Comparison in yield of the microwave-assisted method and conventional method in *Zea mays* var. *ceratina* (glutinous corn) cobs cellulose fiber extraction

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Abstract

Cellulose is a biopolymer that is abundant in nature, often extracted from raw materials via the conventional method. However, a new method of fiber extraction called the microwave-assisted method is said to be cost and energy-efficient. In this study, cellulose fibers were extracted from *Zea mays* var. *ceratina* cobs via microwave-assisted method (MAM) and conventional method (CM), wherein the yield means of the two methods were determined and statistically compared. *Z. mays* cob powder was subjected to 8% (w/v) NaOH for the alkalization process, 5% (w/w) H_2O_2 for the acid hydrolysis, and heated using a microwave oven and hot plate for MAM and CM, respectively. The results showed that the MAM and CM yielded 72.7% and 44.6% cellulose fibers, respectively. Statistical analysis via Mann-Whitney U test showed that there is an observed trend towards MAM yielding a higher percentage of crude cellulose in contrast to CM.

Introduction. - Cellulose is a biopolymer that is abundant in nature, renewable, and biodegradable, making it a potential industrial material [1]. Synthesized cellulose fibers are utilized in various applications such as in textiles, paper, packaging, building materials, and synthetic fibers [2].

For the past several years, climate change and the additional problems involving agricultural waste urge industries to utilize natural sources instead of synthetic materials. According to Zafar [3], the Philippines is an agricultural country that consists of a land area 30 million hectares wide, wherein 47% of which is utilized in the agricultural sector. Due to this, agricultural residues are also abundant which are common sources of renewable materials and most of them are considered as waste materials. Cobs are by-products derived from *Zea mays*, more commonly known as corn plants. In 2020, the Philippine corn production was about 8.12 million metric tons [4]. Subsequently, the corn cob waste by-product had an estimated technical volume of about 1.95 million metric tons, with most of it being discarded or burnt [5]. This resulted in environmental damages such as global warming due to greenhouse gas emissions (e.g. carbon dioxide, methane) [6,7]. Corn cobs, however, have the potential to become a cheap and abundant raw material for cellulose extraction, as corn cobs are primarily composed of cellulose, hemicellulose, and lignin [2]. The cobs typically have a cellulose fiber content of around 40–44% [8].

Cellulose extraction is the process of isolating cellulose from raw materials or natural fibers. It generally involves the use of alkalis or bisulphites for the treatment of the fibers to isolate lignin and obtain the hemicellulose. There are three primary methods used in extracting cellulose: alkaline treatment, bleaching, and acid hydrolysis [9]. Moreover, cellulose can be extracted using different kinds of methods such as the conventional and microwave-assisted. Every procedure has different pros and cons associated with the yield and properties of the cellulose [10].

The conventional method typically involves the use of instruments that heat the walls of reactants through conduction or convection [11]. Most studies use the conventional method for extracting cellulose fiber because it is simple, effective, and has a high fiber yield [12,13]. However, the process is often slow since it requires high external temperatures to generate the required heat. Furthermore, the rate of the heat flow into the body of the material from the surface limits the process time. Moreover, the heat is also uneven since some parts such as the surface, edges, and corners tend to be hotter in contrast to the interior of the material [11].

Contrasting this, a new method of fiber extraction, called the microwave-assisted method, reportedly yields analytes with higher purity compared to the conventional method [11].

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Microwave heating is an alternative method in extracting bio-based materials due to its capability to accelerate chemical processes. As compared to the conventional method, microwave-assisted method is more rapid and volumetric due to the direct interaction between the material subjected and the heat generated by the applied electromagnetic field. Additionally, it is highly selective, uniform, and utilizes less amount of energy for the pretreatment of bioproducts [2].

With the above information in mind, cellulose from glutinous corn cobs was extracted using the microwave-assisted and conventional methods. The resulting yield obtained from both methods were compared and analyzed using Mann-Whitney U test to determine if a significant difference exists in terms of yield. Microwave-assisted mechanism can be implemented in the production of cellulose obtained from corn cobs, specifically *Zea mays* var. *ceratina,* to address problems such as expensive cost and longer duration of the extraction process, as well as the lack of investigation regarding the comparison of yields between the microwave-assisted and conventional methods.

This research aimed to compare the yields of the microwave-assisted method and conventional method in extracting cellulose fibers from *Zea mays* var. *ceratina* cobs. Specifically, it aimed to:

(i) measure the percent yield of the extracted cellulose fibers from corn (*Z. mays* var. *ceratina*) cobs via microwave-assisted chemical extraction method;

(ii) measure the percent yield of the extracted cellulose fibers from corn (*Z. mays* var. *ceratina*) cobs via conventional method;

(iii) determine a significant difference among yield means of crude cellulose extracted via microwave-assisted and conventional methods, respectively, using Mann-Whitney U test, and;

(iv) qualitatively assess the presence of cellulose in the extracted analytes through the development of a purplish hue upon addition of the Schultze's reagent.

Methods. - This study is descriptive in nature. *Z. mays* cobs cellulose fibers were extracted using two extraction methods: microwave-assisted method (MAM) and conventional method (CM). Cellulose fiber extraction is mainly composed of two main processes: alkalization and acid hydrolysis. Ground corn cob powder was subjected to alkalization using sodium hydroxide (NaOH), then followed by acid hydrolysis using hydrogen peroxide (H_2O_2). Three replicates per setup were extracted and percentage yield means were calculated. Extraction processes were then followed by the determination of whether the analyte contains cellulose or not using Schultze's reagent. A purplish color upon contact would then signify the presence of cellulose. In determining whether the percentage yield means are significantly different from each other, the non-parametric statistical analysis tool Mann-Whitney U test was used.

Zea mays Cobs Acquisition, Authentication and Storage. Glutinous corn cobs (*Z. mays* var. *ceratina*) were purchased and collected from a local corn vendor located in Q. Abeto St., Mandurriao, Iloilo City. Corn variety was authenticated by the Department of Agriculture - Iloilo Research Outreach in Sta. Barbara, Iloilo, to which an official certification was issued.

Acquired corn cobs were washed prior to sun-drying. This is to ensure the safety of the researchers and the residents of the household wherein the study was conducted in. The obtained corn cobs were sundried outdoors for 48 hours to remove moisture. The cobs were also turned every six (6) hours to ensure that every part was evenly dried. The corn cobs were stored in an airtight container and kept in a place away from direct sunlight at room temperature to prevent dry matter loss during nighttimes, prior to usage [14].

Zea mays Cob Preparation. Corn cobs were pulverized into fine powder using a blender (Moulinex Turbo Blender). The powder was then sieved using a fine mesh sieve (pore size: 841 μm). Larger particles were reground using a mortar and pestle to enable passage through the sieve. The powder used for all treatments was a mixture of all grounded cobs. Powdered samples were dried in an oven (La Germania SL6031-21) at 135 °C until a constant weight was achieved. Constant weight was determined through the continuous drying and hourly weighing of samples until two consecutive weighings did not differ by more than 0.5 mg per gram of the sample initially taken [15]. Prior to the hourly weighing of heated samples, samples were cooled to room temperature. Ten (10) grams of *Zea mays* var. *ceratina* powder from each replicate were then placed inside airtight containers. They were then stored at room temperature until further use.

Cellulose Extraction via Microwave-assisted Method. Microwave heating was conducted in a 2450 MHz microwave oven (Hanabishi HMO-17M-3), wherein the power per microwave-assisted heating was set to 500W, three (3) minutes per replicate [2,16].

The corn cob powder samples underwent alkalization using NaOH as the alkaline treatment, wherein ten (10) grams of the corn cob powder was added into 150 mL 4% (w/v) NaOH, then subjected to microwave heating with the specified settings. The acquired corn cob pulp was washed with distilled water until neutral pH (around 6.5–8) was acquired, measured using pH test strips [16,17]. After the alkalization process, acid hydrolysis was performed. Here, the alkalized corn pulp was bleached in 50 mL 5% (w/w) H_2O_2 solution. The pulp was then subjected to microwave heating following the same settings stated above. Extracted cellulose fibers were then rewashed until neutral pH was acquired. To rid the analyte of water, it was dried using the oven until a constant weight was achieved [2,18]. Three (3) replicates were used in this step.

Cellulose Extraction via Conventional Method. Corn cob powder was subjected to alkalization then heated using a hot plate equipped with a temperature reader (Biobase MS7-NS50-Pro), wherein the beaker with the alkaline treatment was placed on the hot plate set at 100 °C for four (4) hours with constant stirring. After washing until neutral pH was acquired, the pulp was then submerged and hydrolyzed in the H_2O_2 solution. Extracted cellulose fibers were then subjected once more to washing until neutral pH was acquired. To rid the analyte of water, the analyte was dried using the oven until a constant weight was achieved [18]. Three (3) replicates were used in this step.

Qualitative Determination of Presence of Cellulose using Schultze's Reagent. Extracted cellulose fibers were assessed qualitatively using Schultze's reagent to determine the presence of cellulose in the extracted analyte. In preparing the reagent, twenty (20) grams of zinc chloride ($ZnCl_2$) was dissolved in 9.5 mL warm distilled water then cooled. On a separate beaker, 0.5 g iodine and 1 g potassium iodide were dissolved in 20 mL deionized water, wherein 1.5 mL of this solution was added to the zinc chloride solution until a persistent precipitate of iodine formed. Direct contact of the Schultze's reagent to cellulose fibers would yield a purplish color [19,20].

Computation of Percentage Yield. In the computation of percentage yield, the following formula was used:

$$\%yield = \frac{acquired\ dry\ mass\ of\ cellulose\ fibers\ (g)}{dry\ mass\ of\ raw\ material\ used\ (g)} \times 100$$

The mean percent cellulose fiber yields were then calculated. The calculated mean—one for each method—served as the overall percentage of cellulose fiber yield.

Data Analysis. Mann-Whitney U test was utilized in determining whether the percentage yield means are significantly different from each other, wherein in this process, the equation function of Microsoft® Excel® for Microsoft 365 MSO (16.0.13929.20360) 64-bit. The data would be then deemed significantly different if $p<0.05$ [21].

Safety Procedure. A Materials Safety Data Sheet (MSDS) was secured and provided beforehand wherein hazards regarding the conduct of this study were determined. Mitigation of these hazards were subsequently observed and implemented. Laboratory protective gear consisting of laboratory gowns, gloves, and masks were worn at all times during the conduct of the study.

Results and Discussion. - Crude cellulose fibers were extracted from *Zea mays* var. *ceratina* cobs via MAM and CM. Schultze's agent was then added to the extracted analyte to determine the presence of cellulose. The resulting purple hue signified that the extracted analyte contains cellulose. This is shown in Figure 1. However, a greenish hue was also observed, signifying the presence of non-cellulosic material such as lignin.

Figure 1. Crude cellulose extracted via CM (left) and MAM (right) were tested for analyte validity using Schultze's reagent, to which the purplish color indicated the presence of cellulose.

Furthermore, the crude cellulose extracted via MAM are higher in contrast to the ones extracted via CM, as shown in Table 1. Additionally, replicate yields and means per setup along with their respective standard deviations are presented in Figure 2.

Table 1. The table below shows the percentage yield means and medians per setup.

Setup	Yield mean (%)	Yield median (%)
MAM	72.7	72.0
CM	44.6	42.0

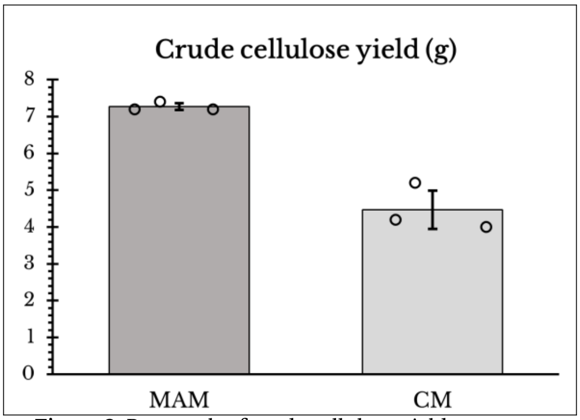

Figure 2. Bar graph of crude cellulose yields per setup.

The higher crude cellulose yield through MAM extraction may be attributed to the uniformity of heat generated by the emitted low-frequency radiation of the microwave [17]. Reactants and solvents are heated rather than the container itself as the radiation passes through the walls of the container, exciting the polar molecules within the substance being heated. Heat is generated through the movement of these polar molecules. This leads to less by-products and decomposition products which enabled the increase of the yield percentage [17,22].

In contrast, heating using CM is prone to temperature gradient which results in overheating and eventually product decomposition leading to a lower yield percentage [22]. The 2015 study of Garadimani et al. [23], which used the conventional method of extraction, found that the extraction of *Z. mays* cobs cellulose fibers yielded a mean of 41.5% of cellulose. In terms of yield, this is close to the findings of the current study using the same method.

Calculations showed that the p-value of the data acquired from both extraction methods is 0.049535. Though less than the significance level of 0.05, a clear significant difference is not apparent; therefore, the p-value only signifies a trend towards MAM extracting a higher amount of crude cellulose in contrast to CM.

Limitations. Due to time and resource constraints, this study only compared the methods based on only one parameter. This is due to the lack of equipment as the study was conducted during a pandemic.

Conclusion. - Findings show that the MAM and CM were able to extract 72.7% and 44.6% of crude cellulose, respectively. The purple hue that developed upon the addition of Schultze's reagent signifies the presence of cellulose. However, the development of a greenish hue signifies the presence of non-cellulosic material within the extracted analytes. Through statistical analysis, it was determined that there is an observed trend towards the microwave-assisted method yielding a higher percentage of *Zea mays* var. *ceratina* crude cellulose in contrast to the conventional method.

Recommendations. - Future related studies are recommended to include more parameters such as thermal stability, morphology, and crystallinity for comparison of MAM and CM [2]. Moreover, in contrast to the Schultze's reagent, the usage of the Van Soest Analysis would provide a quantitative result with regards to analyte purity [24,25]. Furthermore, future studies are recommended to explore more on the non-thermal effects of microwave heating as well as the effects of microwave heating modes (e.g. intermittent, continuous) in biopolymer extraction from raw materials.

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