

## CELLULOSE EXTRACTION FROM PALM KERNEL CAKE USING LIQUID PHASE OXIDATION

FARM YAN YAN, DUDUKU KRISHNIAH, MARIANI RAJIN,  
AWANG BONO\*

Chemical Engineering Program, University Malaysia Sabah,  
88999 Kota Kinabalu, Sabah, Malaysia.

\*Corresponding Author: awang@pc.jaring.my

### Abstract

Cellulose is widely used in many aspect and industries such as food industry, pharmaceutical, paint, polymers, and many more. Due to the increasing demand in the market, studies and work to produce cellulose are still rapidly developing. In this work, liquid phase oxidation was used to extract cellulose from palm kernel cake to separate hemicellulose, cellulose and lignin. The method is basically a two-step process. Palm kernel cake was pretreated in hot water at 180°C and followed by liquid oxidation process with 30% H<sub>2</sub>O<sub>2</sub> at 60°C at atmospheric pressure. The process parameters are hot water treatment time, ratio of palm kernel cake to H<sub>2</sub>O<sub>2</sub>, liquid oxidation reaction temperature and time. Analysis of the process parameters on production cellulose from palm kernel cake was performed by using Response Surface Methodology. The recovered cellulose was further characterized by Fourier Transform Infrared (FTIR). Through the hot water treatment, hemicellulose in the palm kernel cake was successfully recovered as saccharides and thus leaving lignin and cellulose. Lignin was converted to water soluble compounds in liquid oxidation step which contains small molecular weight fatty acid as HCOOH and CH<sub>3</sub>COOH and almost pure cellulose was recovered.

Keywords: Hemicellulose, Lignin, Hot water treatment, Response surface methodology

### 1. Introduction

Cellulose is the most abundant and enormous renewable polymers in the world. Cellulose is produced at an annual rate of 10<sup>11</sup>-10<sup>12</sup> tons by nature [1]. Its extending properties which are generally strong hypophilic, stable to chemicals,

### Nomenclatures

<i>A</i>	Hot water treatment temperature, °C
<i>B</i>	H <sub>2</sub> O <sub>2</sub> : sample, g/g
<i>C</i>	reaction temperature of H <sub>2</sub> O <sub>2</sub> oxidation, °C
<i>D</i>	reaction time of H <sub>2</sub> O <sub>2</sub> oxidation, hr

safe to living bodies, reproducible, recyclable, and biodegradable [2] contributes to many aspect and industries such as paper industry, food industry, pharmaceutical, paint and many more. Hardwood and softwood, which are the major sources of cellulose, have received attention due to forest preservation and rational use of forest and agriculture residues. Recently, high cellulose content of agriculture by-products as alternative renewable resources for cellulose production are receiving much attention. Cellulose fractions were extracted with alkali from sugar beet pulp [3], isolation of cellulose from wheat straw [4,5], cellulose preparation from perennial ryegrass leaves (*Lolium perenne*) [6], cellulose from oil palm empty fruit bunches via ethanol digestion [7], cellulose extraction from orange peel [8] as well as many others alternative sources that are still discovering for cellulose production.

Palm kernel cake (PKC) is one of the abundant by-products from oil palm industries in Malaysia. It is produced annually in large quantities, about 1.9 millions tons of PKC is left over by oil extraction process from oil palm industry in 2003/2004 in Malaysia [9]. These amounts are significant enough to consider palm kernel cake as bioresource of raw materials for many industries, utilizing the predominant constituents of palm kernel cake that are cellulose, hemicellulose and lignin. PKC consists roughly 30% of cellulose [10]. At present time, PKC is used as dairy, swine, aquaculture and ruminants feed [11-13]. Hence, PKC as alternative sources for cellulose production not only enhances the usage of PKC. It is also able to solve the environment problem.

Many biomass process methods have been developed and yet much of the current studies and researches still devoted to cellulose production. Conventional processes for separation of cellulose are alkaline kraft process and sulphite process which most commonly used in papermaking [14]. Alkaline (kraft) pulping of wood is performed in a solution of sodium sulfide and sodium hydroxide in large pressure vessels called digesters. Sulphite process is a process with acid bisulfite solution to soften the wood material by removing the lignin from the cellulose. However, large amount of concentrated toxic waste water from those conventional processes have come under criticism due to negative influence on the environment. The various environmental friendly approaches are elaborated at present in order to prevent ecological damage. Enzyme from Filamentous fungi to recover cellulose from biomass is another green method for cellulose production. The cultivated fungi which obtain nourishment by secreting enzymes are used to degrade lignin. The production of cellulose and hemicellulose-degrading enzymes by cultivation of *Aspergillus niger* ATCC 9029, *Botrytis cinerea* ATCC 28466, *Penicillium brasilianum* IBT 20888, *Schizophyllum commune* ATCC 38548, and *Trichoderma reesei* Rut-C30 was studied by Anders et al [15]. A commercial preparation of *Trichoderma reesei* cellulases with wet oxidation as pretreatment was used for enzymatic study of cellulose production was carried by Carlos et al,

[16]. Wet oxidation, steam explosion, steam cooking, and pressure cooking in water, hydrothermal treatment, and liquid hot water treatment [17] are processes which wet cooking at elevated temperature and pressure in order to render the cellulosic component of the biomass more accessible to enzymatic attack. However, selectivities of final products are generally not so high. The yields and characteristics of cellulose are strongly dependent on the severity of treatment condition [18]. Organosolv is one of the green process method that been commercialized to recover cellulose from biomass using organic solvent. Ethanol [7, 19], acetic acid [20], or aqueous methanol [21] are the common organic solvent for organosolv process. The organic solvent can be recycled in the process. However, one of the main drawbacks of ethanol and methanol organosolv systems is high energy consumed, high pressure is required during the pulping stage and autocatalyzed processes only provide pulp with acceptable quality and high yield from hardwoods [22].

H<sub>2</sub>O<sub>2</sub> has been reported as environmental friendly oxidation reagent [23-25]. It will allow many environmental problems to be solved. In this work, Liquid phase oxidation was used to recover cellulose from PKC. Hemicellulose was removed from palm kernel cake before liquid oxidation was applied. The aims of the present study are to extract cellulose from palm kernel cake in order to separate hemicellulose and lignin and to discover the characteristic of cellulose. The chemical composition and physicochemical properties were analyzed and studied.

## **2. Experimental**

### **2.1. Materials and methods**

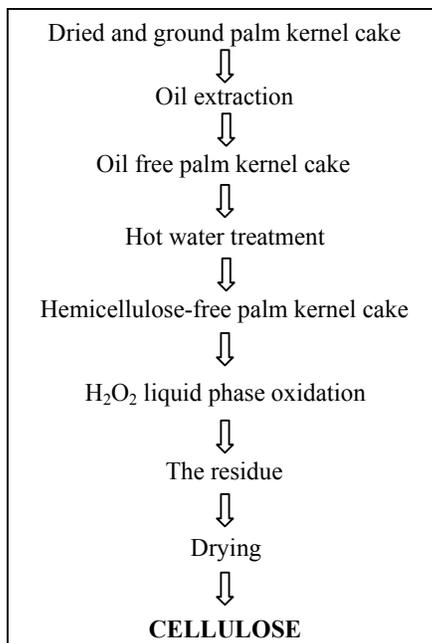
Palm kernel (PK) used in this experiment was purchased from Borneo Samudera Lumadan mill, Beaufort, Sabah. The PK was washed, and then dried in an oven at constant temperature. Oil from PK was removed by using conventional Soxhlet extractor with 100% hexane for 6 hours. The extractive-free sample (PKC) was dried in an oven at 60°C for 18 hours and stored in refrigerator before use.

### **2.2. Isolation of cellulose**

Extraction of cellulose from the PKC was involving a two-step modified liquid phase oxidation method of Kazuhiro Mae et al [26]. Hot water treatment is pretreatment step of cellulose extraction. Hot water treatment was performed in a micro-pressure reactor, which filled with 6 g of grinded PKC and 30 ml of distilled water. The reaction was heated to a temperature of 160-180°C, the reaction pressure increased rapidly up to saturated vapor pressure at the temperature. After 0.5 hour of hot water treatment, the reactor was soaked into cooling water in order to terminate the reaction. The pretreated PKC was washed with distilled water and ethanol to remove the organic acid and saccharides, then dried and sieved to 600 µm.

Liquid phase oxidation was then performed to remove the lignin from PKC and remain the pure cellulose. 2 g of pretreated PKC was mixed with 30% H<sub>2</sub>O<sub>2</sub> by mixing ratio of 1-5, 1-7.5, and 1-10 of weight basis in a 100 ml flask with a tight plug. The flask with the reaction mixture was kept for 10-24 hours at

temperature of 60-80°C as shown in Table 2. After the elapse of reaction time, an excess of cold water was added to the mixture to terminate the oxidation reaction [26]. The mixture was then filtered in order to separate the aqueous solution of organic compounds and solid residue. The scheme of extraction stages for cellulose from palm kernel cake is shown in Fig. 1.



**Fig. 1. Scheme for Extraction of Cellulose from Palm Kernel Cake.**

### 2.3. Analyses of products and yield measurement

FTIR spectra of extracted cellulose were obtained on an FTIR spectrophotometer (Nicolet 750). Pellets were made from extracted cellulose powder ground with KBr and transmission of the sample was measured at the wave number range of 4000-400  $\text{cm}^{-1}$ . The yield percentage of cellulose production was calculated based on the dry basis of PKC.

The recovered solid residue was washed by distilled water and 95% ethanol and dried in oven at 70°C before measuring its weight. The yield of recovered cellulose was calculated based on the mass of extractive-free PKC. The chemical analyses of all samples were performed in duplicate and the experimental error was within 5% for all the experiments.

### 2.4. Experimental design

Three levels Box-Behnken design of Response Surface Methodology (RSM) was used to estimate the interaction of response functions and the process variables in order to optimize the process reaction of cellulose recovery. The process reaction of cellulose recovery from PKC was optimized with respect to yield production

by varying each of the selected reaction parameters. The process parameters are hot water treatment time, ratio of palm kernel cake to  $H_2O_2$ , liquid oxidation reaction temperature and reaction time. Table 1 shows the range of the parameters that used in the experimental.

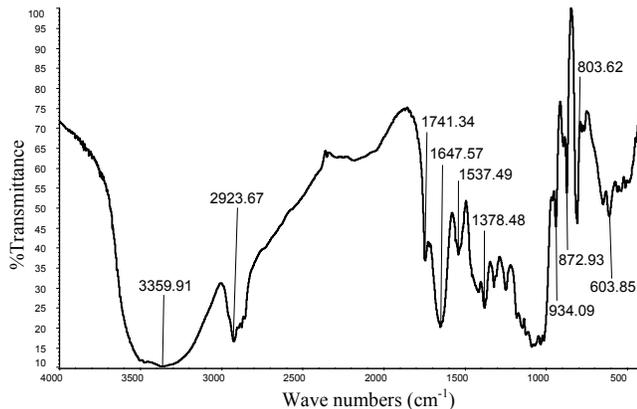
**Table 1. Range of Parameters of Liquid Phase Oxidation Process for Experimental Design.**

Factors	Unit	Level	
		Lower limit (-1)	Higher limit (1)
Hot water treatment temperature	°C	160	180
$H_2O_2$ : sample	w/w	5	10
Oxidation Reaction temperature	°C	40	80
Oxidation Reaction time	hr	10	24

### 3. Results and Discussion

#### 3.1. FTIR Spectrum of cellulose from palm kernel

Figure 2 shows the IR spectrum of extracted cellulose using liquid phase oxidation method by FTIR. The IR spectrum is similar with standard library of cellulose [27]. The IR spectra show the typical adsorption of cellulose backbone at  $1600\text{ cm}^{-1}$ . From the hydrogen-bonded OH stretching at  $4000\text{-}2995\text{ cm}^{-1}$ , is due to the H bonded OH groups and the stretching frequency of the  $-\text{OH}$  group as well as intramolecular and intermolecular hydrogen bonds [14] and a C-H band (c.a  $2923\text{ cm}^{-1}$ ) is due to C-H stretching vibration. The OH bending of adsorbed water at  $1647$ , the CH deformation vibration at  $1375\text{ cm}^{-1}$ , the C-OH out-of-plane bending mode at  $678\text{ cm}^{-1}$ . The band at  $1060\text{ cm}^{-1}$  is due to  $\text{OCH-O-CH}_2$  stretching [27].



**Fig. 2. FTIR Spectra of Cellulose Recovered by Liquid Phase Oxidation.**

#### 3.2. Influence of extraction process parameter on yield recovery

There are 29 sets of experiment from the compilation of RSM for the study in order to determine the optimum reaction of the cellulose recovery as shown in Table 2. By comparing the data in Table 2, it show that a trend yield of cellulose is varies with changes of process parameters. The colour and the yield of the products was

change with the experimental parameters. Hence, further analysis was conducted to study the influence of extraction process parameters on yield recovery.

A statistical analysis was performed on the experimental results. The experimental results of cellulose recovery were applied to obtain the regression models. Quality of the models was evaluated by ANOVA, in which the repetition supplied the freedom degree to obtain the pure error. The quadratic model for yield of recovered cellulose from palm kernel cake using liquid phase oxidation is shown in equation below:

$$\begin{aligned} \text{Yield of Cellulose} = & -4.96 + 0.07A - 0.18B + 0.03C - 0.11D - 0.0002 \times 10^{-4} A^2 \\ & - 0.008B^2 - 0.0001C^2 + 0.0003D^2 + 0.0017AB - 0.00015AC \\ & + 0.00056AD - 0.00056BC + 0.0013BD - 0.0004CD \end{aligned}$$

**Table 2. Design Layout for Liquid Phase Oxidation Cellulose Recovery Process Reaction.**

Exp	Factors				Response
	A: Hot water treatment temperature	B: H <sub>2</sub> O <sub>2</sub> : sample	C: Reaction temperature	D: Reaction time	Yield of cellulose
	(°C)	(g/g)	(°C)	(hr)	(g)
1	170	10	80	17	0.044
2	160	7.5	60	24	0.248
3	170	10	60	10	0.523
4	170	7.5	60	17	0.573
5	160	7.5	40	17	0.817
6	170	7.5	60	17	0.515
7	170	5	60	10	0.707
8	160	5	60	17	0.433
9	180	10	60	17	0.625
10	170	7.5	40	24	0.836
11	170	7.5	60	17	0.485
12	170	7.5	40	10	0.854
13	180	7.5	60	24	0.548
14	180	7.5	60	10	0.775
15	170	5	40	17	0.846
16	160	7.5	80	17	0.098
17	170	7.5	60	17	0.593
18	170	7.5	80	10	0.347
19	160	7.5	60	10	0.633
20	170	5	60	24	0.421
21	180	7.5	80	17	0.055
22	170	5	80	17	0.207
23	180	5	60	17	0.565
24	180	7.5	40	17	0.891
25	170	10	60	24	0.328
26	170	10	40	17	0.796
27	160	10	60	17	0.322
28	170	7.5	60	17	0.573
29	170	7.5	80	24	0.104

The influence and trend of each parameters of the process were studied and shown in detail in further section.

### 3.2.1. Effect of hot water treatment temperature and ratio $H_2O_2$ : Sample on the yield of cellulose recovery

Treatment with the pressurized hot water below  $250^\circ\text{C}$  is known as one of the effective methods to remove hemicellulose from biomass [26]. Therefore, hot water treatment of palm kernel cake in range of  $160\text{--}180^\circ\text{C}$  was conducted in the experiment. The pH of remain water from hot water treatment is shifted to lower pH. The removed hemicellulose from palm kernel cake will be converted to organic compound and dissolve in liquid solution.

Figure 3 shows that the effect of hot water treatment temperature when reaction temperature is  $40.35^\circ\text{C}$  and reaction time at 10 hours as centre point. The yield of recovered cellulose increased when hot water treatment temperature increase. As increase the ratio of  $H_2O_2$ : sample, the yield of recovered cellulose increase moderately. However, the maximum yield of cellulose,  $0.913\text{ g}$  was obtained at  $180^\circ\text{C}$  when ratio of  $H_2O_2$ : sample at 10. Effect of oxidation reaction time of hydrogen peroxide in liquid phase oxidation was further discussed in 3.2.2.

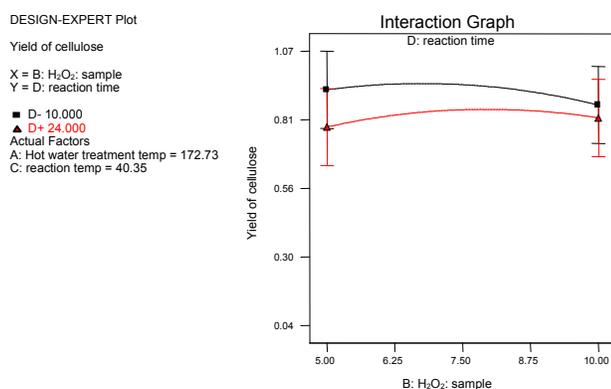


Fig. 3. Effect of Water Treatment Temperature and Ratio  $H_2O_2$ : Sample on the Yield of Cellulose Recovery.

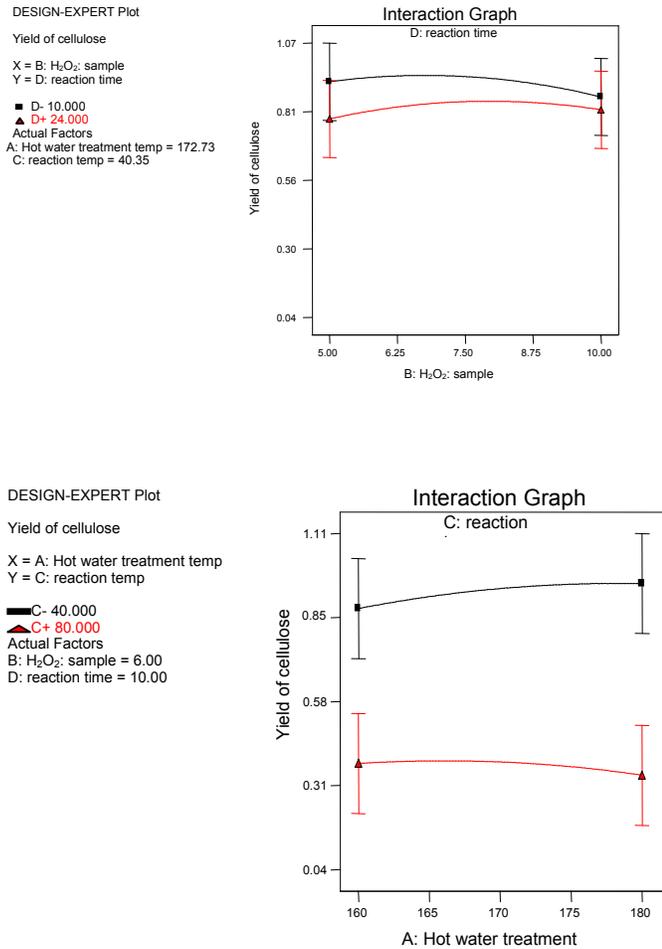
### 3.2.2. Effect of reaction time of $H_2O_2$ in liquid phase oxidation

Figure 4 shows the effect of reaction time of  $H_2O_2$  liquid phase oxidation on the yield of cellulose recovery when reaction temperature maintained at  $40.35^\circ\text{C}$  and hot water treatment temperature at  $172.73^\circ\text{C}$ . It was observed that the reaction time moderate changes on the yield of recovered cellulose. Higher yield was obtained at lower reaction temperature. However, the purity of cellulose is lower. There might be some lignin still remain contact with cellulose.

### 3.2.3. Effect of reaction temperature of $H_2O_2$ liquid phase oxidation

By maintaining the reaction time at 10 hrs and ratio of  $H_2O_2$ : sample as 6, the effect of the reaction temperature was studied as shown in Fig. 5. It was observed that the reaction temperature response distinctively with respect to hot water treatment temperature. Higher yield of recovered cellulose was obtained as low reaction temperature. The yield of recovered cellulose declined slightly as the

reaction temperature increased. Higher yield at higher reaction temperature with  $H_2O_2$  as oxidation agent may lead part of the recovered cellulose of palm kernel cake decomposed [24,25]. The decomposed cellulose was converted as water soluble organic compound.



**Fig. 5. Effect of Reaction Temperature and Hot Water Treatment Temperature on Yield of Recovered Cellulose.**

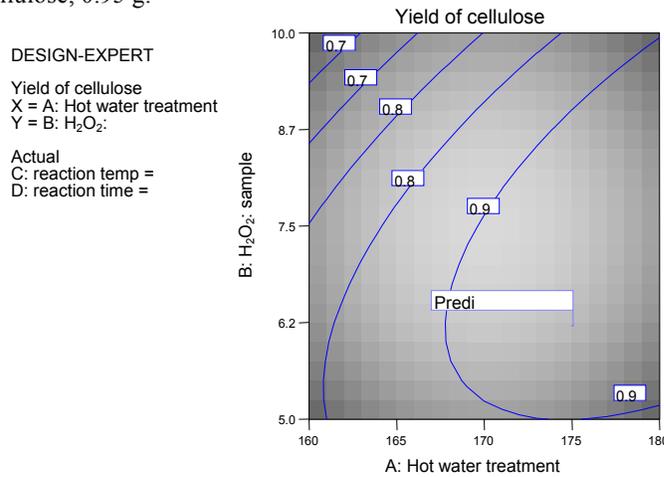
### 3.3. Optimization of the process parameter on the cellulose recovery

Numerical optimization is performed in order to obtain minimum reaction time and maximum cellulose yield as shown in Table 3. As can be seen, the higher temperature of hot water treatment must be used in order to ensure all the hemicellulose removed and dissolved into water. A short reaction time with high ratio of  $H_2O_2$ : sample is recommended to reduce the time consumption and the reaction temperature should be medium to avoid  $H_2O_2$  depolymerize cellulose from palm kernel cake.

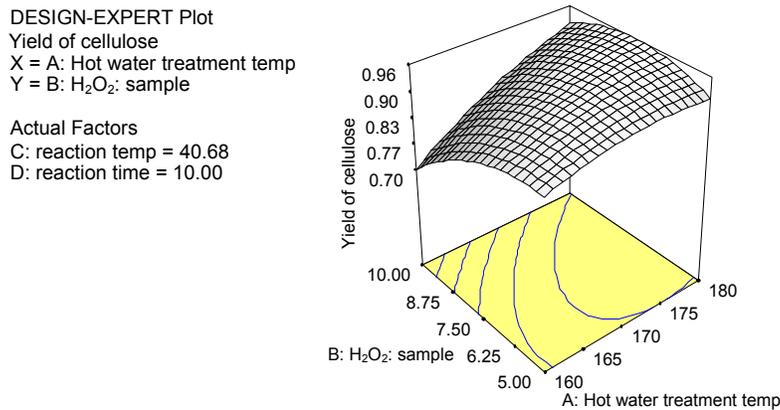
**Table 3. Solution of Optimization on Yield of Cellulose Recovery.**

Process variables	Optimum value	Goal	Lower limit	Upper limit
Hot water treatment temperature	174.74°C	In range	160	180
H <sub>2</sub> O <sub>2</sub> : sample	6.20	In range	5	10
Reaction temperature	40.68°C	In range	40	80
Reaction time	10 hrs	Minimum		
Yield of cellulose	0.95g	Maximum		

Figures 6 and 7 present the contour and three dimensional surface plot of optimization yield of cellulose in liquid phase oxidation using Box-Behnken design. Based on the criteria stated in Table 2 and shown in Figs. 6 and 7, the optimum process condition was obtained using Box-Behnken design at 174.74°C hot water treatment temperature, ratio of H<sub>2</sub>O<sub>2</sub> to sample is 6.20, reaction time in 10 hrs and 40.68°C reaction temperature to obtain optimized yield of cellulose, 0.95 g.



**Fig. 6. Contour Plot of Optimization Yield of Cellulose in Liquid Phase Oxidation Process.**



**Fig. 7. Three Dimensional Surface Plot of Optimization Yield of Cellulose in Liquid Phase Oxidation Process.**

#### 4. Conclusions

Liquid phase oxidation using H<sub>2</sub>O<sub>2</sub> was presented for separating and recovering cellulose from palm kernel cake. The method consists of hot water treatment and oxidation with H<sub>2</sub>O<sub>2</sub> in liquid phase. As an option, water soluble organic compounds obtained in oxidation stage. Hemicellulose from palm kernel cake was converted as saccharides through the hot water treatment, leaving lignin and cellulose for oxidation stage. Lignin from palm kernel cake was dissolved as soluble organic compound after the oxidation with H<sub>2</sub>O<sub>2</sub>. From RSM analysis, the effect of the reaction parameters on yield was studied and performed. It shows the yield of cellulose increase when increase of ration H<sub>2</sub>O<sub>2</sub>: sample, hot water treatment and reaction time. Yield of cellulose however, decreased if continue increasing H<sub>2</sub>O<sub>2</sub>: sample, reaction temperature and reaction time. Therefore, reaction factors have to adjust strictly to obtain optimized yield of recovered cellulose with desired properties. From numerical optimization, the optimum reaction condition was obtained for maximum yield is 0.90 g. The optimum reaction conditions are hot water treatment temperature at 175°C, ration of H<sub>2</sub>O<sub>2</sub>: sample at 6.93, reaction temperature 41°C with reaction time of 10 hrs.

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