Operability Analysis on Upscaled Stirred Tank for Cellulose Recovery from Red Meranti Wood Sawdust

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Abstract—Red Meranti wood sawdust (RMWS), a waste lignocellulosic biomass produced from forest activities, mostly from timber industries is the good source of cellulose. RMWS consists of three major components of polymer, which are cellulose, hemicellulose, and lignin. The huge amount of sawdust particles produced are typically combusted, relinquished or dumped into rivers, streams or drainages. Production of cellulose by chemical, physical and biological pretreatments processes of biomass has been proposed and studied in recent years to handle the environmental issues. Yet, just a few of them appear to be encouraging. This research aimed to bring the potentials scale up a plant from laboratory scale works to a 100l pilot scale of cellulose recovery process, in term of quality attributes to the overall system. The procedures involved in scaling-up the capacity by running the preliminary run using the upscaled stirred tank and identification of operability issues with proposed rectifications for new design features. Then, the rectified issues and the whole process are expected to run in new design of an intensified and improved batch reactor tank for commercialization later.

Keywords—Red Meranti Wood Sawdust; Cellulose; Operability; Stirred Tank

1. INTRODUCTION

As a top world tropical timber producer, Malaysian wood-based industry was experienced remarkable economic growth in past two decades. Malaysian commercial hardwoods range from heavy and medium weight construction timbers to fine furniture woods. The popular Malaysian species wood are Red Meranti, Cengal, Gerutu, Keruing, Merbau and more. Red Meranti wood or the specific name, Shoreaspp is an inexpensive raw material. Typically, its color is dark reddish or purplish brown; commonly with white resin streaks present. Red Meranti has been very commonly used in Southeast Asia region. Recently, it was studied to be used as an absorbent [1].

Malaysia is a blessed country where biomass availability is abundant and generally cheap. Nationwide, the biomass resources could originate from forestry residues, agricultural wastes, dedicated crops and municipal solid wastes. Forestry products, for example, the timbers, in particularly have various important usages in many sectors that include construction, transportation, furniture, energy, and so on. Depending on their purposes, timbers will be cut, grind, drilled and sanded, and these activities will produce sawdust as a by-product. Approximately, half of the log volume can be converted into timber during a sawing process. The rest of the log volume are converted into chips, sawdust and bark.

The sawdust is flammable and can cause a fire hazard to the manufacturing facility. Despite its typical usage is to produce particleboard, scientific efforts are exploring to find the other potentials of the sawdust. Products that could be generated from the sawdust are renewable absorbent as mentioned by [1], mulch, fuel and also to produce cellulose. Specifically, cellulose is a purely organic polymer, consisting repeating monomers of anhydroglucose that held together in a giant straight-chain molecule by the β-(1,4)-glycosidic linkages group [2].Most of lignocellulosic plants are consist of three components, which are cellulose, hemicellulose and lignin, made up commonly at secondary cell walls of plants [3, 4].

In turn, cellulose can be used to produce various value-added products such as nanocrystalline cellulose, carboxymethyl cellulose, acetate, nitrocellulose, hydroxyethyl cellulose and nanofibrillated cellulose Furthermore, cellulose from its glucose monomer could be used to produce alcoholic sugar, called as sorbitol. Having said that, these green materials that are produced from biomass resources are products of the future considering non-renewability of fossil fuels as the main energy, chemicals and materials currently. Hence, it is sensible at this point to consolidate efforts to harness cellulose as renewable feedstock and this research is going to recover cellulose in a pilot scale system. The challenge is to ensure that the operability is achieved by the final design and improved from the previous condition. Operability is the ability to retain and ensure the whole processes including the equipment, devices, system and procedures in a safe and reliable functioning condition. In addition, the entire process also has the capacity and flexibility to achieve a range of operating conditions profitably and excellent performance and product quality [5]. Previously, the engineer and inventor was aim to design and fabricate the system that would function well without focus on other side effects, especially in long term effects. Nowadays, their evolution has changed over longer life span, particularly in scale up the complex system. Understanding and working with designing frameworks demands consideration regarding properties that have longer exposure time. Attention to side effects and the big picture that establishes ground rules and restrictions within which systems operate is extremely important, as these factor are part of the systems [6] For this paper, the aim was about how the system is suppose to be or specifically referred as quality attributes of cellulose recovery system, the research was starting with the identification of typical issues in cellulose recovery such as type of raw material, selection of pre-treatment, mixing imperfection, liquid solid ratio, inefficient filtration, product discharge, improper hazardous gas emission treatment using the existing 100L pilot batch stirred tank and then, improve all into a new design of reactor combined with filtration and lifting systems.

2. METHODOLOGY

A. Red Meranti Wood Sawdust as Raw Material

Red Meranti wood sawdust is selected as a potential forestry residue known as lignocellulosic biomass resources. This selection based on the facts that this wood offers many advantages such as high yield production, availability and low input cost. The raw material that will be utilized is Red Meranti wood sawdust (RMWS) that was collected from Kilang Papan Sg Charu Sdn. Bhd, located at Sungai Lembing, Kuantan, Pahang. The RMWS harvested from Sg Lembing forest area. Figure 2.1, Red Meranti wood sawdust (RMWS) is one of the most cellulose-rich from lignocellulosic hardwood plant compared to the other types. Table 2.1 shows the composition of Red Meranti wood sawdust (RMWS) adapted from Forest Research Institute of Malaysia (FRIM) laboratory as a reference. The RMWS sawdust was taken by bulk for quality purpose.

Figure 2.1: Red Meranti Wood Sawdust as a raw material

Table 2.1: The composition of Red Meranti wood sawdust
Source: Forest Research Institute of Malaysia (FRIM) laboratory

Compositions	Percentage (%)	Test Method
Lignin	33.9	T222 om-02, acid Insoluble Lignin in wood and pulp*
Holocellulose	71.7	In-house method*
Cellulose	48.1	T203 os-74, Alpha, beta and gamma cellulose in pulp*
Ash	0.4	T211 om-02, Ash in wood, pulp, paper and paperboard*

B. Flow Diagram of Procedures

The overall process flow for this research focused on three major steps , which were preliminary run for cellulose recovery from Red Meranti Wood Sawdust using the best methodology from previous study, product analysis from preliminary run and the improvements in new design features of reactor system for higher recovery product, Cellulose, as Figure 2.2.

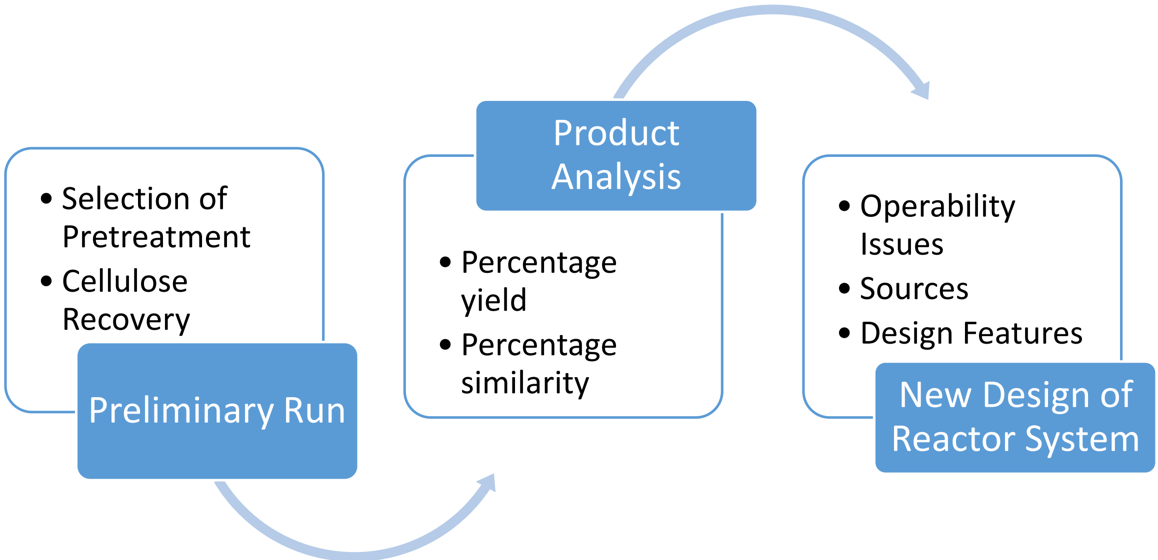

Figure 2.2: The flow diagram for operability analysis

C. Selection of Pretreatments

The goal of pretreatment is to make the cellulose reachable to hydrolysis by separating the lignin structure and disrupt the crystalline structure of cellulose [7]. This study will focus on producing the cellulose from RMWS using two-stage pretreatment processes; i) acidified chlorite delignification and ii) alternate alkaline treatment. Firstly, the methods to produce cellulose are screened and compared to select the suitable one for this study. For the preliminary run test, the procedure was selected from the combination of previous literature studies[8s,9,10], to make sure these procedures will recover the cellulose with high percentage yield and also the percentage of similarity between the sample and commercial standard analyzed from FTIR device tool. The percentage of yield is determined as follows:

$$Percentage\ Yield = \frac{Actual\ Yield}{Theoretical\ Yield} \times 100\%$$

During chemical hydrolysis, two processing steps involved in cellulose recovery. The primary step involves a delignification process. This method employs sodium chlorite ($NaClO_2$) to completely free the lignin from the RMWS. As an oxidizing agent, $NaClO_2$will generate chlorine dioxide in situ for bleaching as well as delignification of RMWS. With the addition of acid, particularly acetic acid (CH_3COOH) to an aqueous solution of sodium chlorite, it liberates the chlorine dioxide to reacts with sawdust to produce chlorite holocellulose. The subsequent step procedure involves the isolation of cellulose from holocellulose of RMWS by alkaline extraction method. The step will base on the alkaline solubility of holocellulose. In any isolation method, cellulose could not be obtained in pure form, but with the best quality which is generally termed as α-cellulose or cellulose. This type of cellulose is insoluble in a strong sodium hydroxide solution. The portion that is soluble in alkaline medium but precipitable upon a neutralized solution. The procedure uses the solubility power of certain percentage of sodium hydroxide to capture only theundissolved α-cellulose from the RMWS.

D. New Design of Reactor System

Clearly, before continue to design the new reactor, the source of variability was defined through preliminary runs by using the existing *100l* stirred tank equipment, as figure 3.1 in extracting cellulose from Red Meranti wood sawdust (RMWS), located at Specialty Chemical Laboratory, University Malaysia Pahang. From the preliminary result, the improvement to new reactor design features and the optimization of key operating parameters in laboratory scale will be done later.

3. RESULTS AND DISCUSSION

A. Preliminary Test Run

As figure 3.1 below, the tank testing was done for suitable process of cellulose recovery from Red Meranti Wood Sawdust (RMWS). Firstly, the test runwas done using water only to make sure the tank without leakage and all the mechanical and electrical was function properly such as the agitator, temperature probe, heater. From the evaluation, the valve at the bottom discharge was not suitable for the solid-liquid product. The valve was taking off and changed to direct outlet discharge with a valve. All the mechanical and electrical devices was functioningwell, and the tank was in good condition without any leakage.

The next testing was using chemicals and raw material referred to procedure from previous literature study to remove the lignin from 8 kg of Red Meranti Wood Sawdust (RMWS) and recovered the cellulose from it [8,9,10, 11]. The raw material and chemicals were prepared for delignification, extraction also neutralization processes to recover Cellulose from RMWS.

		
100*l*Stirred Tank	T-Joint with valve at bottom discharge	Direct bottom discharge

Figure 3.1: A water test run on Stirred Tank for Preliminary Run

Figure 3.2: Water inlet and mixing- reaction in delignification process

As figure 3.2 and 3.3 shows the delignification process of Red Meranti Wood Sawdust to produce holocelullose. At 1st trial, the mixture in the tank was not blended well because the agitator can spin at setting speed. At this time, only *50%* total volume used for the testing. When 2nd trial, the procedure was adjusted with increased the volume of solution and fixed the solid (RMWS) with total volume was *80%*. The agitator was operating as setting speed and the mixture was mixed well. In the delignification process, the smoke came out from the tank as a by-product. This issue needs to figure out, as a future solution is to link pipe from gas outlet to waste treatment for environmental and safety purpose. Once delignification process finished, the holocellulose was discharged through the bottom discharge outlet. The other issue was only liquid came out from the tank, the solid (holocellulose) cannot went through the small outlet discharge even the agitator was on to make sure no precipitation at the bottom tank, to prevent clogging at the discharge pipe. As a temporary solution, the mixture in the tank was diluted with water and the agitator kept on.At the same time, the manual hand pump sucked the mixture of liquid and holocellulose from top of the tank to other storage tank.

Figure 3.3: Process discharge and manual filtration process in delignification process

Base on previous experiment, the solid would clog at the bottom discharge outlet if continue using the same tank. Alternatively, for cellulose extraction process, the holocellulose was blend manually in other tank starting from the washing process holocellulose until neutral, almost *pH 7*, cellulose extraction and filtration process. In the extraction process, there was no issue came out, but planning to use the same tank for new design reactor to reduce product loss as well as when filtration process occurred.

Figure 3.4: Sunlight drying process

The drying process of cellulose was used under sunlight since this time the weather was hot and sunny suitable for rapid drying process as figure 3.4 since this was the preliminary run. The total dry cellulose recovered from *8kg* of Red Meranti Wood sawdust was *3.3kg*. For the future real process, the drying process will be in drying oven using temperature *60°C* for *12hrs* to *24hrs* (until the weight is constant).

B. Percentage Yield and FTIR Analysis

From the preliminary run, *8 kg* of Red Meranti Wood Sawdust (RMWS) was recovered *3.3kg* of cellulose with an actual yield, *41.25%* compared with theoretical yield was *6.85%* more than actual one. From these yields, the percentage yield from the run was *85.76 %*. This result was acceptable because the procedure was not optimized yet and the reasons for this can include in-complete or complete reactions and product loss during recovery. The optimum conditions for the pretreatment will be identified later based on percentage yield aid by Design Expert software with two level factorial technique.

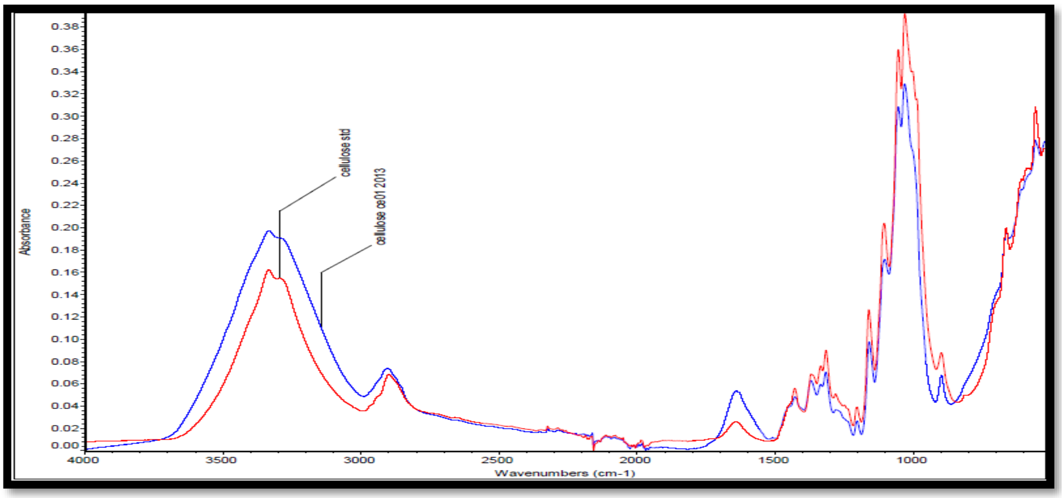

Figure 3.5: FTIR analysis of comparison between Cellulose Standard and Cellulose CE01

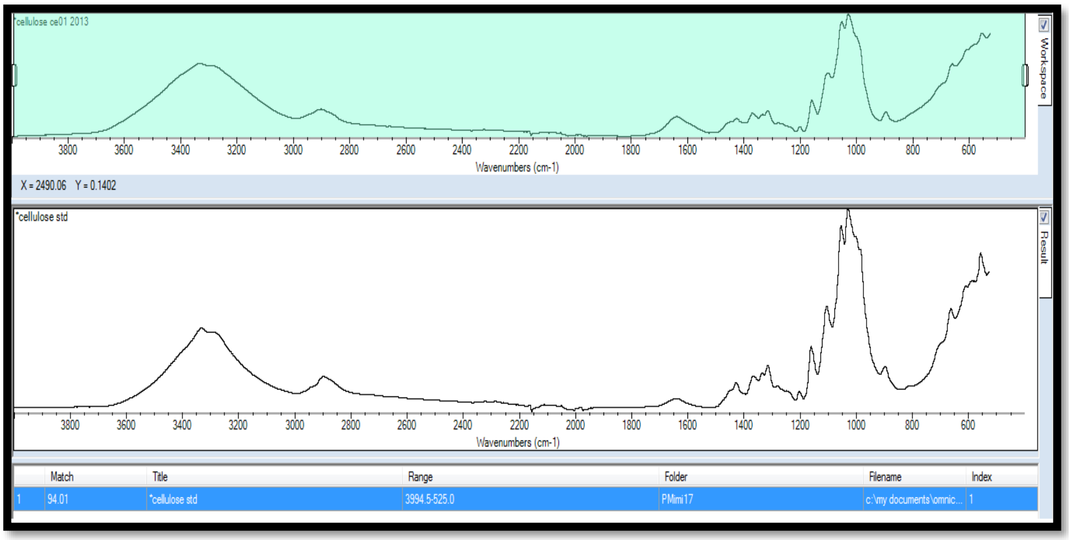

Figure 3.6: Percentage similarity between Cellulose standard and Cellulose CE01

Fourier-transform infrared spectroscopy *(*FTIR*)*device tool was known as an easy and quick to validate the property of carbohydrates and to identify various functional groups present in a sample or product [12]. Beside that, this tool also can analyze the structure and property of physiochemical and polysaccharides for the selection and validation of that material [13]. Hence, the present study aimed to validate the FTIR spectra of the cellulose from RMWS and compare it to the commercial cellulose standard from Sigma-Aldrich[14]. Figure 3.5 shows the spectral pattern of RMWS Cellulose CE01 and commercial cellulose standard after FTIR spectroscopic analysis, which were showed two absorbance regions which were at a high wave number, (range of *1800–600*) and thesecond one was at lower wave numbers (range of *4000–2800*). The summary result showed from FTIR with the percentage similarity as shown in figure 3.6 for cellulose from RMWS, CE01 and commercial cellulose standard from Sigma Aldrich was *94.01%*. This result validates that the selected pre-treatment was recover cellulose from RMWS with similarity *94.01%* and further analysis would proceed after the optimization method was done.

C. Operability Topics, Sources and Design Features

In system engineering, the requirement of quality attributes was very important to mark the difference between whether the research development succeeded or failed. These qualities, often called as non-functional requirements have two main categories including the execution qualities (safety, security and usability during run time) and evolution qualities (scaibility, testability, extensibility and maintainability)[6]. A summary from overall operability issues from observation during preliminary run are presented is presented in Table 3.1.

Table 3.1: Summary of Operability Issues during Preliminary Run and Design Features

Operability Topics	Source of Variability	Design Features
Raw material	Red Meranti Wood Sawdust	From the analysis, confirm that RMWS consist was high yield production and availability and low input cost.
Pretreatment Scheme	Acidified chlorite delignification Alkaline pretreatment	From analysis, theseprocedures were applicable with the new reactor system/
Capacity and space	Volume of tank Space and multiple unit of operations	The process would be design for the optimum volume in 80% of one single reactor tank. The 6 unit operations consist of delignification, extraction as well as filtration process would be in one complete system
Flexibility and Dynamic operation	Lifting system Filtration system Removable baffle, filter nylon and agitator	The design will have the flexibility in term of lifting system for reactor lid attachable with agitator, and filter basket, Filtration system in one system reactor Attachable accessory suitable for current process: baffle and adjustable agitator blade. Pitch blade turbine will be use in blending two or more liquids and are effective in low bottom clearance with less liquid submergence.
Transition	Heating system	The heating system will be design as jacketed reactor instead of internal heater coil. Prolonged operation at acidic medium inside the tank will corrode the coil and lead to poor heat transfer
Efficiency and Profitability	Liquid Solis Ratio (LSR) Mixing and baffles	The LSR too low using the selected pretreatments, the methods were adjusted to maximize the mixing efficiency and need to optimize it later by aid Design Expert Software using two level factorial and CCD techniques. The adjustable and removable agitator's blade and baffles will increase the performance of mixing.
Reliability	Product Discharge	The existing T-joint and valve at the bottom discharge was removed to prevent the clog. The diameter of discharge outlet also too small for solid product even the agitator was on to prevent blockage. The feature design is all the process will process inside the filter basket located inside the reactor and final product in basket will move and transfer to the ground using lifting system.
Safety	Smoke out and wastewater	A reactor system would be design linked directly to air waste treatment system and waste water treatment

4. CONCLUSION

The summary of operability analysis proved that the new system was potentially applicable for cellulose recovery and a proper and efficient operating performance of pilot plant should be chosen with upgraded the system from an existing *100l* stirred tank to a new complete and intensified *100l* reactor tank system with combined filtration and lifting systems as well as to evaluate possible strategies for gas waste treatment.

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