Hemicellulose and hexenuronic acid removal selectivity to give boosting effect to ECF bleaching

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Abstract. Varies wood structure need difference way of treatment to get the appropriate quality of wood pulp. In pulp industry, wood chips should be cooked and bleached in order to have a good quality of pulp suitable for paper industry. A modified pulp cooking such as Continuous Isothermal Cooking and Super Batch Cooking followed by ECF bleaching is a good alternative for the achievement the necessary standard quality. Effort to achieve better quality in higher yield has so many times been done. Enzyme treatment using xylanase using Ecopuylp TX-200 is a good alternative that could be done in the ECF bleaching stage. Two kinds of unbleached wood pulp from Eucalyptus camaldulensis from AA Thailand, Acacia mangium from RAPP Indonesia is used in this experiment. The bleaching results are analysed for yield, viscosity, hexenuronic acid content, and brightness. The results give an indication that wood structure has a close correlation with the accessibility of enzyme and chemistries in giving peeling effect of lignin from lignin carbohydrate complex (LCC) to get high brightness of pulp with minimum fibre losses. The presence of Na_2CO_3 in the cooking liquor to gather with the xylanase has positive effect in the cleavage of hemicellulose and lignin through hydrogen bond at – OCH_3.

Keywords: wood structure, enzyme, ECF bleaching, hexenuronic acid, LCC, yield, viscosity

1. Introduction

Tropical hardwood is the main raw material in pulp industry beside soft wood and non wood. Physically their structure is very complex. Wood density, coarsness, its fiber length, fiber width, and fiber-cell wall thickness is varies and this properties determine the easyness of chemical penetration into wood structure. How chemical able to react with wood fiber is very depend on the LCC in the fiber. LCC structure it self can be synthesis for carbohydrate-rich LCC and lignin-rich LCC. These LCC theoritically has a prospec for further used in pulping [1], but naturally the structure of LCC is very depend on the wood species eventhough the possibility of LC bond in LCC in the form of ester, ether, and phenyl glycoside types the acetylation on hemicelluloses played a key role in regulating LC bonds on the benzyl ether type [2]. Chemically, wood fiber consist of cellulose, hemicellulose and lignin as its major constituent. Other constutent are glucomannan, hexenuronic and pentosan as the minor constituent. Hemicellulose in various hard wood species is differ from each other both quantitatively and qualitatively, but the major component is an O – acetyl – 4 – O – methylglucurono - β – O – Xylan. The glucuronoxylan (xylan) content is 15 – 30 % [3]. Lignin itself might be isolated and degraded through the breaking of C-C and C-O inter-unit lingkage through oxidative cleavage, hydrogenolysis, two steps redox neutral process, etc. [4]. Other research mention that the hydrogen bond at –OCH3 in lignin could be disrupted by Na2CO3 to facilaitate the dissolution and degradation of lingin [5].

The use of molecular chlorine as a bleaching agent results in the formation and release of chlorinated organic compounds, sometimes at concentrations that present an unacceptable risk to the receiving environment and the food chain [6]. This chemical has been totally substituted by chlorine dioxide in elementary chlorine free (ECF) bleaching for ecological and environmental reason. Alternatives to the use of chlorine dioxide, result in reductions the degree of chlorine substitution in the organochlorines formed. This leads to the reduction of the potential of bioaccumulation and food chain transfer, and sequencely reduces toxicity and ecological effects [6]. Research to substitute molecular chlorine as a bleaching agent has been done. Research concerining bleaching sequences such as TCF [7] and ECF [8] have also been done.

Research of ECF bleaching has so many time been done and the result have been applied in pulp and paper industries, but effort to get an improvement in bleaching is very interesting. The use of xylanase treatment in pulp before bleaching promote increase efficiency of the subsequent delignification. The kinetics of enzyme of bleached hardwood has been investigayed [9]. Effort to boost the bleaching effect of ECF bleaching of several hardwoods (acasia mangium, eucalyptus camaldulensis, and mixed tropical hardwood) using xylanase enzyme with a marginal brightness improvement has been done. In this study, the respons of these hardwoods to ECF bleaching are completely difference [10]. For SW kraft pulps, it is determined that ~60% to 65% of the overall ClO_2 charge should be applied in the D0-stage. Peroxide addition to an (EOP) can replace 0.6 to 2.5 kg. ClO_2 per kg H_2O_2. Boosting the (EO) temperature to 80°C is equivalent to a 70°C (EOP) with 0.25% to 0.30% H_2O_2,whereas a 90°C (EO) is equivalent to 0.50% – 0.75% H_2O_2 in a 70°C (EOP) (11). It is also mention that the alkaline extraction variables, such as temperature and/or peroxide inclusion, affect overall chlorine dioxide consumption and distribution for various bleach sequences [11]. Residual of peroxide from EP or EOP stage can carry over into D_1 stage that may consume additional ClO_2. The amount of ClO_2 displaced by an (EOP) stage can vary from 0.6 – 2.5 kg per kg H_2O_2 that depend on the process condition and wood species [12].

In other case the response of Eucalyptus camaldulensis and Acacia mangium kraft pulp to other type of ECF bleaching is also studied. A conventional Do stage followed by DZ or (ClO_2 and O_3), hot acid treatment before Do, and hot acid treatment before DZ, hot ClO_2 treatment and hot acid followed by hot ClO_2, after these previous stage then the pulp were bleached using D_1. In this research, the first steps of bleaching that give the best result is Do – DZ [13]. Prehydrolysis of kraft

pulp using H_2SO_5 in ECF bleaching O_3-P_{sa}-D-EP sequences for Eucalyptus globulus and Eucalyptus hybrid able to reduces the consumption of ClO_2 to only 0.,5 % to achieve target brighness 88 % ISO [14].

Cooking condition has an effect on the degradation of hexenuronic acid, xylan, glucomanan and cellulose of softwood pulp [15]. It is reported that in hardwood kraft pulp about 20 – 55 % of kappa number is attributed to hexenuronic acid, whereas for softwood kraft pulp the contribution of hexenuronic acids to the pulp kappa number was not as substantial. It is also reported that the formation of this unsaturated hemicellulose could be influenced by the extent of delignification [16]. Other researcher mention that hemicellulose is easier to be pre extracted prior to pulping to preserve it for futher use, instead of lossing it in the black liquor. This leading to the LCC structural change that in turn more accessible for pulping and enzymatic hydrolysis and have possitive impact in bleaching [17].

Cooking of plantation hardwood by pre-hydrolysis polysulfide – anthraquinone with low liquor to wood ratio show that the kappa number is clearly decrease with slightly decerase of pulp yield, but the hexenuronic acid content is not affected [18]. In Kraft cooking the chemicals used in NaOH and Na_2S and in the chemical recovery process produces green liquor that consist of Na_2S and Na_2CO_3 with the the purpose to produces $NaHCO_3$. These chemical $NaHCO_3$ and Na_2CO_3 are presence in the recovered liquor that mixed with NaOH and Na_2S for cooking purposes.

2. Experimental

2.1 Materials

Unbleached hard wood pulp request from pulp mill before ECF bleaching stage. The unbleached pulp are Eucalyptus Camaldulensis pulp from AA Thailand, Acasia Mangium pulp from RAPP Indonesia. Xylanase enzyme used is Ecopuylp TX-200 from AB Enzyme Oy Finland. The chlorine dioxide, H_2O_2 and $MgSO_4$ are supplied from the local supplier. Oxygen gas is supplied from Samator Gas Probolinggo, Indonesia.

2.2 Operating condition

The unbleached pulp was taken and performed for sequencial steps of bleaching O-D-EOP-D wth xlanase bleaching before and after O_2 delignificaton alternately. The design of experiments shown in Figure 1. All unbleached hardwoods pulp was washed thoroughly to remove the remaining black liquor to eliminate the effect of on the subsequence treatment and systimatic error in the experiment results. O_2 delignification was prepared by 15 minutes heating up periode to 90 ^{o}C at 350 kPa O_2 pressure and 1 hr at 700 kPa operating pressure and less than 5 minute to release pressure. For Eop the preparing step was 15 minute heating up periode to 80 ^{o}C at 200 kPa and 1 hr at 400 kPa and less than 5 minute to release pressure. For xylanase treatment, conditioning pH to 5.5 – 6.5 before heating at water bath for 120 minute at 70 ^{o}C and kneading every 15 – 20 minute. For D_o and D_1 bleaching, heating at 75 ^{o}C was performed for 60 and 120 minute for D_o and D_1 respectively and kneading every 15 – 20 minute.

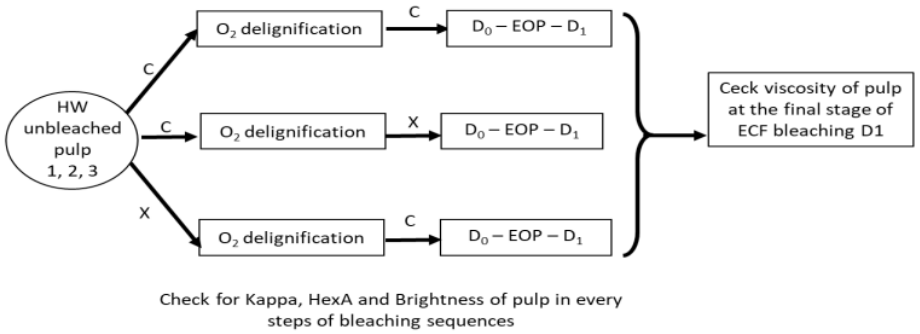


Figure 1. Experimental sequences of bleaching

Xylanase activity was analyzed in accordance with interlaboratory testing method using DNS solution [19]. Eucalyptus camaldulensis xylan (extracted from Eucalyptus camaldulensis saw dust) was used as a substrate for xylane activity determination. The operating condition used in this research were shown in Table 1. Some of the chemical composition of woods used in this experiment are shown in Table 2.

Table 1. Operating condition of ECF bleaching

Operating Condition	Xylanase treatment pre O_2 delig.	Oxygen delig.	Xylanase treatment post O_2 delig.	ClO_2 treatment	Extraction EOP	ClO_2 treatment
Temperature °C	70	90	70	75	80	75
Ph	5.5 – 6.5		5.5 – 6.5	2.5		4 – 5
Pressure, kPa	Atmosferic	700	Kneading atmosferic	Kneading atmosferic	400	Kneading atmosferic
Time (minute)	120	60	120	60	60	120
Od pulp g	250	250	225	200	175	150
Consistency %	10	10	10	10	10	10
Dosage	1 IU 10/ g od pulp	3 % NaOH	1 IU 10/ g od pulp	0.27–0.3 x kappa (act. Cl)	2 %	1.5 %
Mg SO_4		0.5 %				
H_2O_2					0.5 %	

Table 2. Woods chemical compositon

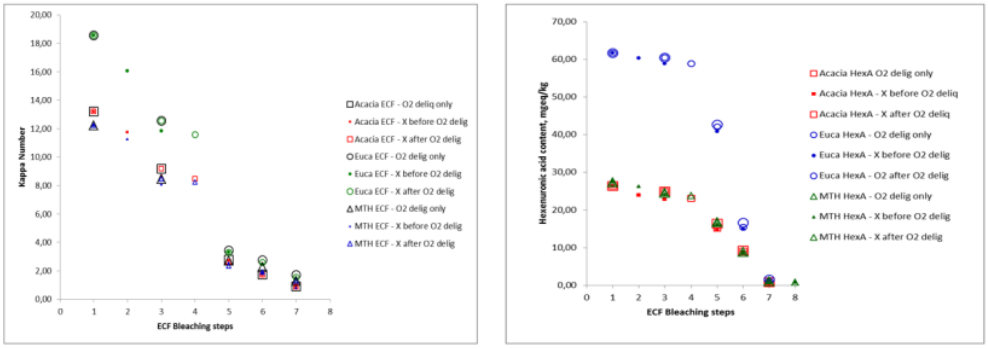
Wood composition	Acacia mangium [2]	Eucalyptus camaldulensis [1]	Birch [1]	Spurce [1]
Cellulose (%)	44	45	41	37
Hemicellulose %				
- Glucomannan (%)	1,3	3,1	2,3	17,2
- Glucoronoxylan (%)	14	14,1	27,5	10,4
- Other polysacharides (%)	0,8	2.0	2,6	3
Lignin	29,7	31,3	22	27,5
Total Extractive	4,45	2,8	3	2,1

3. Results and discussion

Conventional ECF bleaching has been applied in fiber line operation. Effort to improve the bleaching result without any pulp viscosity reduction has been done by using O_2 delignification procces in the previous step of ECF bleaching. Research of xylanase treatment in ECF bleaching has been done several times and mosty focused on the final result achievement. In this research, xylanase in used as pretreatment before and after O_2 delignification in ECF bleaching of pulps of acacia mangium, eucalyptus camaldulensis, and mixed tropical harwood harvested from Sumatra forest.

The ECF bleaching stage in this experiment is O-D-EOP-D for Acacia mangium and Eucalyptus camaldulensis and O-D-EOP-D-D for Mixed tropical hardwood pulp in order to achieve the target brightness ≥ 90 % ISO. This ECF bleaching stage is choosed in this experiment as a standard bleaching stage because it is a common ECF bleaching stage applied in fiber line operation.

Kappa number is evaluated to see the beneficial effect of xylanase treatment and the role of every step in the ECF bleaching stage to reduce the removeable lignin content. In Figure 2a it is shown that the decrease of kappa number of Eucalypthus camaldulensis pulp in ECF bleaching is faster than others. It is also shown that the decrease of kappa number is very fast up to the first step of chlorine dioxide stage that mostly known as D_o stage. After this step, the kappa reduction is slower eventhogh the temperature used in this ClO_2 stage (D_o and D_1 and even D_2) is reasonable high = 75 °C. Temperature of 75 °C was choosed for D_o, D_1, and even D_2 stages because this reasonable high temperature give the beneficial effect to ECF bleaching. This fact is supported by other research, mention that high temperature for D stages is better than hot acidic treatment prior to D stage or (A/D) stage especially in case of ClO_2 consumption [13]. Also shown that hot ClO_2 treatment offers many benefits compared to hot acid treatment followd by ClO_2 bleaching, in term of bleaching chemical consumption, bleaching yield, and development of tensile strength [20].



(a) (b)

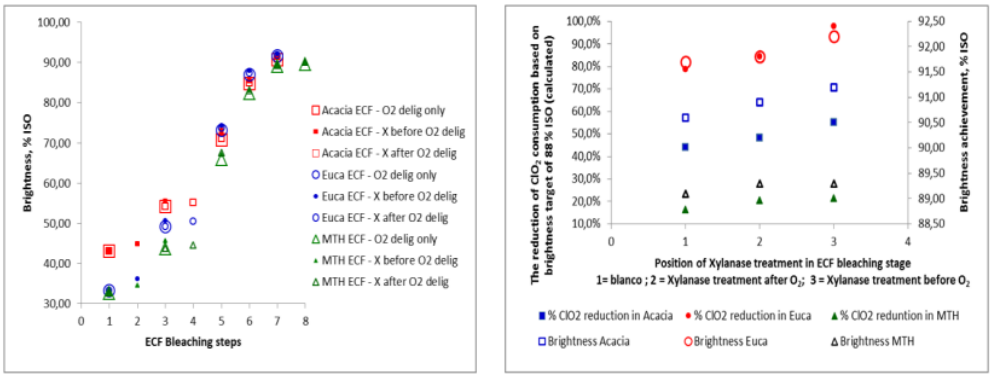
Figure 2. Effect of xylanase treatment on the ECF bleaching stage O-D-EOP-D on: a) Kappa number achievement, b) Hexenuronic acid content of Acacia mangium, Eucalyptus camaldulesnis, and Mixed tropical hardwood

Instead of hot acid treatment and hot ClO_2 bleaching, xylanase treatment is used for pre treatment in ECF bleaching. The use of xylanase treatment before O_2 delignification gave better result in case of kappa number reduction than the one if the xylanase treatment was performed after O_2 delignification and also if using conventional treatment only with out xylanase treatment.

The purpose xylanase treatment is to clevage the covalent bond between lignin and cellulose in lignin carbohydrate complex (LCC). Eventhough there is no evidance shown any cleavage of bond between lignin and carbohydrate as reported by [21,22], it is beliave that the xylanase treatment initiate the cleavage of them [10].

Figure 2b shows that there was a hexenuronic acid reduction in ECF bleaching stage when xylanase treatment is applied in this sequence prior and after O_2 delignification. In this case the prior one shown higher reduction than others. In this case Eucalyptus camaldulensis shows far better in hexenuronic acid reduction as it was compared to other pulps (acacia mangium and mixed tropical hardwood). This must be has any corelation with the physical wood structure and physical properties of cell wall that affected by lignin – carbohydrate bond [23]. The nature and amount of LCC linkages and lignin substructures affect the efficiency of pulping, hydrolysis, and digestibility of biomass [24]. In hardwood, esters and phenyl glycosidic bonds were found to be dominant in LCC, and these negatively affect kraft pulping and delignification performance because this lingkage has alkali resistance.

In Figure 2b it is shown also that the xylanase treatment prior to O_2 delignification has improved the hexenuronic acid reduction better than others. This mean xylanase treatment before O_2 delignification has initiate the cleavage of lignin and hexenuronic acid from LCC. This fact seem contradictive with the fact that the esters and phenyl glycosidic bonds that found to be dominant in LCC has negatively affect kraft pulping and delignification performance because this lingkage has alkali resistance [24]. By this fact that seem contradictive it can be inferred that the xylanase treatment able to reduce the resistance toward alkali treatment and has boosting the delignification process in the ClO_2 treatment. Instead of that, enzymatic hydrolysis may preserve xylan better from degradation as it has been mentioned by other research [25].



(a) (b)

Figure 3. a) Effect of xylanase treatment on the ECF bleaching stage O-D-EOP-D on the brightness achievement of Acacia mangium, Eucalyptus camaldulensis, and Mixed treopical hardwood, b) Possibility reduction of ClO_2 consumption as a result of xylanase treatment and the highest possible achievement of ECF bleaching using xylanase pretreatment before O_2 delignification

In Figure 3a it is shown that, the initial brightness of Eucalyptus camaldulensis after kraft cooking is lower that that of Acacia mangium but the final brightness is higher than that of Acacia mangium. The crossing point of this anomaly is at ECF bleaching step number 5 or D_o step of O-D_o-EOP-D_1 stage. The use of xylanase treatment prior and after O_2 delignification stage is studied to get an improvement in having a higher brightness achievement or a reduction of ClO_2

consumption while achieving a target brightness 90 % ISO. This target brightness of 90 % ISO is rarely needed for industrial purposes except for high brightness and high whiteness of paper, for ordinary paper the quality of pulp with brightess of 88 % ISO is commonly used. This fact has shown an indication that the xylanase treatment has initiate the cleavage of lignin and hexenuronic acid prior ClO_2 bleaching so that the achievement of brightness is higher or the ClO_2 consumption to achieve the same brightness is lower.

Table 3. Chemical consumption and brightness

STEP	Brightness of ACACIA			Brightness of EUCA			Brightness of MTH		
	O_2	X1 before O_2	X1 after O_2	O_2	X1 before O_2	X1 after O_2	O_2	X1 before O_2	X1 after O_2
EOP	84,70	85,40	84,90	87,00	87,90	87,30	82,30	83,20	82,90
ClO_2 consumption, %/g od	1,5	1,5	1,5	1,5	1,5	1,5	1,5	1,5	1,5
D_1 the best brightness achievement	90,60	91,20	90,90	91,70	92,20	91,80	89,10	89,30	89,30
Brightness Target	88,0	88,0	88,0	88,0	88,0	88,0	88,0	88,0	88,0
ClO_2 consumption to achieved target (calculated), %/g od	0,84	0,67	0,77	0,32	0,03	0,23	1,26	1,18	1,20
% ClO_2 reduction in D_1 (calculated)	44,1%	55,2%	48,3%	78,7%	97,7%	84,4%	16,2%	21,3%	20,3%

In Figure 3b it is clearly shown that in the last steps of ECF bleaching, the xylanase treatment has promoted better result when it is performed before the O_2 delignification. In case to get a brightness target of 88 % ISO, the use of xylanase treatment before O_2 delignification gave the ClO_2 consumption around 97 % for the best result in case of Eucalyptus camaldulensis pulp, 55 % for Acacia mangium and 21 % for Mixed tropical hardwood. The ClO_2 consumption is listed in Table 3.

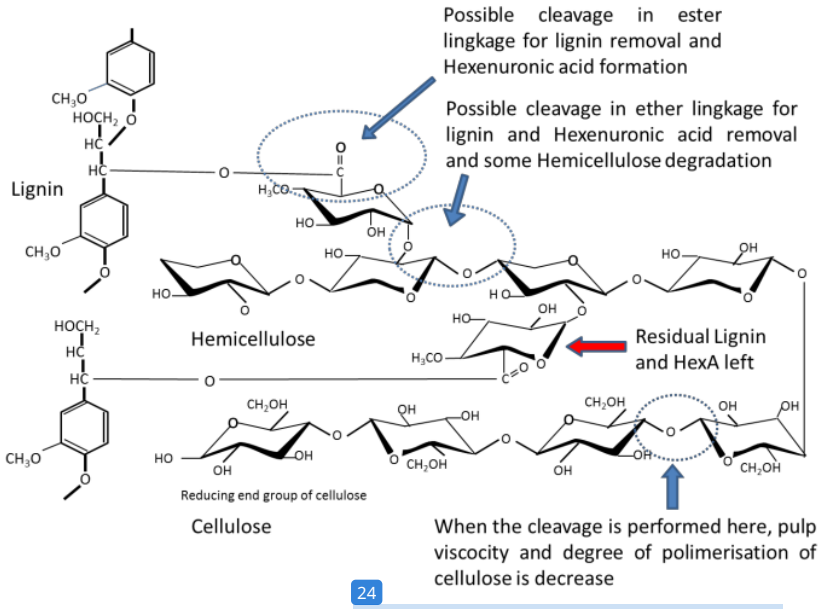


Figure 4. Overall Hardwood structure that consist of cellulose, hemicellulose and lignin molecules

The result of bleaching of several hardwoods discussed above should have any correlation with the xylan–lignin and cellulose–lignin complexes in the LCC as it has been explain by other researcher [24]. The dissolution and degradation of lignin that promoted by the initial cleavage because of xylanase treatment, it may also affected by Na_2CO_3 presence in the cooking liquor as it has been reported by other reseacher [25].

The xylanase enzyme may attack the ester and ether bond that might initiate the cleavage of lignin and HexA and also might cause hemicellulose degradation from cellulose in LCC. The HexA formation itself was happened in the cooking step. In Figure 4 shows that the possible attachment of either xylanase or ClO_2 are in ester and ether bonds. It is still not clear how the lignin and HexA removal from LCC, but this cleavage has been initiated in xylamase treatment followed by further step in ClO_2 bleaching stage. This fact can be seen from the analyses result of Kappa number, Hexa, and brightness that shows the figure of these are a little bit lower than the other one treated by only using O_2 delignification and ClO_2. In case of Eucalypthus camaldulensis, the progress is far better than other. It is suppose that it has a close correlation with the physical structure and chemical composition of this wood as compared with others. The covalent bond may also has effect to this situation [26]. This may has a correlation with the kinetic of enzyme treatment that the delignification is a function of the relative change of the reducing suger as the kinetic variable [9].

Table 4 shows that the viscocity of pulp treated by xylanase that performed before O_2 delignification give better reluts than that of others. It shows that the xylanase treatment before O_2 delignification has facilitated the cleavage of lignin from LCC so that further cellulose degradation can be eliminated. This fact is in accordance with other reasearch [25]. In case of the xylanase treatment was performed after O_2 delignification stage and then followed by D_o-EOP-D_1 stages the LCC cleavage in O_2 stage has severely occured and follows by others cleavages in the next step of stage in the bleaching sequence that mean further decrease of viscosity was occured.

Table 4. Pulp viscocity after ECF bleaching stages

TREATMENT	Bleached Pulp viscocity, kg m^{-3} after D_1		
	Acacia mangium	Eucalyptus camaldulensis	MTH
Cooked unbleached pulp	792	918	654
ECF - O2 delig only	590	542	501
ECF - X before O2 delig	604	625	624
ECF - X after O2 delig	537	589	573

4. Conclusion

From fact obtained above, it can be infered that the cleavage of hydrogen bond in LCC plays an importance role di determining the hemicellulose and lignin left in the LCC after bleaching, and thus affected the brightnees achievement. In case of Eucalyptus camaldulencis that able to achieve the most highest brightness as it is compared to the achiement in Acacia mangium and mixed tropical hardwood, it suppose has a close correlation with the kinetic of enzyme treatment that facilitate further the kinetic of clorine dioxide bleaching in D_o and D_1. The Na_2CO_3 presence in cooking liquor supposed affect the cleavage of hemicellulose. In case of the xylanase treatment, the steps is better performed before O_2 delignification to avoid to much decerase of viscosity.

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