Pretreatment and fiber decomposition analysis of *Cannabis sativa L*

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Abstract:

Hemp, along with marijuana, are subspecies of Cannabis sativa L. The two differ in chemical constituent levels of delta-9-tetrahydrocannabinol (THC) and cannabidiol (CBD).Hemp contains 0.3% THC, compared to marijuana's THC content of 17.1%, allowing it to be a safe and compelling biomass for investigation. Hemp is one of the fastest growing plants and its refined products have immense commercial value, including 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. Hemp is a high yielding, sustainable, and environmentally friendly crop due to its various qualities, and has the potential to yield valuable raw materials for a great number of applications. Our research evaluates the pretreatment of hemp as well as the comparative analysis of the fiber content with the goal of determining the suitability and the potential use of ionic liquid-based pretreatment (1-Butyl-3-methylimidazolium chloride) for the breakdown of hemp lignocellulosic biomass. The collected data is presented and discussed in the following sections.

Introduction:

Cannabis sativa L, commonly referred to as hemp, is a popular sustainable fiber used in many agro-industrial applications. As a species of the Cannabis family, hemp contains 0.3% THC compared to marijuana with 17.1% THC, allowing it to be a safe and compelling raw material. Hemp has a high potential to be used for a great number of applications such as in the textile, agricultural, pharmaceutical, and fuel industries due to many factors. 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 is an adaptable species that 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 comprises three polymers: cellulose, hemicellulose, and lignin. Together, these polymers account for the structural stability, high strength, and stiffness of hemp's cell wall. Due to the cell wall's structural composition, ionic liquids were 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. Therefore, this study seeks to evaluate efficiency of an ionic liquid-based pretreatment (1-Butyl-3-methylimidazolium chloride) for the breakdown of hemp lignocellulosic biomass 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 18 hours. Once samples reached their target heating period, they were removed and left to cool.

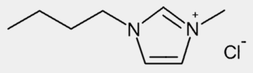


Figure 1. Chemical structure of 1-Butyl-3-methylimidazolium chloride

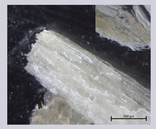
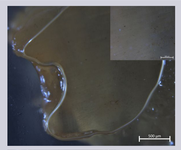
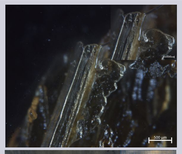
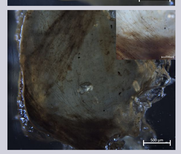
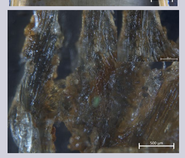
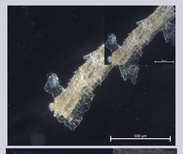
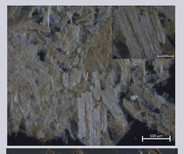
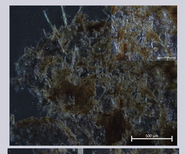
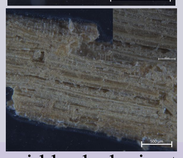
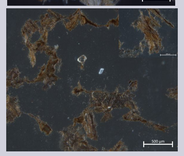
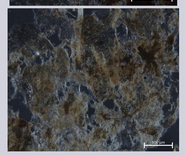
Raw 	GH 18hr	SH 18hr	BH 18hr
Ionic Liquid Trial 1 (Top)			
Trial 2 (Bottom)			
Acid Hydrolysis Trial 1 (Top)			
Trial 2 (Bottom)			

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 18 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 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 data presented in table 1 shows an increase in fibrous material after each treatment step. Additionally, the data shows that an 18 hour acid hydrolysis treatment is too long as the hemp fibers in certain samples were degraded completely after acid hydrolysis. Overall, the data confirms the success of our target ionic liquid method to be a promising pretreatment candidate for further studies on hemp.

Future Directions:

Future directions for this experiment would be to decrease the acid hydrolysis treatment time and analyze if this would result in better fibrous material for analysis. Comparisons from past data with future results will allow for a more in depth understanding of differences in fiber breakdown after ionic liquid and acid hydrolysis treatment.

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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