Fiber decomposition and pretreatment analysis of *Cannabis sativa L.*: Hemp

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Abstract:

Hemp is a subspecies of *Cannabis sativa L.* along with marijuana, yet the two differ in chemical constituent levels of delta-9-tetrahydrocannabinol (THC) and cannabidiol (CBD). Hemp contains 0.3% THC, compared to marijuana 17.1%, allowing it to be a safe and compelling biomass for investigation. The refined products of hemp are vast due to its fast-growing properties; therefore various commercial industries have included refined hemp in biofuels, biodegradable plastics, textiles, dietary supplements, paper, clothing, and much more. Construction and manufacturing applications have also been seen to include hemp to strengthen their composite products. The high-yielding, sustainable, and environmentally friendly qualities of hemp have the potential to yield valuable raw materials for a great number of applications. Hence, our research seeks to evaluate the suitability and the potential use of ionic liquid-based pretreatment (1-Butyl-3-methylimidazolium chloride) for the breakdown of hemp lignocellulosic biomass. Using past collected data from our research, we hope to cross-examine through stereomicroscopic analysis to affirm if a consistent trend is observed across pretreatment stages for our samples. All collected data is presented and discussed in the following sections.

Introduction:

Cannabis sativa L., hemp, a popular sustainable fiber for production contains 0.3% THC, compared to its relative marijuana with 17.1% THC, this allows it to be a safe and compelling raw material. There are many factors that positively support hemp's resurgence in the textile, agricultural, pharmaceutical, and fuel industries. These factors include hemp's natural properties that allow it to replenish poor soil, thrive with little assistance, and grow without the need for pesticides, fertilizers, and much water. Hemp's adaptable species can be sustained in harsh environmental conditions, and the environmental impacts associated with the production of hemp fibers are smaller than those associated with most other crops.

Hemp is classified as second-generation biomass due to its composition of lignocellulose and pectin. Lignocellulose is comprised of three polymers: cellulose, hemicellulose, and lignin. These polymers account for the structural stability, high strength, and stiffness of hemp's cell wall. Therefore, ionic liquids (IL) are investigated as a dissolving agent for hemp in this study. Dissolving biomass in ILs has been reported to lead to a full release of all the functional groups and bonds from the matrix. These results have shown that lignocelluloses dissolved in ionic liquids are more susceptible to chemical attack by various reagents/catalysts. This study aims to determine how efficient the use of ionic liquids is for the pretreatment of hemp's lignocellulosic material and evaluate the quality of fiber obtained thereof.

Procedure:

I. Biomass Preparation

The hemp used in this study was donated by SUNYrf. The hemp was washed in deionized water, chopped into three sizes (Ground ¼", Short ½", and Large 1"), and placed in an oven at 70 degrees Celsius for a total of three days to dry. Once dried, the "Ground" samples were ground with mortar and pestle to resemble a fine powder. Then 0.3g of each sample size was weighed using an analytical balance and distributed into Erlenmeyer flasks for pretreatment.

II. Ionic Liquid (IL) Pretreatment

The ionic liquid pretreatment employed for the hemp samples was 1-Butyl-3-methylimidazolium chloride. Each flask received 3.0 grams of 1-Butyl-3-methylimidazolium chloride along with a magnetic stirrer. A 1:3 ratio of IL to biomass was maintained for each sample. Flasks were heated in mineral oil baths in crystallizing dishes at 75-80 degrees Celsius for either 3, 6, or 9 hours. Once samples reached their target heating period, they were removed and left to cool.

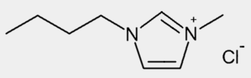

Figure 1. Chemical structure of 1-Butyl-3-methylimidazolium chloride

Raw	GH 3hr	GH 6hr	GH 9hr	SH 3hr	SH 6hr	SH 9hr	BH 3hr	BH 6hr	BH 9hr
Ionic Liquid 2020 (Top)									
2021 (Bottom)									
Acid Hydrolysis 2020 (Top)									
2021 (Bottom)									

Table 1. Stereomicroscope images of hemp from ionic liquid and acid hydrolysis stages.

Procedure Continued:

III. Acid Hydrolysis

After ionic liquid pretreatment, sample flasks were filled with 10.0 mL of 0.5 Hydrochloric acid and heated in mineral oil baths for 3, 6, or 9, hours at 80 degrees Celsius. Samples were left to cool for 30 minutes before adding 10.0 mL of 0.5 M sodium hydroxide to neutralize the acidic samples.

IV. Slide Preparation

Hemp fragments were collected at each treatment stage for fiber examination. Fragments were removed from each flask, rinsed with deionized water, and placed on glass microscope slides. Appropriate labels were marked for each slides: A for acid hydrolysis, IL for ionic liquid, and RAW for samples without any treatment; numerical labels were also noted for the hours spent in each treatment stage.

V. Stereomicroscope

Examination and imaging of sample slides were by the ZEISS SteREO Discovery.V20™ microscope. The objective lens applied for all images was the Achromat S 1.5x FWD 28mm. For optimal imagery, a z-stack was applied to properly account for the topography of the hemp fibers. Additionally, images underwent further processing methods such as "Extended View of Focus" to sharpen resolution, "White Balance" to adjust light reflection or both. Displayed images demonstrate two fields of vision of each hemp sample, a half and zoomed view. Optimal images for each sample in both treatment stages is presented.

Figure 2. Stereomicroscope

Results:

The complete results from our past 2020 and present 2021 experiments are displayed in table 1 to effectively demonstrate the efficacy of our experiment. The data confirms that the ionic liquid pretreatment of 1-Butyl-3-methylimidazolium chloride and acid hydrolysis of hydrochloric acid and sodium hydroxide, together further digest the hemp to produce increasing fibrous material. The data confirms the success our target IL method to be a promising pretreatment candidate for further studies on hemp.

Future Directions:

Future directions for this experiment would be to add an additional time period of 12 hours to both treatment stages and analyze if and how much variation is visible when doing a comparing between all four time periods. It is possible that the addition of another time period may demonstrate further increase in hemp fiber production.

Additionally, further investigation is possible by conducting Thermal Gravimetric Analysis. This analysis will record the mass of the sample over time as the temperature changes, providing information about the thermal decomposition and stability of our hemp.

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