24] Ongpin M. [Internet]. 2015. Philippine textile industry on the rise. *The Manila Times* (PH): manilatimes.net; [cited 13 March 2015]. Available from: <http://www.manilatimes.net/philippine-textile-industry-on-the-rise/168985>
- [25] Preston J. 2015 [Internet]. *Britannica*; [2015, cited 2015 Dec 12]. Available from: <http://www.britannica.com/technology/man-made-fiber/625386>. Man fibers, a fiber whose composition are modified.
- [26] Rayapura A. [Internet]. 2014. *University of Nebraska Engineers Creating Textiles from Corn Husks*. *Sustainable Brands*: sustainablebrands.com; [cited 08 April 2014]. Available from: <http://www.sustainablebrands.com/news-views/chemistry-materials/aarthi-rayapura/university-nebraska-engineers-creating-textiles>

- [27] Reddy N, Yang Y. 2014. Properties and potential applications of natural cellulose fibers from cornhusks. *The Royal Society of Chemistry* [Internet]. [18 October 2017]; 7:190-195. Available from: <http://pubs.rsc.org/en/Content/2005/GC/b415102j>
- [28] Reddy N, Yang Y. 2004. Natural cellulose fibers from corn stover. *Innovative Biobers for Renewable Resources*. DOI: 10.1007/978-3-662-45136-6-2.
- [29] Abdullah M, Anderson M, Anderson M. *Indian Corn*. Britannica. [Internet] Available from <http://www.britannica.com/plant/corn-plant>
- [30] A second life for rice husk [Internet]. IRRI; [cited 2017 Oct 16] Available from: <http://irri.org/rice-today/a-secondlife-for-rice-husk> A second life for rice husk [Internet]. IRRI; [cited 2017 Oct 16] Available from: <http://irri.org/rice-today/a-secondlife-for-rice-husk>
- [31] Sarmiento, B. 2012 April 17. A decade after: Bt corn farming robust in PH. *MindaNews*. Available from: <http://mindanews.com/business/2012/04/17/a-decadeafter-bt-cornfarming-robust-in-ph>.
- [32] Saxena D, Stotzky G. 2001. Bt corn has a higher lignin content than non-Bt corn. *American Journal of Botany* 88 (9): 1704-1706.
- [33] Sobuj SR. N.d. Fiber Length and Length Distribution [Internet]. Available from: <https://textilestudycenter.com/ber-length-lengthdistribution/>
- [34] Tahir P, Ahmed A, Syeed OA. 2011. Retting Process on Bast Fibers and its effect on Fiber Quality: A Review. *BioResources* (**Edition**) [Internet]. [**Last Updated**], cited 2017 October 17] 6(4):5260-5281. Available from: <http://ojs.cnr.ncsu.edu/index.php/BioRes/article>
- [35] Thakur VK. 2014. *Lignocellulosic Polymer Composites: Processing, Characterization, and Properties* [Internet]. Beverly(MA):Scrivener Publishing; [cited 2016 Mar 24]
- [36] Thomasson SC. [Internet]. 2013. *The Philippines: Textile and Apparel Industry on the Mend*. Billian Publishing Inc. [updated 2015; cited 2013]. Available from: <http://www.textileworldasia.com/issues/2013/January-FebruaryMarch/Country-Proles/The-PhilippinesTextile-and-Apparel-Industry-on-the-Mend>
- [37] Torres CS, Romel A, Daya MTB, Gopela, JN. 2013. *Adoption and Uptake Pathways of GM/Biotech Crops by SmallScale, Resource-Poor Filipino Farmers*. Los Baos, Laguna, Philippines: College of Development Communication, International Service for the Acquisition of Agribiotech Applications (ISAAA) SEAsiaCenter, SEAMEO
- [38] Southeast Asian Regional Center for Graduate Study and Research in Agriculture (SEARCA). 90p.
- [39] [USDA] United States Department of Agriculture n.d..Harvesting Retting and Fiber Separation [Internet]. *Industrial Hemp in the United States*; [cited 2017 October 11]. Available from: <http://www.globalhemp.com/wpcontent/uploads>
- [40] Whewell C. Textile [Internet]. Britannica; [2015, cited 2015 Nov 15]. Available from: <http://www.britannica.com/technology/textile> Willbanks A. [Internet]. 2009. The importance of textiles. *Textile Fabric Consultants, Inc.* [cited 2009]. Available from: <http://www.textilefabric.com/site/main/articles.php?id=26>.
- [41] Yilmaz ND. 2013. Effect of chemical extraction parameters on corn husk fiber characteristics. *Indian Journal of Fibre and Textile Research* 38: 29-34.
- [42] Yilmaz, ND, Caliskan E, Yilmaz K. 2013 Effect of xylanase enzyme on mechanical properties of fibers extracted from dried and undried corn husks. *Indian Journal of FiberTextile Research* 39: 60-64.
- [43] Yussuf AA, Massoumi I, Hassan A. 2010. Comparison of Polylactic Acid/Kenaf and Polylactic Acid/Rice Husk Composites: The Influence of Natural Fibers on the Mechanical, Thermal and Biodegradability Properties. *Journal of Polymers and the Environment* 18 (3): 422-429.
- [44] Asim M, Abdan K, Jawaid M, Nasir M, Dashtizadeh Z, Ishnak M, Hoque M. 2015 A Review on Pineapple Leaves Fibre and Its Composites. *International Journal of Polymer Science*. Available from: <https://www.hindawi.com/journals/ijps/2015/950567/>