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Production of Dissolving Cellulose from Rice Straw in Vietnam by Pre-hydrolysis Kraft Pulping

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Abstract

Two-stage treatment of rice straw was used for preparation of cellulosic pulp containing high content of alpha cellulose, which can be used for production of dissolving cellulose. First, rice straw was pre-hydrolyzed by sulphuric acid solution under the conditions of liquor-to rice straw ratio of 8:1 (ml/g), temperature of about 100°C in 40-45 min. reaction time. Then, pre-hydrolyzed rice straw was treated by Kraft pulping process using sodium hydroxide solution combined with sodium sulphide, with active alkaline dosage of 6.5% (w/w), at temperature of 95-100°C for 95-100 min. After hydrolysis stage, the yield of reducing sugar of 15.2-15.4% (w/w) over dry starting material was obtained. Reducing sugar hydrolysate was analyzed by HPLC showing that the reducing sugars consist of 96% of arabinose and 4% of glucose, which could be converted to bioethanol or furfural. The yield of cellulosic pulp of 36.3% (w/w) was achieved in Kraft pulping process which contains 78.7% alpha cellulose. Cellulosic pulp that was obtained after Kraft pulping was then treated by ECF bleaching and sodium hydroxide purification. This stage resulted in yield of 26.4% over dry rice straw with whiteness of 88% ISO and alpha-cellulose content of 86.8%. Moreover, the changes of fibre morphology after treatment of rice straw were revealed by the microscopic observations performed by Scanning Electron Microscope (SEM).

Keywords

Rice Straw, Acid Hydrolysis, Kraft Pulping, Dissolving Cellulose, Fibre

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1. Introduction

As known that dissolving cellulose is important product, which can be obtained from wood or non-wood materials. It has special properties including as a high level of brightness, low degree of polymerization and uniform molecular-weight distribution [1]. This material is manufactured for industrial uses that require a high chemical purity and particularly low hemicelluloses content. In contrast to paper-grade pulps, dissolving pulps must contain a high content of cellulose (90–99%), low content of hemicelluloses (2–4%), and traces of residual lignin, extractives and minerals [2, 3]. Hemicelluloses are undesirable impurities in dissolving

pulps, affecting the cellulose process ability such as the filterability and the xanthanation in the viscose process, and properties of the cellulose-end products such as the viscose strength [4, 5]. Most of the hemicellulose is reduced from wood by acid sulphite and pre-hydrolysis Kraft processes, the two major methods used to produce dissolving pulps [4]. The quality of dissolving pulp depends on its purity (cellulose content), and thus on the production process [6].

Dissolving-grade pulps are used as raw material in the manufacture of different cellulose-derived products, including viscose rayon, the first commercially manufactured regenerated cellulose fiber. In the regenerated cellulose process cellulose is treated with carbon disulfide (highly

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toxic solvent) in the presence of a base to produce cellulose xanthate which dissolves easily in caustic soda [3, 7]. The resulting viscous liquid can be extruded through spinnerettes and regenerated as man-made fibres. Cellulose can also be dissolved in some organic solvents directly and processed to regenerate the cellulose fibres in different forms. The lyocell process uses an amine oxide to dissolve cellulose and Tencel is the only commercial example of this direct-dissolution process, which unlike the viscose process is pollution-free [3]. The 90-92% cellulose content sulfite pulps are used mostly to make textiles (like rayon) and cellophane. The 96% cellulose content sulfate pulps are used to make rayon yarn for industrial products such as tire cord, rayon staple for high-quality fabrics, and various acetate and other specialty products [8]. Moreover, dissolving-grade pulps are used as raw material for production of cellulose esters, cellulose ethers, other derivatives such as the base material of heat hardened resins, and specialty paper [6, 9, 10].

Currently, the raw fiber materials used for dissolving pulps are mainly woods and cotton linters in industry. However, non-wood plants such as agriculture residues, bagasse, bamboo, etc. are a copious and cheap source for cellulose fibers recently have gained many interests from scientists to use as raw material for production of composite, textile, pulp and paper [11, 12]. Their biofibers have the composition, properties and morphological structure similar to woods. Furthermore, non-wood plants offer several advantages such as requiring moderate irrigation and fertilization during short growth cycle of plant and low lignin content resulting in reduced energy and chemicals consumption during pulping. Besides, using non-wood as feedstock could save wood raw materials for other uses and prevent massive deforestation, reduce cellulose fiber export [6, 13–15].

Rice straw is main agricultural product in Vietnam as well as South-East Asia countries. Annually, there is huge amount of rice straw eliminated after harvesting in Vietnam. Rice straw is mostly burnt directly on the field generating lot of smoke and dust. The combustion or elimination of rice straw will affect seriously to the environment and public health. Thus finding a suitable way to utilize this rice straw biomass is effective solution as it brings us great benefit in economics and environment.

Therefore, in this study rice straw in Vietnam was subjected to exploit as raw material for achievement of value-added product, dissolving cellulose for several further applications. The objective of this work was to investigate the possible pulping of rice straw as an alternative, non-wood pulping raw material. In particular, the two-stage process including of pre-hydrolysis and Kraft pulping (sulphate cooking) was used for preparation of cellulosic pulp containing high content of alpha cellulose, which can be used for production

of dissolving cellulose. The influence of pre-hydrolysis and Kraft pulping variables (process parameters) on the properties of the resulting dissolving pulp such as yield, alpha-cellulose, kappa number was also studied.

2. Materials and Methods

2.1. Materials

Oryza sativa L. rice straw which was harvested in November, 2013 from the North of Vietnam was used for experiments. The over dry rice straw was grinded and screened. The fraction that was retained on 24 mesh screen was used for study. Prepared straw was stored in plastic bags at room temperature and was not washed before being subjected to acid hydrolysis and Kraft pulping.

2.2. Acid Pre-hydrolysis of Rice Straw

Rice straw pre-treatments were carried out at temperature of 70-120°C, in 20-60 minutes with solid/liquid ratio of 1:10 in 1000 ml rotary stainless steel digesters, which heated in glycerol tank. Amount of sulphuric acid is about 0.1-1.0% (weight percent) over dry rice straw. Residual solid sample was then separated from the pre-treatment liquid by filtration through pre-dried and pre-weighed glass fiber to get the solid residue and the liquid hydrolysate. Residual solid (pre-hydrolyzed rice straw) was then used for Kraft pulping.

2.3. Kraft Pulping

Pre-hydrolyzed rice straw was cooked at temperature of 80-120°C, in 80-120 minutes with solid/liquid ratio of 1:10 in 1000 ml rotary stainless steel digesters, which heated in glycerol tank. Amount of active alkali (amount of NaOH or NaOH + Na₂S) is about 10-30% weight percent over dry rice straw, and sulfur degree (Na₂S/NaOH+Na₂S ratio) is 25% weight percent [16]. Residual solid sample (pulp) was then separated from the liquid by filtration through pre-dried and pre-weighed glass fiber. Pulp was neutralized by acetic acid, washed by DI water several time and collected for determination of moisture content, yield and other characterization.

2.4. Bleaching

Bleaching of the pre-hydrolysis/Kraft pulp was performed following D_0 – (EOP) – D_1 bleaching sequences. The detail of bleaching process sequence was as follows: (1) first chlorine dioxide treatment, D_0 . The pulp with consistency of 10% was treated for 60 min at 60°C with chlorine dioxide solution. Total chlorine charge for bleaching was 2.4 kg chlorine/ton of pulp and was distributed as 30% in D_0 and 70% in D_1 . The resulting product was filtered and washed with water to remove all chlorine ions; (2) sodium hydroxide

extraction, E. The previously obtained product was suspended in an aqueous solution (10% consistency) containing NaOH (2.5 kg/ton of pulp), agitated for 90min, at 60°C, filtered, and washed with water until obtaining neutral conditions; (3) oxygen treatment, O. The pulp product (10% consistency) was agitated for 60min, at 60°C in closed digester with oxygen gas (3 kg/ton of pulp), filtered, and then washed with water; (4) hydrogen peroxide treatment, P. The product of the previous step at 10% consistency was treated for 60min, at 60°C and a pH of 9-11 (adjusted by sodium hydroxide), with H₂O₂ (2 kg/ton of pulp), filtered, and then washed with water neutralization; (5) second chlorine dioxide treatment, D₁. Finally, pulp with consistency of 10% was treated with chlorine dioxide solution for 60min, at 60°C. The mixture was filtered, washed with water until neutralization, and air-dried [3, 6, 16].

2.5. Analysis

For determination of chemical composition, rice straw was prepared by TAPPI method T264 cm-97. The chemical composition of straw was determined as follow: cellulose content 34.7% (TAPPI T17 wd-70); lignin content 16.3% (TAPPI T222 om-98); pentosane 19.8% (TAPPI T223); Ash 11.2% (TAPPI T211 om-93).

Liquid hydrolysate was characterized by spectrophotometer (Optima-sp 300) to determine the yield of reducing sugar following the methods that reported by Miller (Dinitro salicylic acid (DNS) method) [17] and high-pressure liquid chromatography system (Prominence HPLC standard automated system; Shimadzu Scientific Instruments, Columbia, MD) equipped with an auto-sampler (SIL-20A/C).

Cellulose content of rice straw was determined by Kushner – Hoff method. Alpha-cellulose content of pulp was determined by TAPPI method T203 cm 09. Scanning electron microscope (SEM) images of fiber of Kraft pulp and prehydrolyzed/Kraft pulp were taken in a Jeol 5410 LV machine.

3. Results and Discussion

3.1. Dilute Acid Pre-hydrolysis and Hydolysate Composition

As known that pre-hydrolysis Kraft pulping is widely applied for producing of cellulose from wood (mainly softwood), which is used as raw material for conversion to dissolving cellulose in production of cellulose-based chemicals and materials [6]. This process technology consist two stages: pre-hydrolysis with sulfuric acid at temperature of $130 \div 140^{\circ}$ C followed by sulfate cooking (Kraft process) at temperature of $160 \div 170^{\circ}$ C. Then, the as-obtained cellulose

pulp is washed, cleaned, bleached and further purified (removal of hemicellulose) to achieve dissolving cellulose. Hemicelluloses with amorphous structure and relatively low degree of polymerization are easy to hydrolysis. Hence, prehydrolysis was used prior to any alkaline pulping process helps to produce pulp with a satisfactorily high content of cellulose and with a low hemicellulose content, resulting from the destruction or degradation of hemicelluloses with alkali soluble substances. Pre-hydrolysis could also increase the extractability of lignin during the subsequent alkaline pulping process, thus substantially reduce the lignin and ash contents and open up the cellulose fibers for further reactions [6, 18]. By introduction of pre-hydrolysis prior to pulping process could help to conduct the sulfate pulping with milder condition (lower active alkaline content, lower temperature and shorter digestion time) resulting in low degree of degradation of cellulose thus increase the yield of pulp and improve the physical-mechanic properties of cellulosic pulp. Moreover, pentose was achieved as by product after prehydrolysis and could be used for production of value-added products such as bioethanol, Torula yeast, furfural, etc.

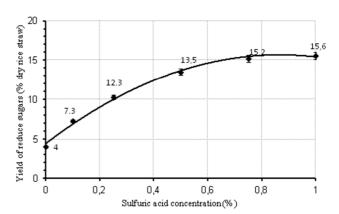


Figure 1. The effect of sulfuric acid concentration on yield of reducing sugars (Time of treatment: 50 min.; Treatment temperature: 100°C).

Preliminary research showed that water hydrolysis of rice straw only happened drastically at high temperature (180-200°C). At low temperature, the process occurred very slowly with low degree of deconstruction and degradation of hemicelluloses (up to 7-9%). Therefore, dilute acid hydrolysis of rice straw was chosen to improve the hydrolysis stage. The effect of technical parameters such as sulfuric acid concentration, temperature and treatment time on reducing sugar (mainly pentose) were investigated to establish the suitable technical regime for acid hydrolysis of rice straw. Figure 1 showed the effect of sulfuric acid concentration on yield of reducing sugars. It could be seen that increase of H₂SO₄ concentration would increased the yield of reducing sugars. When H₂SO₄ concentration increased from 0.1% to 0.8%, sugar yield increased from 5% to 15.2% (w/w percent over dry rice straw), and reach to

maximum at acid concentration of 0.8%. When Acid concentration was further increased, the yield of reducing sugars increased insignificantly. Thus, the suitable acid concentration for hydrolysis of rice straw was chosen to be 0.8%.

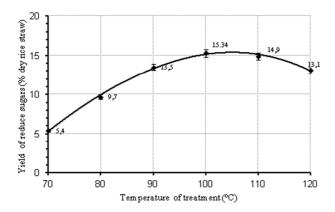


Figure 2. The effect of treatment temperature on yield of reduce sugars (Sulfuric acid concentration: 0.8%; Time of treatment: 50 min).

Similarly, the effect of pre-hydrolysis temperature was also investigated in the range of $70 \div 120^{\circ}\text{C}$ and the results were shown in Fig. 2. It's found that at temperature lower than 100°C the yield of reducing sugars increased with the increase of temperature and reached to maximum of 15.34% (w/w over dry rice straw) at temperature of 100°C . At temperature greater than 100°C yield of reducing sugars decreased with the increase of temperature. It could be assumed that the decrease of reducing sugars after prehydrolysis due to the degradation of sugars at high temperature. Hence temperature of 100°C was chosen as suitable temperature for pre-hydrolysis of rice straw.

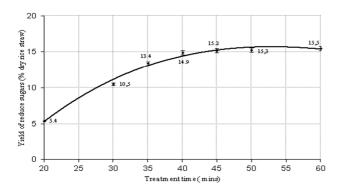


Figure 3. The effect of treatment time on yield of reduce sugars (Sulfuric acid concentration: 0.8%; Treatment temperature: 100°C).

The hydrolysis time also affected certainly on the conversion of rice straw. The investigation of effect of hydrolysis time was conducted in range of 20-60 min and at suitable acid concentration and temperature. As seen in Figure 3, when treatment time increased from 20 to 50 min the yield of reducing sugars increased. However, amount of sugar did not

increase and even slightly decreased when treatment time further increased to 60 min. Therefore, 50 min could be considered as suitable reaction time for pre-hydrolysis of rice straw.

The suitable conditions for pre-hydrolysis process were established to be acid concentration of 0.80%, reaction time of 50 min at temperature of 100oC. Under these suitable conditions, yield of reducing sugars of 15.4% (w/w over dry rice straw) and yield of pre-hydrolysis rice straw of 72% could be achieved. The hydrolysate after pre-hydrolysis was also analysed by HPLC to determine the composition of sugars (as shown in Figure 4). It's seen that reducing sugar solution included mainly arabinose and a little of glucose. Thus, with above mentioned appropriate technological regime, mainly pentosane was hydrolyzed, while a small part of cellulose was degraded. From one ton of dry rice straw can be obtained 160 kg of arabinose and 6.6 kg of glucose. Equivalence, about 70% of pentosane (in comparable with the content in the original rice straw) was hydrolyzed and about 17% of cellulose hydrolyzed. This amount of cellulose is amorphous fraction, which had low polymerization degree and needed to remove from pulp for preparation of dissolved cellulose. It also can be seen that over 30% of the pentosane of rice straw is hard to hydrolyze.

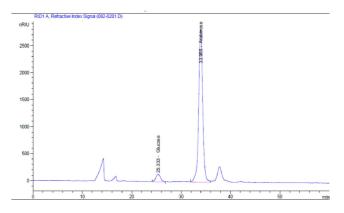


Figure 4. HPLC of hydrolysate after hydrolysis of rice straw.

3.2. Kraft Pulping of Pre-hydrolyzed Rice Straw and Cellulose Characterization

Kraft pulping is an alkaline process that uses a solution of NaOH and Na₂S as reactant solution. Alkaline hydrolysis reactions reduce the molecular weight of the lignin structures and remove the methoxyl groups (–O–CH₃) that lead to the formation of phenolate ions. Sodium sulfide accelerates the cooking reactions and decreases cellulose degradation caused by sodium hydroxide. The hydro-sulfide ion (SH⁻) participates in the "blocking" reaction and inhibits lignin condensation reactions, which impairs the removal of lignin. Basically, it functions as a pulping catalyst and thus speeds up delignification [6]. Even thought the Kraft pulping

process of wood in pulp and paper industry is well-known and the optimal conditions have been established, but the optimal conditions for pulping of rice straw is quite different. Because the optimal conditions for Kraft pulping of different types of raw materials are different, especially for wood and non-wood. Therefore, we've conducted the experiments to find out the suitable condition for Kraft pulping of rice straw by changing the process parameter in the certain range. After pulping, the efficiency of the process was determined based on the yield of cellulosic pulp and alpha cellulose content.

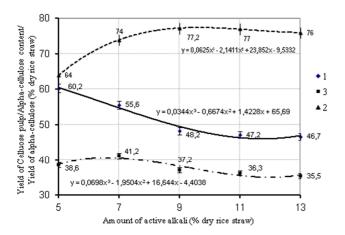


Figure 5. The effect of amount of active alkali on yield and properties of cellulose pulp. 1. Yield of cellulose pulp, 2. Alpha-cellulose content of pulp, 3. Yield of alpha-cellulose.

Figure 5 showed the effect of amount of active alkali on yield and properties of cellulose pulp. It could be seen that by keeping the solid/liquid ratio of 1:8, temperature of 120°C, pulping time of 120 min and sulfidity of 25% the yield of cellulosic pulp decreased with the increase of active alkali charge in range of 5-13% (w/w percent over pre-hydrolyzed dry rice straw). The yield of cellulosic pulp decreased significantly when the amount of active alkali increased from 5% to 9%, and decreased insignificantly when the amount of active alkali further increased to 13% (trend 1 in Fig. 5). Moreover, under the active alkali charge in the range of 5-9%, the degradation of alpha cellulose occurred trivially but the degradation of other components such as lignin, extractives and inorganic occurred drastically led to the slight decrease of yield of alpha cellulose (about 1%, trend 3 in Fig. 5) and noticeable increase of alpha cellulose content in pulp (from 64% to 77.2%, trend 2 in Fig. 5). When the active alkali charge higher than 9%, the degradation of cellulose happened drastically led to the decrease of yield of cellulosic pulp, yield of alpha cellulose as well as alpha cellulose content in pulp. Thus the active alkali charge of 9% was considered as suitable for Kraft pulping of pre-hydrolyzed rice straw for production of dissolving pulp.

The effect of temperature on yield and properties of

cellulosic pulp had similar trend with active alkali charge. When temperature increased from 80oC to 100oC, the yield of pulp decreased drastically. However, when temperature increased further from 100°C to 120°C, the yield of pulp decreased slightly (Figure 6). In contrast, the alpha cellulose content in pulp increased with the increase of temperature. The alpha cellulose content increased significantly by varying the temperature in the range of 80-100°C. The increase of temperature also induced the degradation a part of α -cellulose, about 1-2%. Therefore, by consideration of economic and technical efficiency the suitable temperature for Kraft pulping of pre-hydrolyzed rice straw for production of dissolving pulp was chosen to be 100°C.

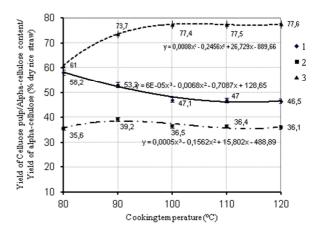


Figure 6. The effect of cooking temperature on yield and properties of cellulose pulp. 1. Yield of cellulose pulp, 2. Yield of alpha-cellulose 3. Alpha-cellulose content of pulp.

The effect of cooking time on yield and properties of cellulosic pulp was shown in Figure 7. It's seen that when pulping conducted with longer cooking time resulted in lower yield of pulp and lower yield of alpha cellulose. To achieve high α -cellulose content with relative high yield of pulp the cooking time should be controlled about 100 min.

The suitable conditions for Kraft pulping process were established to be solid/liquid ratio of 1:8, temperature of 100°C, cooking time of 100 min, sulfidity of 25% and active alkali charge of 9% over dry pre-hydrolyzed rice straw. Table 1 summarizes the results of acid pre-hydrolysis/Kraft pulping (under suitable conditions) and Kraft pulping (without prehydrolysis) of rice straw. The results from Table 1 showed that the pre-hydrolysis stage had a positive effect on the purity of the dissolving pulp, resulting, in particular, in an increase of the alpha-cellulose content of about 27% as well as in an increase of yield of alpha-cellulose of 11%. Moreover, the rice straw consists of 34.7% cellulose, and during the pre-hydrolysis approximately 17% of cellulose was degraded, so there was an amount of hard-hydrolyzed pentosane remained in the composition of alpha-cellulose. This kind of pentosane had high polymerization degree and

relatively as stable as cellulose in sulphate cooking.

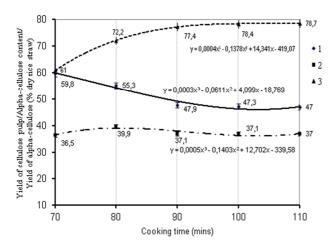


Figure 7. The effect of cooking time on yield and properties of cellulose pulp. 1. Yield of cellulose pulp, 2. Yield of alpha-cellulose, 3. Alpha-cellulose content of pulp).

The positive effects of this stage were accompanied by a lower yield of pulp that could be attributed to the removal of the remaining hemicelluloses and probably, to a lesser extent, to lignin [6]. These results indicated that the pre-hydrolysis/Kraft pulping process was well suited for the pulping of rice straw to obtain dissolving pulp. Moreover, these results also implied that the chosen process parameters were suitable for pre-hydrolysis/Kraft pulping of *Oryza sativa L*. rice straw to produce dissolving pulp.

Table 1. Result of pre-hydrolysis/Kraft pulping and Kraft pulping (without pre-hydrolysis) of rice straw.

Type of rice straw	Yield of pulp, (% dry rice straw)		Yield of α-cellulose, (% dry rice straw)
Non-hydrolized rice straw	49.7	61.6	30.6
Prehydrolized rice straw	43.1	78.7	33.9

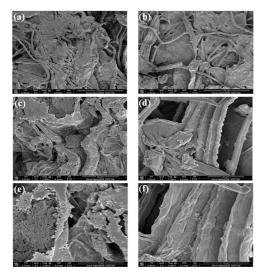


Figure 8. SEM of original rice straw Kraft pulp (a, c, e) and pre-hydrolyzed Kraft pulp (b, d, f) at different magnification.

The samples of Kraft pulp (obtained from Kraft pulping of rice straw without pre-hydrolysis stage) and pre-hydrolyzed Kraft pulp were characterized by SEM with different magnification and shown in Figure 8. It's found that Kraft pulp had a lot of broken fiber and contained the fibrils that were shorter and more aggregate than fibrils in pre-hydrolyzed Kraft pulp. It could be attributed to the lower alpha-cellulose content in the Kraft pulp that contributed to the longer fiber of pulp and less degradation of lignin and extractives in the Kraft pulp that could make the fibril aggregated together [19].

3.3. Bleaching of Pre-hydrolyzed Kraft Pulp

The principal aims of the bleaching treatment are to selectively oxidize lignin and to minimize the oxidation of cellulose [6]. Bleaching could reduce the kappa number without drastically decreasing the degree of polymerization of the pulp. The elemental chlorine free (ECF) and the total chlorine free (TCF) bleaching sequences are based on oxygen-derived compounds, among which hydrogen peroxide has received much attention due to environmental friendly and cheap properties [6, 20]. Peroxide bleaching may either be carried out as a single-stage process, or may be preceded by a pre-treatment to eliminate peroxide decomposition catalysts and/or followed by conventional bleaching stages.

The pre-hydrolyzed Kraft pulp (with Kappa number of 12-13 units) was bleached by D₀ (EOP) D₁ bleaching process (ECF process) with chlorine dioxide dosage 2.4 kg/dry tons of pulp followed with cellulose purification by 6% sodium hydroxide solution at 70°C in 70 min. As-obtained cellulosic pulp after bleaching and purification had yield of 26.4% over dry rice straw with whiteness of 88% ISO and alpha-cellulose content of 86.8%, which could be used for production of cellulose derivatives. Compared to the dissolving pulp obtained from other wood and non-wood materials, the alpha-cellulose content of pulp from Vietnamese rice straw is relatively high, but it has higher ash content [6]. This indicates that rice straw has the potential to be used as raw materials for the production of dissolving pulp as well as cellulose derivatives.

Vietnam is the World's second largest rice export country with rice export of approximately 7 million tons per year. Vietnam produces about 45 million tons of rice and about 50 million tons of rice straw biomass, annually. The most popular methods for exploitation of rice straw are using them as the combustible in life or making the compost. The rest is eliminated to the surrounding environment as waste. Therefore, rice straw could be collected and transported to mill at a very low price. Therefore the cost of production reduced noticeably considerably due to cheap feedstock. It is evident that new sources of pulp raw materials would provide

an economic incentive to the agricultural and industrial sectors and an environmental solution.

4. Conclusion

Pre-hydrolysis/Kraft pulping process was suitable for the pulping of rice straw to obtain dissolving pulp. Two-stage process consists of sulfuric acid pre-hydrolysis and Kraft pulping could be applied. At sulfuric acid pre-hydrolysis stage, suitable conditions were found to be solid/liquid ratio of 1:8, acid concentration of 0.80% and reaction time of 50 min at temperature of 100°C. At Kraft pulping stage, suitable conditions were found to be solid/liquid ratio of 1:8, temperature of 100°C, cooking time of 100 min, sulfidity of 25% and active alkali charge of 9% over dry pre-hydrolyzed rice straw. Sugar liquid that was obtained after sulphuric acid pre-hydrolysis had reducing sugar concentration of 26 g/l. This sugar liquid could be used as feedstock to produce other value products. Cellulosic pulp that was obtained after Kraft pulping and ECF bleaching and sodium hydroxide purification had yield of 26.4% over dry rice straw with whiteness of 88% ISO and alpha-cellulose content of 86.8%.

The properties of the dissolving pulp obtained from *Oryza sativa L*. rice straw were similar to those of Kraft pulp from other sources. This result implied that the rice straw have a good potential to produce dissolving pulp with high alphacellulose content. This is preliminary research for production of cellulose derivatives from agricultural residue at commercial scales.

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