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Pretreatment with xylanase and its significance in hemicellulose removal from mixed hardwood kraft pulp as a process step for viscose

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ABSTRACT

The upturn of viscose fiber market has triggered an augmented dissolving pulp usage over the last decade. Dissolving pulp is feasible to obtain from kraft pulp after two essential steps including hemicellulose removal and subsequent pulp activation. Prerequisite of conversion being hemicellulose reduction can be gently done by using xylanase treatment prior to alkali extraction. Herein, the significance of xylanase treatment and the optimum xylanase dose required in conjunction with subsequent alkali extraction was investigated. An increase in xylanase dose prior to alkali extraction had no significant effect on pentosans while the Fock reactivity and viscosity both improved at the dose of 50 AXU/g. Also, alkali extraction without xylanase pretreatment resulted in decreased Fock reactivity, alpha cellulose, brightness and viscosity of paper grade pulp. A moderate dose of xylanase prior to alkali extraction can thus be used to facilitate the hemicellulose removal while simultaneously protecting the native structure of cellulose.
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1. Introduction

Dissolving pulp is a special chemical pulp with more than 90% cellulose and low content of hemicelluloses, residual lignin, extractives and minerals. Being predominantly cellulosic in its chemical nature, dissolving pulp is commonly produced by acid sulfite cooking and the alkaline pre-hydrolysis kraft (PHK) cooking (Gehmayr, Schild, & Sixta, 2011). In any of the cases, it must exhibit high reactivity and accessibility towards different solvents and reagents used in various derivatisation reactions.

The dissolving pulp is more expensive to obtain than paper pulp in terms of chemical consumption, production rate, inventories, higher wood cost and storage space (Köpcke, 2010) and hence there is an interest to upgrade paper grade pulps to dissolving quality pulps. On the other hand, low pulp reactivity and high hemicellulose content in paper grade pulp make it unsuitable to be used as dissolving pulp. Even if the reactivity of paper grade pulp is enhanced by endoglucanase treatment, then also the higher content of hemicellulose hampers the viscose process and results in poor product quality. It's therefore, a prerequisite to reduce hemicelluloses down to an acceptable level prior to the reactivity improvement in the upgradation process of paper grade pulp into dissolving pulp. A well known method for the removal of hemicelluloses is cold caustic extraction (CCE) of xylan by using an approximately 2 M sodium hydroxide solution at temperatures slightly above the room temperature (Gehmayr et al., 2011; Wallis & Wearne, 1990). Although it reduces the xylan content significantly but it also results in hornification of pulp due to modification of cellulose I to cellulose II as shown in Fig. 1. This happens when the pulp is subjected to alkali dose higher than approximately 6–7% (Janzon, Puls, Bohn, Potthast, & Saake, 2008; Köpcke, 2010). Since the change in orientation of microfibrils from the *tg* (*trans-gauche*) position to the *gt* (*gauche-trans*) position in cellulose II introduces an extra inter-sheet hydrogen bond, forces involving fibrillar interactions become stronger for Cellulose II (Gehmayr & Sixta, 2012). In contrast, Cellulose I involves weaker van der waals and dipole interactions rendering it relatively easy dissociation of microfibrils to provide more accessibility to larger molecules (Köpcke, 2010; Kvarnlöf et al., 2007). Besides, the swelling in water is higher for cellulose II than cellulose I due to higher availability of OH groups and hence more hydrophilic character of former. Thus, the main factors on which the reactivity of pulp depends can be concluded as: (1) compactness of cellulose microfibrils in the cellulose fiber (2) number and size of pores in the fiber (3) quantity and type of xylan present in the pulp (4) type of solvent or reagent used. Therefore,

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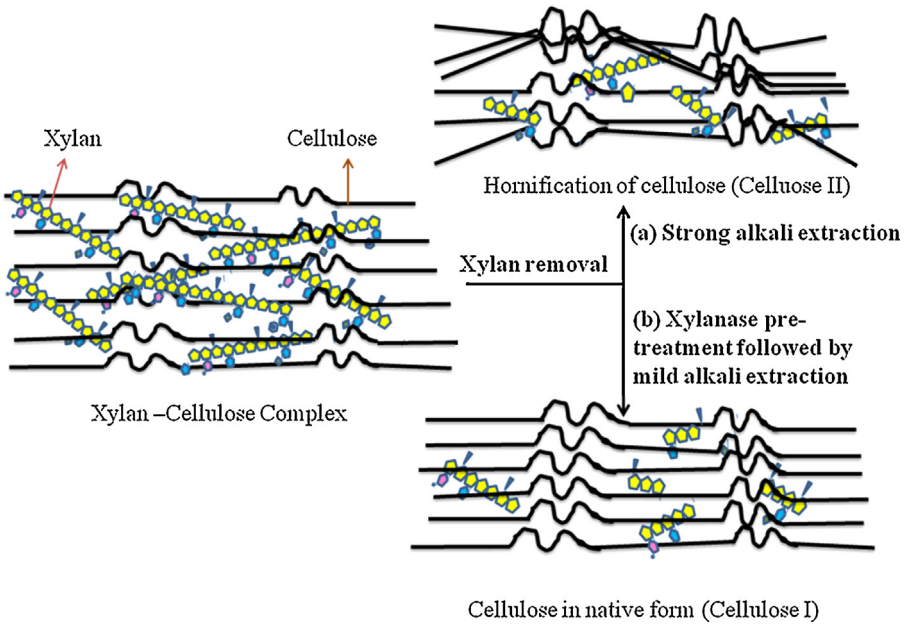

Fig. 1. Hypothesis of xylan removal from Xylan-Cellulose complex by (a) Strong alkali (b) Xylanase treatment followed by mild alkali extraction.

the hemicellulose removal is a critical process step in conversion of paper grade pulp to dissolving grade pulp and requires gentle method of treatment. Recently, several studies focusing on conversion of paper grade pulp to dissolving grade pulp have been conducted (Gehmayr et al., 2011; Gehmayr & Sixta, 2011, 2012; Ibarra, Köpcke Larsson, Jääskeläinen, & Ek, 2010; Köpcke, 2010; Wang et al., 2014). The main challenge has been found in selectively reducing the hemicellulose content down to a satisfactory extent and enhancing the pulp reactivity to achieve the appropriate quality viscose. For improvement in pulp reactivity, cellulase treatment specifically monocomponent endoglucanase with CBD (Cellulose Binding Domain) has been found to be an effective technique (Engström, Ek, & Henriksson, 2006; Gehmayr & Sixta, 2011; Henriksson, Christiernin, & Agnemo, 2005; Östberg, 2012; Kvarnlöf et al., 2007; Miao et al., 2014) whereas for hemicellulose removal various techniques have been explored including xylanase treatment followed by alkali extraction (Gehmayr & Sixta, 2011; Ibarra et al., 2010; Köpcke, 2010; Wang et al., 2014).

Xylanases are hydrolytic enzymes which catalyze the endohydrolysis of 1,4-β-D-xylosidic linkages in xylan, a major component of hemicellulose found at the interface between lignin and cellulose (Collins, Gerday, & Feller, 2005). There, it protects the cellulose microfibrils against biodegradation and maintains the structural integrity of cell walls. Wood xylan exists as *O*-acetyl-4-*O*-methylglucuronoxylan in hardwoods and as arabino-4-*O*-methylglucuronoxylan in softwoods requiring a multiple enzyme system for its complete hydrolysis (Collins et al., 2005). Since endoxylanases randomly cleave the xylan backbone, the reaction products are diverse which may include xylose, xylobiose, *xylo*-oligomers, hetero-disaccharides of xylose and glucose and their positional isomers (Motta, Andrade, & Santana, 2013). The benefit of using enzyme lies in its substrate specific and environmental friendly nature. Thus, xylanases also being specific do not pose any harm to native cellulose in pulp except for some higher doses at which they are found to cause some structural modifications in cellulosic matrix (Ambjörnsson, Östberg, Schenzel, Larsson, & Germgård, 2014). Also, it has been reported that if enzyme dose is too high, a more dense fiber with reduced accessibility for the endoglucanase in the subsequent stage will result due to internal collapse of the fiber (Ambjörnsson et al., 2014) along with an increased xylan content (Ambjörnsson et al., 2014; Liu, Zhou, Qi, & Pu, 2013).

Hence, the purpose of this study was to explore an adequate dose of enzyme prior to alkali extraction to ensure the desired removal of hemicelluloses for the efficient conversion of paper grade pulp to dissolving pulp. The main focus was to investigate the effect of xylanase dose in conjunction with alkali treatment in terms of pentosan, Fock reactivity, viscosity, S10, S18, alpha cellulose and brightness. Besides, alkali extraction is an essential step after xylanase treatment since it facilitates the removal of precipitated reaction products. Many researchers have studied the effect of various dosages of xylanase on dissolving pulp properties during the conversion of paper grade pulp to dissolving pulp, however, to our knowledge, the impact of different dosages of xylanase followed by alkali extraction has not been studied so far.

2. Materials and methods

2.1. Materials

Mixed hardwood paper grade kraft pulp (Veneer: 50%; Eucalypt: 33–34%; Bamboo: 12–15%; Poplar: 1–2.5%) procured from a mill in Northern India was dewatered before air drying and finally stored at 4 °C in plastic bags. Two different commercial xylanase preparations SEB BL 1 and Pulpzyme HC 2500 were kindly provided by Advanced Enzymes, India and Novozyme, Denmark respectively. The xylanase activity as determined by the manufacturers was 10000 U/g for SEB BL 1 and 2500 AXU/g for Pulpzyme HC 2500.

All the chemicals used were of AR grade available commercially.

2.2. Treatment of mixed hardwood paper grade pulp

Paper grade pulp (PP) was characterized for its chemical properties and then subjected to different treatments including xylanase treatment, alkali treatment (AE) and xylanase treatment followed by alkali treatment to understand the effect on various pulp properties. The xylanase was charged on the basis of units of activity per g o.d. (oven dry) fiber. Two different xylanase samples i.e. SEB

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