Conference Paper

Hemicellulose Extraction and Characterization of Rice Straw and *Leucaena Leucocephala*

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Abstract

Local production of agricultural waste is increasing. It is not fully utilized and can cause an environmental issue if it is not handle wisely. Thus, it is important to increase utilization of lignocellulosic biomass by improving their added value and subsequently decrease the agriculture waste. In this study, rice straw and *Leucaena leucocephala* were subjected to alkali treatment (4% sodium hydroxide) with different concentration ratio of samples to sodium hydroxide (1:10 to 1:50). The physical and chemical properties of extracted hemicelluloses were studied. The yield of hemicellulose was higher from rice straw compared to *Leucaena leucocephala*. The chemical functional groups present in hemicellulose were confirmed by Fourier tranform infrared spectroscopy (FTIR). The surface morphology and roughness of xylan were examined by scanning electron microscopy (SEM).

Keywords: rice straw, hemicellulose, sodium hydroxide, alkali

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1. Introduction

In recent years, scientist has highlighted agro-industrial waste as important renewable sources to be explore due to their beneficial and promising potential. Many researchers show interest in agricultural biomass study because its practical applications in various agro-industrial process, for example, the conversion of hemicellulosic biomass into fuel or chemicals (Rabetafika, et al., 2014), renewable fuel (Said, et al., 2013), bioethanol (Moradi, et la., 2013; Park et al., 2010) and biobutanol (Ranjan, et al., 2013). Malaysia produce substantial amount of agricultural biomass such as rubber (39.67%), oil palm (34.56%), rice (12.68%), cocoa (6.75%) and coconut (6.34%). Most of the residues has been disposed by burning and only 27% of agriculture residue are used as value added products(Zafar, 2015).

Hemicellulose are the second most abundant polysaccharides and content from 17%-35% of the dry mater lignin (Hassan et al. 2013; Karimi, Kheradmandinia, and

Taherzadeh 2006; M. J. Feria et al. 2011). Hemicellulose become one of the alternative in agriculture waste because of its practical applications in various agro-industrial process, for example, conversion of hemicellulosic biomass into fuel or chemicals (Rabetafika et al. 2014), renewable fuel (Said et al. 2013), bioethanol (Moradi et al. 2013; Park et al. 2010) and biobutanol (Ranjan, Khanna, and Moholkar 2013).

There are many alternative methods of isolating hemicellulose from plant cell walls of lignocellulosic materials such as organic solvent extraction (Y. C. Sun et al. 2011), hydrothermal treatment (S. L. Sun et al. 2013) and ionic liquid extraction (Froschauer et al. 2013), and alkali treatment. However, alkali treatment is the mostly used in industrial and laboratory practices in isolating hemicelluloses. However, there are few studies on the optimum extraction ratio of alkaline. Hence, this study aims to investigate the optimum parameters that could be use in hemicellulose extraction to produce the highest yield of hemicellulose. The biomass was treated with 4% alkali with ratio of 1:10 to 1:50 respectively.

2. Methodology

2.1. Sample Preparation

Leucaena l. was collected from nearby university campus, cut and chopped into small form before transported to the laboratory. The rice straw was obtained from rice field at Sekinchan, Selangor. All samples were dried and chopped into smaller size before ground and milled using Wiley Mill machine. The samples were sieved into 100 meshes and kept in airtight container for further analyses.

2.2. Chemical composition of samples

Extractive free wood was adopted from TAPPI T204om-97, cellulose method was adopted from James and Jeffrey (1996), lignin method was followed by TAPPI T222 om-02 method and Proximate analysis was adopted from AOAC method (AOAC, 2005).

2.3. Extraction Process

Prior to extraction process, the samples were going through defatting process to remove the fat in the samples. EFB powder was treated with 80% ethanol (1:10 w/w) for 18 hrs. The sample was then wash with tap water and dried in the oven at 45°C overnight. The isolation of hemicellulose was conducted according to the method of (Chimphango, et al., 2012) with slight modification. Defatted samples were soaked in 4% sodium hydroxide in ratio of 1:10, 1:20, 1:30, 1:40 and 1:50 and agitated in water bath at 65°C for 1 hr. The sample was then wash with warm water (2L) and the supernatant was collected. The solution was then centrifuged at 6000g for 5 min to remove the large particle in the solution before filtered using disc filter funnel. The solution was adjusted to pH 5 using acetic acid and concentrated with rotary evaporator. Ethanol was added to the solution to precipitate the hemicellulose. The sample was centrifuged at 6000 g for 5 min and the precipitate was collected and oven dried overnight at 55°C before ground and kept for further analyses.

2.4. Scanning Electron Microscopy

Morphology study of samples was carried out using scanning electron microscope (SEM) (model LEO 1450VP). Biomass samples were collected before and after pretreatment. The collected samples were oven dried at 40 °C for 3h prior to sample preparation. The samples were fixed on aluminium stubs and coated with gold- palladium. Finally, the morphology of sample was observed under SEM at magnification power in range 100-2000×.

3. Result and Discussion

3.1. Chemical composition

Table 1 below shows chemical compositions of rice straw and *Leucaena l.*. Rice straw and *Leucaena l.* shows significant amount of cellulose (47.4% and 53.02%), hemicellulose (22.77% and 37.88%) and lignin (30.98% and 19.11%). This value is in agreement with other researchers (Cabrera et al., 2014; Manuel J. Feria et al., 2012; Sreekala). Meanwhile, all subjected samples have high value of hemicellulose, which is important in order to optimize the extraction method. Furthermore, high content of ashes in rice straw is due to existence of silica content (Said et al. 2013). The higher content of ash is agreed with result obtained by (Akinfemi and Ogunwole 2012; Said et al. 2013). Other than that, fat content and extractive free content of rice straw and *Leucaena l.* are 9.89%, 5.4% and 4.2%, 3.70% respectively. Higher content of fat and extractive in samples could interfere the extraction process. After defatting process, fat content of each sample decreased to 87%-70% (data not shown).

Table 1: Chemical composition of rice straw and *leucaena l.*

Sample	Rice Straw	Leucaena l.
Cellulose (%)	53.02±2.08	47.41±1.58
Hemicellulose (%)	22.77±7.94	37.88±4.42
Lignin (%)	30.98±6.1	24.28±1.71
Ash (%)	12.00±0.57	0.50±0.42
Fat (%)	1.70±0.01	1.60±0.10
Extractive free (%)	4.20±0.85	3.70±0.71
Moisture content (%)	7.5±0.24	6.5±0.71

3.2. Extraction of hemicellulose

Rice straw and *leucaena l.* have been treated with 4% NaOH in different ratio (1:10 to 1:50). Figure 1 shows the hemicellulose yield obtained from different ratio of sodium hydroxide. Hemicellulose yield obtained from rice straw is higher as compared to *leucaena l.* Different ratio of sample to NaOH will give different yield of hemicellulose for both samples. It is observed that lower ratio (1:10) gives more yield than higher ratio of NaOH (1:50).

Alkaline pre-treatment at temperate temperatures is a common method extracting hemicelluloses from biomass without severe degradation (Panthapulakkal, Pakharenko, and Sain 2013). Alkali hydrolyses the ester linkage between plant polysaccharides and lignin, which releases polysaccharides and increases their solubility of polysaccharides, without reducing their molar mass, under moderate reaction conditions. Hydroxyl ion can cause swelling of cellulose, disruption of hydrogen bonds between cellulose and hemicelluloses, and hydrolysis of ester bonds most likely connecting cell wall polysaccharide, resulting the solubilisation of substantial amounts of hemicelluloses and lignin (R. C. Sun and Sun 2002). Figure 2 shows how solubilisation occurs during NaOH treatment.

3.3. Scanning Electron Microscopy

Surface morphology of rice straw and *leucaena l.* were performed by scanning electron microscopy (SEM) (Leo 1455 VP SEM, made in USA). Figure 3 (a and c) shows surface morphology of rice straw, *leucaena l.* without any treatment. Morphology of native rice straw shows the existence of silica cells that were uniformly deposited on the exterior surfaces with a regular pattern (Figure 3a). However, there are only a few silica cells have been spotted in native *Leucaena l.* Silica in rice straw can provide protection from fungal diseases and provides physical support for paddy.

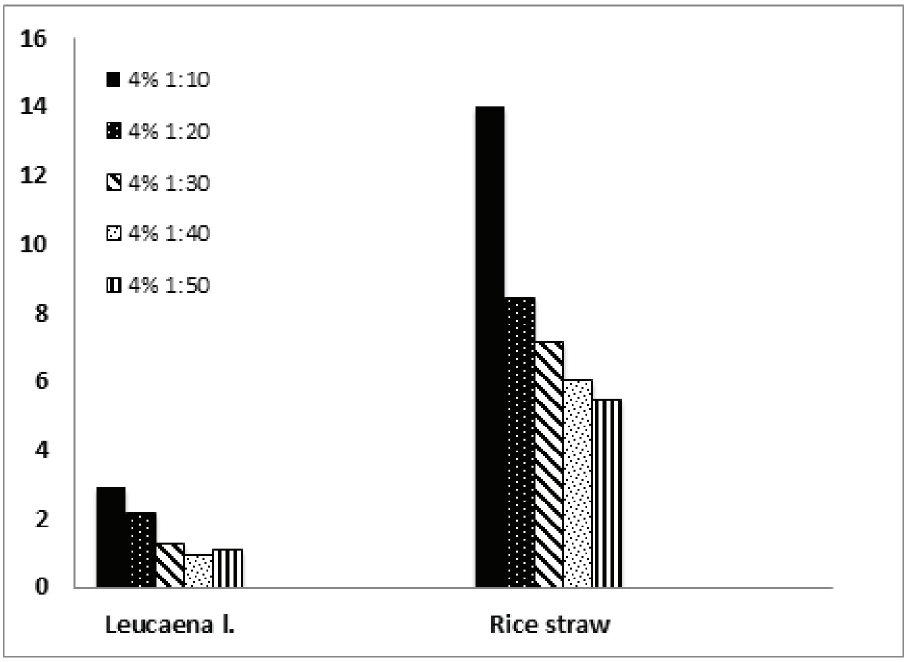

Figure 1: Yield of hemicellulose extracted with 4% sodium hydroxide with different concentration ratio

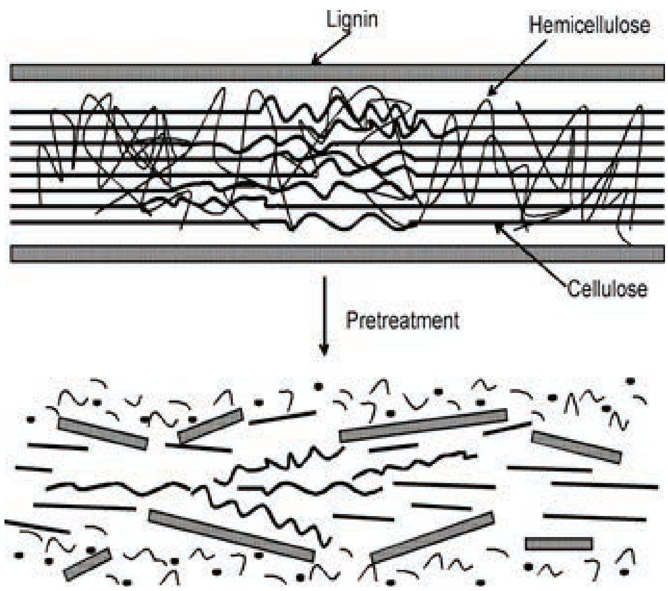

Figure 2: Effect of alkali pre-treatment of in various lignocellulosic (Hsu, T.A., Ladisch, M.R., Tsao 1980)

However, rice straw and *leucaena l.* surface morphology changes after treated with alkali. The alkali-treated samples shows grape-like structure rougher surface. The rigidity and order of the molecules were lost and micro fibrils and cracks were observed. This indicated that the increased of porosity on these fiber cells, might improved the exposure of cellulosic material for effective bioconversion (Shetty et al. 2017).

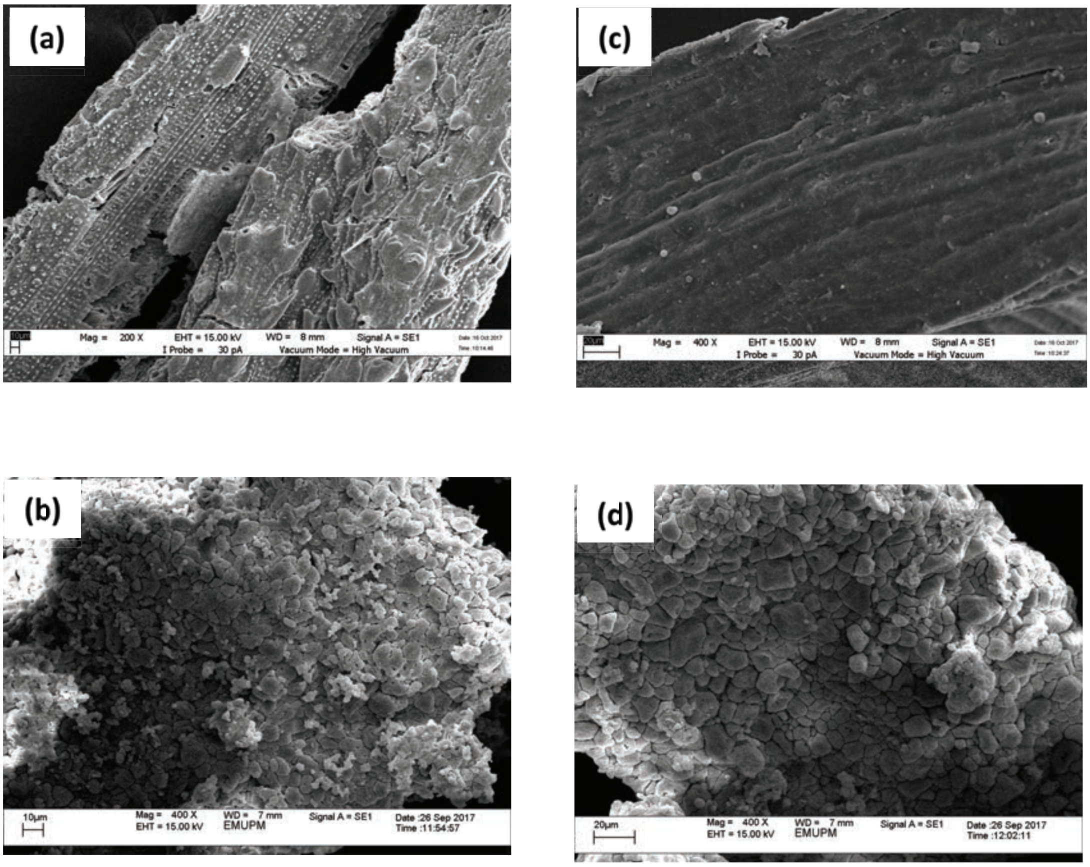

Figure 3: (a) untreated rice straw (b) 4% alkali treated (1:10) rice straw (c) untreated leucaena l. (d) 4% alkali treated (1:10) *Leucaena l.*

4. Conclusion

As for conclusion, the chemical composition of rice straw and *Leucaena l.* has been measured. Both plant shows high amount of hemicellulose (22.77% and 37.88%). High amount of hemicellulose will facilitate the extraction process. Furthermore, higher amount of ash content in rice straw describe the existence of silica. Alkali treatment (4% NaOH) with different ratio of alkali: sample (1:10, 1:20, 1:30, 1:40, 1:50) has been used for extraction of hemicellulose from rice straw and *Leucaena l.* Highest amount of hemicellulose were obtained from rice straw (1:10). It is also observed that different amount of samples has been obtained from different ratio of sample to NaOH. Scanning electron microscope also shows the changes of fiber arrangement. Rougher surface with disoriented order and rigidity has been observed on the alkali treated samples. Thus indicated that alkali treatment can increased the porosity of fiber cells and subsequently improved the conversion of hemicellulose to another value added products.

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