

# Enhanced Delignification Selectivity of Alkali-Oxygen Pulping by Wet-Storage Pretreatment with Bleaching Wastewater

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Wet-storage is the most common way to maintain sugarcane bagasse in the pulping and paper-making industry, although there are few studies focused on the selectivity of delignification in pulping for bagasse treated by wet-storage. In this study, wet-storage of bagasse was carried out before alkali-oxygen pulping. The influence of wet-storage pretreatment on the chemical compositions, morphology of bagasse, and the consumption of NaOH in alkali impregnated bagasse meal were investigated. The wet-storage of bagasse resulted in significant improvement in delignification selectivity of alkali-oxygen pulping. After the bagasse was pretreated by wet-storage with bleaching water, the screened yield and crystallinity of the resulting alkali-oxygen pulp were increased.

*Keywords:* Bagasse; Wet-storage pretreatment; Alkali-oxygen pulping; Delignification selectivity

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## INTRODUCTION

Bagasse is a plentiful agricultural waste in Brazil, India, China, and other countries (Martínez *et al.* 2003). With the shortage of wood fiber in China, bagasse plays an important role in the paper-making industry (Xu 2001; Sridach 2010) due to its fiber composition and excellent properties (Vallejos *et al.* 2012; Rocha *et al.* 2012; Fu *et al.* 2013). The application of bagasse in the pulping and paper-making industry greatly alleviates the shortage of raw materials. In fact, bagasse pulping has attracted much attention from researchers (Khristova *et al.* 2006; Ogunsile *et al.* 2010; Khakifirooz *et al.* 2012; Li *et al.* 2015). Bagasse pulping has been carried out mostly using the soda, sulfate, and neutral sulfite methods. However, industrial processes for bagasse pulp production face several problems such as high cooking temperature, low pulp yields, and environmental pollution (Rainey *et al.* 2004; Yue *et al.* 2016).

Alkali-oxygen pulping is a particularly adequate method for bagasse (Chen *et al.* 1994; Vilay *et al.* 2008) due to the injection of oxygen before the pulping process. It helps delignification of fibrous raw materials and reduces the chloronome groups in the resulting pulp. Based on the mechanism of alkali-oxygen pulping, permeation enhancement of cooking liquor in bagasse is one of the keys to improve the delignification selectivity in alkali-oxygen pulping (Zhang and Chen 2007; Yue *et al.* 2016). Efficient alkali impregnation by pretreatments is beneficial for uniform pulping and shortening cooking time (Gullichsen and Sundqvist 1995; Malkov *et al.* 2001). The pretreatment methods investigated for bagasse include steam explosion (Martín *et al.* 2002; Sendelius 2005; Hernández-Salas *et al.* 2009), liquid hot water (Laser *et al.* 2002), peracetic acid (Teixeira *et al.* 1999), and ammonia water (Kurakake *et al.* 2001). Cui *et al.* (2012) reported that wet-storage is an easy and safe way to increase alkali impregnation.

In this study, wet-storage pretreatments with bleaching wastewater (BW) and tap water (TW) were used for alkali-oxygen pulping of bagasse to enhance the delignification selectivity of alkali-oxygen pulping. The mechanism of the pretreatments and their effects on the delignification selectivity was investigated by the Brunner-Emmett-Teller (BET) method, scanning electronic microscopy (SEM), alkali impregnation and chemical composition of pretreated bagasse, and X-ray diffraction (XRD) of the resulting pulp.

## EXPERIMENTAL

### Materials and Methods

Sugarcane bagasse was provided by the Yunnan Xinping Nan'en Sugar and Paper Co., Ltd. (Yunnan province, China). Air-dried and de-pithed bagasse was stored in sealed plastic bags. Other chemical reagents were analytical grade and made in China.

#### *Wet-storage pretreatment of bagasse*

Bleaching wastewater (BW) from the OP bleaching stage was provided by Yunnan Xinping Nan'en Sugar and Paper Co., Ltd. (Yunnan province, China). A total of 1.0 kg bagasse (based on oven-dried bagasse) was pretreated with BW and tap water (TW) at the ratio of solid to liquor 1:6 in 16 L cubic container for 8 and 16 days, respectively. The pretreated bagasse was washed by tap water until it reached neutral pH and then air-dried. The relevant chemical compositions were determined according to TAPPI standards T4 wd-75 (1975), T17 wd-70 (1970), T13 wd-74 (1974), and T19 wd-71 (1971). The solid content of the permeate was determined according to TAPPI T692 om-04 (2004).

#### *BET and SEM of the wet-storage pretreated bagasse*

The samples were dried in vacuum at 120 °C for 2 h before testing. The specific surface area and pore size of the pretreated bagasse were measured by the Brunauer-Emmett-Teller analysis (BET) of nitrogen adsorption isotherms (JW-BK222, Specific Surface and Pore Size Instruments, Beijing, China). The morphologies of the pulps and pretreated bagasse were observed by a tungsten filament scanning electron microscope (SEM) (VEGA-3SBH, Tescan Corporation, Brno, Czech Republic). The crystallinity of pretreated bagasse was tested by X-ray diffraction analysis (XRD) (DMAX-2500, Rigaku, Tokyo, Japan) at a generator voltage of 40 kV, tube current of 40 mA, divergence slit of 0.19 mm, continuous scan of scan range of 10° to 40°, scan step size of 0.01°, and scan rate of 7 s/step.

#### *Alkali impregnation of bagasse*

The alkali impregnation of pretreated bagasse (10.0 g, based on oven-dried bagasse) was performed in a 500 mL flask in water bath at 80 °C with alkali charge of 23% and bagasse consistency of 12%, and the alkali impregnation times were 5, 10, 15, 20, and 60 min. After alkali impregnation was completed, the slurry was filtered immediately, and the filtered liquor was collected. The alkali concentration in black liquor was determined according to TAPPI T625 cm-14 (2014).

Pretreated bagasse was pulverized by micro plant sample mill (FZ102, Ke heng Co., Beijing, China), and then screened to obtain the 40/60 mesh fraction. The NaOH concentration of the spent liquor was used to calculate the amount of NaOH consumption during alkali impregnation, according to the following equation:

$$M = \frac{(C_1 V_1 - V_2 C_2) * 100}{m_0} \quad (1)$$

where  $M$  is the NaOH consumption in the alkali impregnation in g/100 g oven-dry test sample,  $V_1$  is the initial volume of NaOH (L),  $C_1$  is the initial concentration of NaOH (g/L),  $V_2$  is the liquid volume after alkali impregnation (L),  $C_2$  is the concentration of NaOH after alkali impregnation (g/L), and  $m_0$  is the weight of the dried bagasse (g).

### Alkali-oxygen pulping

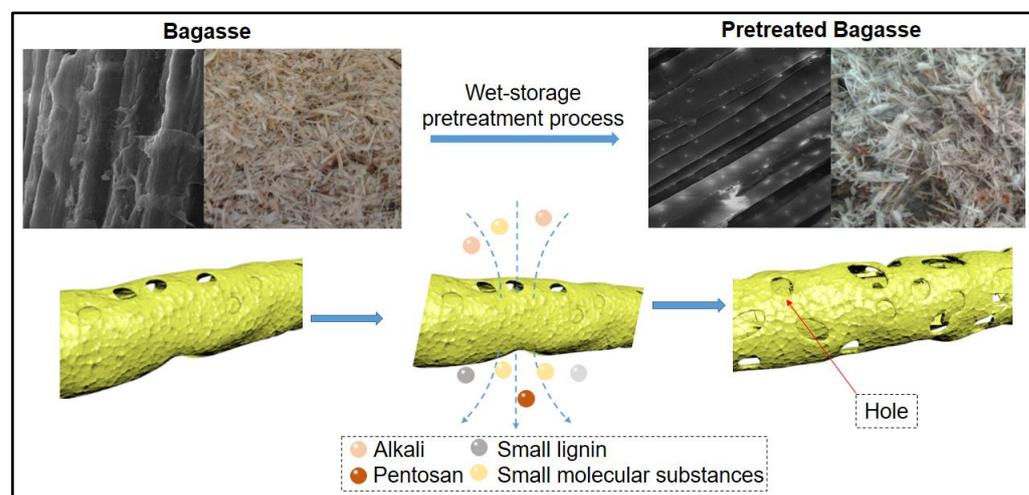
The cooking parameters of alkali-oxygen pulping were as follows: NaOH charge, 23% (based on oven-dried bagasse);  $MgSO_4$  charge, 0.5% (based on oven-dried bagasse); bagasse consistency, 12%; initial oxygen pressure, 0.6 MPa; and the maximum temperature, 100 °C for 3 h.

All resulting pulps were washed by tap water and screened by 0.20 mm sieve plate. The screened yield was determined gravimetrically on the samples following drying at  $105 \pm 2$  °C for 8h. The Kappa number and intrinsic pulp viscosity were determined according to TAPPI T236 om-13 (2013) and T230 om-13 (2013), respectively. The test of alkali-oxygen pulp's crystallinity was the same as for bagasse.

## RESULTS AND DISCUSSION

### Proposed Concept of Enhancing Alkali Impregnation Effectiveness By Wet-Storage Pretreatment

Figure 1 presents the hypothetical mechanism for improving alkali-oxygen delignification selectivity by wet-storage pretreatment. The residual chemicals in OP bleaching wastewater (BW) had an effect on dissolving small molecular substances from bagasse, which provided nutrients for the growth and reproduction of microorganisms from the inoculated bagasse during wet-storage process. The effect of BW and microorganisms on bagasse fiber improved the dissolution of some lignin small molecular and formation of porous structure of bagasse fiber during the wet-storage pretreatment. Regarding the alkali-oxygen pulping process, with the formed porous structure, more cooking liquid permeated in the inner cell wall structure of bagasse and reacted with lignin. The selectivity and efficiency of delignification were increased by the enhanced liquid permeability, which led to the improvement in pulping.



**Fig. 1.** A schematic illustration of mechanism for wet-storage pretreatment improved the delignification selectivity of alkali-oxygen pulping

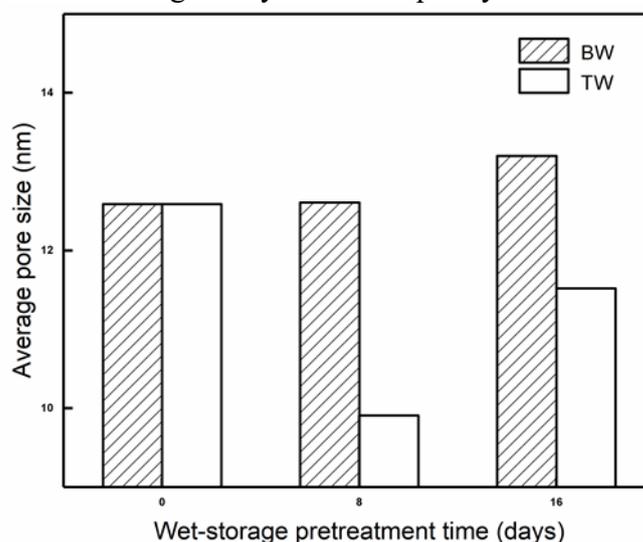
### Effect of Wet-storage Pretreatment on the Pore Diameter of Bagasse

The effect of wet-storage on the porosity and average pore size of the bagasse raw materials is shown in Table 1 and Fig. 2. With the wet-storage time extended, the average pore size of BW reached the maximum 13.2 nm at the 16<sup>th</sup> day; the average pore size 11.5 nm of TW was less than that of un-pretreated bagasse. New pores gradually appeared, and the original small aperture pores ( $\leq 10$  nm) gradually transformed into large apertures pores (10 to 50 nm or  $\geq 50$  nm) with the increasing pretreatment time. The changes in pore size and pore size distribution provided possibilities for the enhancing the penetration of the cooking liquor.

**Table 1.** Effect of Wet-storage Pretreatment on the Pore Size Distribution of Bagasse

Wet-storage Pretreatment Time (days)	Pretreatment Method	Pore Size Distribution (%)		
		$\leq 10$ nm	10 to 50 nm	$\geq 50$ nm
Un-pretreated	-	49.4	26.4	24.1
8	BW	47.0	28.8	24.2
	TW	58.4	28.7	12.9
16	BW	46.4	28.2	25.4
	TW	51.3	28.7	20.0

The above results demonstrate that the wet-storage pretreatment has an important impact on the structure of bagasse. It should be noted that the pore size and pore volume were increased resulting from the dissolution of small molecules. Furthermore, the increase of average pore size is beneficial to retaining more alkali liquor, improving fiber swelling ability and fiber quality.



**Fig. 2.** Effect of wet-storage pretreatment on the average pore size of bagasse

### Effect of Wet-storage Pretreatment on the Alkali Impregnation Effectiveness of Bagasse Meals

Alkali impregnation is the key factor of the pulping process, and it involves chemical reactions and swelling that occurs during penetration (Zanuttini *et al.* 2003). Table 2 shows that the amount of NaOH consumption increased with the extended of wet-storage pretreatment time, indicating an enhanced alkali impregnation.

**Table 2.** Effect of Wet-storage Pretreatment on the Amount of NaOH Consumption of The Alkali Impregnated Bagasse Meal

Wet-storage Pretreatment Time (days)	Pretreatment Method	NaOH Consumption (g/100g o.d. samples ) at Different Alkali Impregnation Time				
		5 min	10 min	15 min	20 min	60 min
Un-pretreated	-	3.36	4.98	5.21	5.49	6.07
8	BW	7.86	8.39	7.98	8.20	9.99
	TW	3.68	5.16	5.49	5.67	5.77
16	BW	8.97	9.14	9.23	9.76	10.93
	TW	3.60	7.10	7.57	8.13	8.42

Taking the alkali impregnation time of 60 min as an example, when the wet-storage pretreatment time was prolonged to the 16<sup>th</sup> day, the amount of NaOH consumption increased from 6.07 g/100 g (un-pretreated sample) to 10.93 g/100 g treated by BW, and increased to 8.42 g/100 g treated by TW. This phenomenon occurred because the BW with chemicals dissolves small molecular substances from bagasse, and the dissolved small molecular substances are good for the growth and reproduction of microorganisms, compared with TW. Simultaneously, the amount of NaOH consumption of bagasse increased with the time extension of wet-storage pretreatment. For the bagasse pretreated on the 16<sup>th</sup> day by BW, the amount of NaOH consumption was increased by 8.9% at the early stage of alkali impregnation (5 min to 20 min), and increased by 12.2% at the late stage (20 min to 60 min). A possible reason can be explained as follows: under the condition of natural inoculation, microbes take full advantage of bagasse and dissolved substances during the wet-storage pretreatment. More importantly, naturally inoculated microbes degrade and utilize the dissolved small molecular lignin. Therefore, wet-storage pretreatment could enhance the subsequent alkali impregnation effectiveness of bagasse in alkali-oxygen pulping.

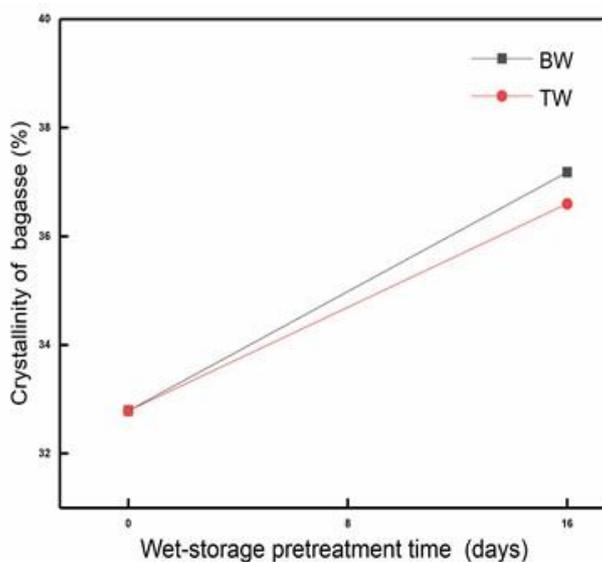
### Effect of Wet-storage Pretreatment on the Chemical Composition of Bagasse

The chemical composition analysis is the most common method to evaluate the influence of the wet-storage pretreatment on fiber raw material (Zhu *et al.* 2005). The chemical composition of bagasse in the wet-storage pretreatment is shown in Table 3. The content of the 1% NaOH extract of the raw material can be used to determine if the deterioration or decay of raw material is due to the action of oxidation or bacteria. In the whole wet-storage pretreatment process (16 days), the content of the 1% NaOH extract showed a trend of increasing first and then decreasing. Thus, different substances were dissolved with the increasing pretreatment time. The content of cellulose showed a trend of decreasing but was maintained at around 52%; this result indicated that part of the cellulose was consumed as nutrients. The content of lignin decreased at first and then slightly increased. This may be due to the fact that some lignin had been exposed and dissolved, which would increase delignification selectivity of subsequent pulping more or less. Combined with the results of the porosity change of the fibers, this data showed that chemicals of BW improved the aperture of bagasse with little effect on its chemical composition.

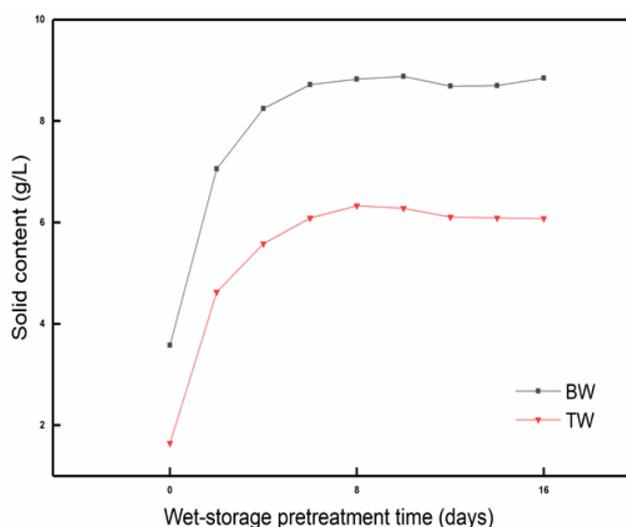
As shown in Fig. 3, the crystallinity of bagasse pretreated by BW or TW was increased to 13.4% (from 32.8% to 37.2%) and 11.6% (from 32.8% to 36.6%), respectively, at the 16<sup>th</sup> day. The change of crystallinity might be due to the effect on the change of chemical compositions by wet-storage pretreatment. Small molecular substances are dissolved by residual chemicals and microorganisms, which formed porous structures in bagasse fiber and the increased crystallinity of pretreated bagasse.

**Table 3.** Chemical Composition Analysis of Bagasse by Wet-Storage Pretreatment

Wet-storage Pretreatment Time (days)	Pretreatment Method	1% NaOH Extract (%)	Cellulose (%)	Lignin (%)	Pentosan (%)
Un-pretreated	-	34.31	52.80	20.63	31.15
8	BW	38.36	52.71	20.14	31.64
	TW	36.18	52.84	20.35	31.80
16	BW	32.73	52.55	21.30	31.28
	TW	35.59	52.45	21.19	31.33

**Fig. 3.** The effect of wet-storage pretreatment on crystallinity of wet-storage pretreated bagasse

The solid content of the permeate indicated the amount of small molecular substances removed from the bagasse into the permeate during the wet-storage pretreatment. As shown in Fig. 4, the solid content of BW and TW increased rapidly at the beginning, then stabilized (around 8.0 of BW and 6.0 of TW).

**Fig. 4.** Effect of wet-storage pretreatment on the solid content of permeate

This rapid increase in the early stage of pretreatment was attributed to the decreased solids (cane, dust, and lignin small molecules) from the bagasse and dissolution of solids into permeate. With the increase of pretreatment time, the dissolution and degradation of small molecules reached dynamic equilibrium, leading to a stabilization of content.

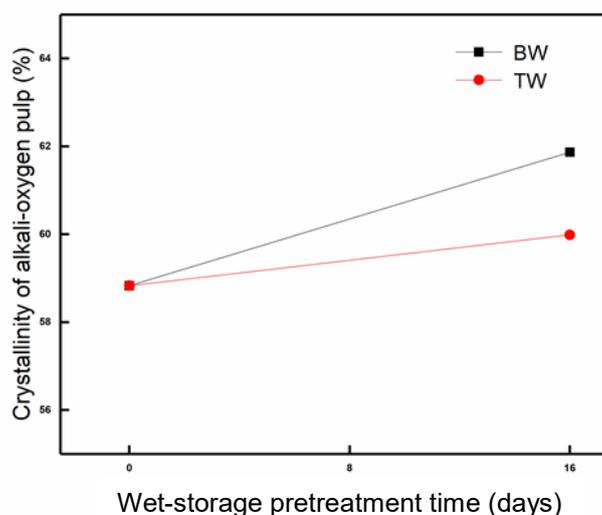
When the pretreatment time was from 0 to 8<sup>th</sup> day, the solid content increased from 3.58 g/L to 8.83 g/L for BW, after that the solid content was stabilized at around 8.80 g/L. The solid content increased from 1.65 g/L to 6.33 g/L for TW, and then the solid content was stabilized at around 6.10 g/L. This indicated that the solution ability of BW was higher than that of TW.

### Effect of Wet-storage Pretreatment on the Property of Alkali-Oxygen Pulping of Bagasse

The results of bagasse alkali-oxygen pulping indicated that the delignification selectivity was enhanced by wet-storage pretreatment, and the improvement of bagasse pretreated by BW was better than TW. As shown in Table 4, for the bagasse pretreated by BW, from 0 day to 16 days, the Kappa number of pulp was reduced by 11.54%, and the viscosity was raised by 8.9%. The decrease of Kappa number and the increase of viscosity also increased the screened pulp yield. For the bagasse pretreated by TW, although the screened pulp yield was raised, the Kappa number and viscosity were reduced, which indicates a bad effect for improving delignification selectivity.

**Table 4.** Effect of Wet-storage Pretreatment on the Alkali-Oxygen Pulping Property of Bagasse

Wet-storage Pretreatment Time (days)	Pretreatment Method	Properties of Pulping		
		Screened Pulp Yield (%)	Kappa Number	Viscosity (mL/g)
Un-pretreated	-	58.75	15.16	805
8	BW	59.90	16.52	860
	TW	58.42	15.83	827
16	BW	62.67	13.41	877
	TW	59.99	13.63	747



**Fig. 5.** The effect of wet-storage pretreatment on crystallinity of alkali-oxygen pulp

As shown in Fig. 5, the crystallinity of pulp pretreated by BW was higher than by TW. Therefore, the property of BW pretreated bagasse alkali-oxygen pulping was the best in this study, and then that of TW, and both were better than that of un-pretreated bagasse. The results suggest that more cooking liquid permeated the inner cell wall structure of bagasse and reacted with lignin during the alkali-oxygen pulping process. The enhanced liquid permeability was attributable to the presence of oxygen in alkali-oxygen pulping process; however, the increase of porosity and the change of chemical composition were the important reason for improving the delignification selectivity. The results demonstrated that the delignification selectivity was improved by wet-storage pretreatment. Furthermore, the pulp properties of BW pretreated bagasse was obviously better than that of TW pretreated bagasse. The selectivity and efficiency of delignification were increased by the enhanced liquid permeability, which led to the improvement in pulping. Han and Chen (2008) indicated that pulping properties were promoted when the bagasse raw materials were pretreated with alkali.

## CONCLUSIONS

1. The effect of wet-storage pretreatment on enhancing delignification selectivity of the bagasse in alkali-oxygen pulping was studied.
2. After the bagasse pretreated by wet-storage, the inner structures of the cell wall were opened and porosity was increased. As a result, alkali impregnation effectiveness of the pretreated bagasse raw material was improved. Compared with tap water (TW) pretreatment, bleaching wastewater (BW) pretreatment provided advantages in improving liquid permeability, as much more molecular substance dissolved from bagasse during wet-storage by the residual chemicals in BW.
3. The bagasse wet-storage pretreated for 16 days was beneficial to alkali-oxygen pulping. The screened pulp yield and property of pulp were improved, resulting from the enhanced delignification selectivity and improved liquid permeability. The alkali-oxygen pulping property of BW pretreated bagasse was better than that of TW.

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