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The potential of different hemicelluloses extraction methods in conversion of environmentally friendly ECF and TCF bleached paper-grade bagasse soda pulp to dissolving-grade pulp

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List of Abbreviations

CCE: Cold Caustic Extraction
CED: Cupriethylenediamine
DBNH: 1,5-diazabicyclo[4.3.0]non-5-enium
DMAc: N,N-dimethylacetamide
DTR: Dissolution-base Torque Reactivity
ECF: Elemental Chlorine Free
emim: 1-ethyl-3-methylimidazolium
GPC: Gel Permeation Chromatography
HCE: Hot Caustic Extraction
IDR: Initial Dissolution Rate
IL: Ionic Liquid
IONCELL-P: IONCELL-Pulp, an ionic liquid treatment for hemicellulose extraction from pulp.
MMD: Molar Mass Distribution
PDI: Poly Dispersity Index
TCF: Totally Chlorine Free
X: Xylanase enzymatic treatment

Abstract

Dissolving pulp as a purified chemical pulp with high cellulose content, is an important raw material for many applications such as cellulose derivatives and regenerated cellulose, hence paper-grade bagasse pulp as a cheap alternative non-wood raw material can be used to produce dissolving-grade pulp. In this work, the potential of ECF and TCF bleached paper-grade soda bagasse pulp in the production of dissolving pulp was studied. To find an effective method, bleaching sequences followed by the different hemicellulose extraction processes including CCE, xylanase treatment, IONCELL-P, combination of xylanase treatment and IONCELL-P were investigated. The unbleached bagasse pulp was subjected to the IONCELL-P process using mixtures of 1-ethyl-3-methylimidazolium acetate/water for the extraction of xylan from an initial of 30.5 to 7.0%. The ECF ($ODEpQP_{1.5\%}$) sequence resulted in the optimum bleached pulp in terms of physical and chemical properties and the consumption of bleaching chemicals, which was selected for subsequent treatments to remove xylan to produce dissolving pulp. In the IONCELL-P treatment of bleached pulp, the xylan content could even be reduced to 5.9% (80% removal) and in X-IONCELL-P to 3.3% (90% removal). All the extraction methods investigated in this study removed xylan with differences in the selectivity and efficiency successfully.

Keywords: Bagasse soda pulp; Cold caustic extraction; Dissolving pulp; IONCELL-P; Xylanase.

1 Introduction

Dissolving pulp is defined as a high-purity cellulose without lignin and low hemicelluloses content as raw material for cellulose-based products such as regenerated fibers, films and cellulose derivatives (esters and ethers) [1]. Over the past years, the demand for dissolving pulps has been constantly increasing [2]. The production capacity of dissolving pulp has increased from 2.7 Mt in 2001 to 6.7 Mt in 2014 and 10.0 Mt in 2021 [3]. Despite its higher value compared to paper pulp, global production of dissolving pulp still has a relatively low, but increasing in share (less than 8% of that of the total chemical pulp production) [4]. Dissolving pulp is currently produced from wood (by acid sulfite and pre-hydrolysis kraft pulping) or from cotton linters [5, 6]. However, the higher cost of cotton linters, environmentally sustainable agriculture, the high land irrigation, and pesticide requirements of cotton production limit its use [7]. The scarcity and depletion of forest resources combined with the increased demand for dissolving pulp, accompanied by environmental concerns, has encouraged the use of non-wood lignocellulosic materials as alternative sources for dissolving pulp production. Bagasse, an abundant lignocellulosic residue from sugarcane mills, is a promising source for dissolving pulp production [8-12].

Extensive research effort has been spent on the conversion of paper pulps to dissolving pulps by hemicelluloses extraction as an economically viable process [1, 13]. However, the important challenges in the modification of a paper pulp to a dissolving pulp are not only the selective removal of hemicelluloses but also the increase of pulp reactivity. Pulp reactivity can be considered as the ability of the pulp to react with derivatizing agents and is determined, among others, by methods that evaluate its solubility and the accessibility of chemicals to the cellulose in a defined solvent system. Pulp reactivity is influenced by various factors, such as the morphology of fibers, the presence of a partial cellulose-II lattice structure, the homogeneity of the pore structure of the fiber wall, the presence of polyvalent cations, hemicelluloses, residual lignin and/or resins and the hornification of the surface layers that occurs when the pulp is overdried [14].

Depending on the raw materials and preparation methods, dissolving pulp can be purified to higher quality by various methods including various bleaching sequences, cold caustic extraction (CCE), hot caustic extraction (HCE), enzymatic treatment using xylanases, alone or in combination with alkaline extraction or the novel IONCELL-P(ulp) method [1, 15, 16].

Currently, the ~~most selective~~ commercial method for the removal of hemicelluloses is CCE for the production of acetate grade pulp either after the prehydrolysis-kraft or the combined acid sulfite-HCE treatment. CCE consists of the alkaline extraction at moderate temperatures, 25 – 45°C, and high alkali concentrations of 5-10% (w/w) NaOH [15]. Depending on the cellulose source, CCE treatment at alkali concentrations above 7% leads to the partial formation of alkali cellulose, which is regenerated as cellulose-II upon alkali removal and may affect the reactivity of the pulp to derivatization, especially after drying at elevated temperatures. Therefore, the NaOH concentration should be kept as low as possible during the CCE treatment to avoid the formation of cellulose-II to a large extent.

In several studies, enzymatic treatment as an environment-friendly method demonstrated to be beneficial for the selective reduction of the hemicelluloses content without damaging effects on cellulose [14, 16, 18]. Although efficient removal of hemicelluloses from the pulp cannot be achieved by a single xylanase treatment step, as only the surface xylan can be reached by the enzyme, enzymatic treatment followed by a CCE treatment might be able to reduce the hemicellulose content in the pulp to a very low level [17].

Recently, the search for new solvents for polysaccharides, such as ionic liquids, has attracted much research interest. Ionic liquids are room temperature salt fluids, which can dissolve the wood components [18, 19]. The dissolution of cellulose in ionic liquids was first reported in 2002 [20]. In general, ionic liquids (ILs) with a high H-bond basicity such [emim] DMP (1-ethyl-3-methylimidazolium dimethylphosphate), [emim] OAc (1-ethyl-3-methylimidazolium acetate), [DBNH] OAc (1,5-diazabicyclo[4.3.0]non-5-enium acetate) exhibited good dissolution properties towards cellulose.

Froschauer et al. [21] suggested a novel process for the selective removal of hemicelluloses from bleached paper pulps using a mixture of [emim] OAc with an appropriate amount of water. In a mixture with a non-solvent, the solubility of the IL can be adjusted so that only polysaccharides of a certain molecular mass can be extracted. This method, called the IONCELL-P (IONCELL-Pulp) process, employs an aqueous solution of an ionic liquid to fractionate paper pulp into relatively pure cellulose and hemicellulose fractions. By adjusting the water content in the ionic liquid, the dissolution capacity towards cellulose was specifically reduced and thus the selectivity towards the dissolution of hemicelluloses was improved. The IONCELL-P process enable the efficient separation and recovery of hemicelluloses and dissolving pulps with high yields and high purity. Studies demonstrated the highly selective and efficient extraction of hemicelluloses from a paper birch pulp using a 1-ethyl-3-methylimidazolium acetate/water solvent system [21, 22]. Froschauer et al. [21] reported that at suitable water concentrations of 17.5 − 20.0% in [emim]OAc, hemicellulose extraction is efficient and a high-purity cellulose fraction (only 2 − 3% xylan)

can be obtained. Roselli et al. [22] investigated the performance of IONCELL-P on pine, birch, and eucalyptus paper pulps based on two 1-ethyl-3-methylimidazolium [emim]-based ILs containing the anions acetate or dimethylphosphate and demonstrated that the most suitable IL depends on the pulp type. Under optimum conditions, the xylan content decreased from 25.4% to 1.3% in birch pulp, from 16.6 to 2.4% in eucalyptus pulp and from 8.1% to 0.9% in pine pulp.

This study focuses on the production of dissolving pulp from bagasse and the efficient removal of xylan by different extraction methods such as CCE, IONCELL-P, Xylanase (X) treatment from unbleached, Elemental Chlorine Free (ECF) and Totally Chlorine Free (TCF)-bleached bagasse soda pulps.

2 Marterials and methods

2.1 Materials

The unbleached bagasse soda pulp was obtained from a pulp and paper mill (Pars Paper Co., Haft Tapeh, Iran). The pulp was air dried and stored as pulp sheet in plastic bags at room temperature. After being sent to Finland, the pulp sheets were converted into pulp slurry with a hydro-pulper, and finally the water was drained and the pulp with a consistency of 35-40% was stored in the refrigerator for the next steps. Characteristics of the unbleached bagasse soda pulp are summarized in Table 1. Xylanase was purchased from Pulpzyme (by Novozymes, Denmark). The enzyme activity was determined by manufacturer and expressed in Active Xylanase Units (AXU) per unit mass of material as 2 000 AXU/g.1-ethyl-3-methylimidazolium acetate ([emim][OAc], 95%) was supplied by BASF (Germany).

Table 1

2.2 Methods

2.2.1 Pulp bleaching

The unbleached bagasse soda pulp was bleached using ECF ($DEpQP_{1.5\%}$, $DEpQP_{4\%}$, $ODEpQP_{1.5\%}$, $ODEpQP_{4\%}$, OA/DEpP, $ODEpD_1$) and TCF ($OQOPQP_{1.5\%}$, $OQOPQP_{4\%}$) sequences. The bleaching conditions are summarized in Table 2.

Table 2.

2.2.2 IONCELL-P extraction

Hemicelluloses were extracted from the unbleached bagasse soda pulps by IONCELL-P treatment in aqueous solution of [emim][OAc] containing 13 – 20% water. Bleached pulp was treated solely with [emim][OAc] containing 13% water. The pulp suspension (pulp : solvent ratio = 1 : 20) was prepared in 50 mL test tube, which is incubated at 60°C for 3h under constant mixing. The pulp and IL were then separated from the suspension by a syringe-filter with 20 µm porosity. Subsequently, the extracted pulp was washed with IL solvent (with the same concentration (13 – 20% water) as in the extraction) and pulp: solvent ratio = 1: 20) and then twice with 20 ml of hot water (about 90°C). The residual pulp was air dried. The effluents (IL and washing water) were combined to induce the precipitation of the xylan fraction, which is then collected by centrifugation (15 min, 4500 rpm). The regenerated xylan fraction was washed twice with hot water, then air-dried. The yields of the isolated xylan and pulp fractions were determined gravimetrically.

2.2.3 Cold caustic extraction (CCE) treatment

The alkali extraction of pulp was conducted in 12% NaOH solution. The pulp suspension of 5% consistency was prepared in a steel container and subjected to constant mixing, which was incubated at 60°C for 60 minutes. The extracted pulp was collected by filtration with a nylon bag. The pulp was washed (at 5% consistency) firstly with hot water (about 90°C) and then with 3% acetic acid (with room temperature). All the liquid filtrates were collected and combined, from which the xylan precipitation was induced by ethanol addition. The xylan fraction was collected by centrifugation at 4500 rpm for 15 minutes, and then air-dried. The yields of the pulp and precipitated xylan fractions were determined gravimetrically.

2.2.4 Xylanase treatment (X)

Unbleached and bleached pulps were treated with xylanase under the conditions: 0.01 g of xylanase/g of pulp (equivalent to a xylanase dosage of 2 000 AXU/g pulp), 50°C, 2 hours, pH of 5 and 3% consistency. The xylanase treated pulp was collected by filtration with a nylon bag and then washed twice (at 3% consistency) with hot water (about 90°C).

2.2.5 X- IONCELL -P (X-IP)

The xylanase treated unbleached pulp was subjected to IONCELL-P treatment in [emim][OAc] containing 15% water with the procedure described in section 2.2.2.

2.2.6 X-CCE treatment

The xylanase treated bleached pulp was subjected to CCE treatment with the conditions and procedure described in section 2.2.3.

2.2.7 Analyses

The gravimetric yield, total lignin and the sugar composition of each sample was determined according to NREL/TP-510-42618 standard. In this method, the sample was subjected to a two-stage total hydrolysis with 72% H_2SO_4 and 4% H_2SO_4. The monosugars were determined in a Dionex ICS3000 HPAEC-PAD system (Dionex, Sunnyvale, CA, USA). The carbohydrates composition (the cellulose and xylan content) was calculated from the individual monosaccharide contents according to the Janson formula [23]. Acid soluble lignin (ASL) was determined by measuring the absorbance at the wavelength of 205 nm using the spectrophotometer Shimadzu UV-2550 while the acid insoluble (Klason) lignin was measured gravimetrically.

The molar mass distribution (MMD) of the pulps was measured by gel permeation chromatography (GPC). Prior to the analyses, the samples are subjected to a solvent-exchange sequence. In this method, the samples were activated in water, acetone and N,N-dimethylacetamide (DMAc), respectively. Finally, the activated samples were dissolved in a lithium chloride-DMAc solution at room temperature and under gentle stirring and analyzed with Dionex Ultimate 3000 system equipped with four PLgel MIXED-A 7.5 × 300 mm columns, refractive index detector Shodex RI-101 and with LiCl/ DMAc as the eluent. Pullulan standards (343 Da–708 kDa, Polymer Standard Service GmbH, Mainz, Germany, and 1600 kDa, Fluka GmbH, Germany) were used to calibrate the system. The molar masses of pullulan standards were converted to correspond to the molar masses of cellulose ($MM_{cellulose} = q \times (MM_{Pullulan})^{p}$). The yields of the isolated cellulose and xylan fractions were used as the coefficient for adjusting the height of their corresponding GPC peaks.

The ISO Brightness, intrinsic viscosity, kappa number and ash content of the pulp were measured according to the standards ISO 2470, SCAN-CM 15:99, SCAN-C 1:00, and TAPPI 211 om-93 respectively.

The hexenuronic acid (HexA) content of samples was measured by UV Resonance Raman spectroscopy with the method introduced by Jääskeläinen et al. [24].

The BET surface area was measured with nitrogen adsorption isotherm using a Micromeritics Tristar II 3020 (Micromeritics, Norcross, USA) as described by Brunauer et al. [25].

The reactivity of the samples was determined by a dissolution-based torque reactivity (DTR) test as described by Ceccherini and Maloney [14] and performed with a Physica MCR 300 Rheometer equipped with a 4-blades-vane and basket geometry. In this method, the samples were cold disintegrated and concentrated at 10% solid content in water for storage. Then, an amount of 12.5 mL pulp suspensions was adjusted at a solids content of 1.5% at 23 °C, shaken for about 15 min, and finally placed into a vane-and-basket geometry of a Physica MCR 300 rheometer. The sample was stirred at a constant shear rate of 200 s^{-1} for 5 min, after which the solvent (12.5 mL of 1 M cupriethylenediamine, CED) was quickly injected into the system. From the moment of the injection, the dissolution of sample was recorded acquiring a torque value each 5 s and the increase was related to the dissolution of the sample fibres in the solvent. Torque profiles showed four consecutive phases: decreasing torque (first phase), linear increase of torque (second phase), nonlinear torque increase (third phase) and plateau (final phase). This analysis estimated the reactivity in terms of the initial dissolution rate (IDR) (the dissolution rate reaches its maximum) and the overall dissolution time (T0.1) by monitoring the rheological behavior of pulp fiber during dissolution in CED under constant conditions (ESI 1). The initial dissolution rate (IDR) increases with pulp reactivity, while the overall dissolution time (T0.1) decreases.

3 Results and discussion

3.1 Bleaching of bagasse soda pulp

3.1.1 ECF- bleaching of bagasse soda pulp

The characterization of ECF -bleached bagasse soda pulp is summarized in Table 3. An oxygen delignification stage prior to D reduced the pulp kappa number to 6.3, which significantly reduced the chlorine dioxide charge while

maintaining the desired bleached pulp quality. The pulp viscosity losses in all the bleaching sequences were insignificant except for the OA/DEpP sequence where the pulp viscosity dropped dramatically from 968 to 627 ml/g. Using the hot acid stage pH below 3 results in significant viscosity loss. On the other hand, higher temperatures (at 90 °C or above) impaired the pulp viscosity [26]. An increase in hydrogen peroxide dose from 1.5 to 4.0% yielded a 1.0 – 3.0% improvement in ISO brightness at the expense of pulp intrinsic viscosity.

A final P stage was successful in achieving brightness of 84-85% ISO. The improvement in ISO brightness using oxygen treatment and hydrogen peroxide stage was consistent with the previous works [27].

Table 3

Liu et al. [27] reported that the H_2O_2 reinforced oxygen delignification (1.5% H_2O_2) increased the selectivity of the delignification process by improving the brightness stability and bleachability of the pulp but slightly decreasing the strength of the paper.

3.1.2 TCF- bleaching of bagasse soda pulp

The characterization of TCF -bleached bagasse soda pulp is summarized in Table 3. TCF bleaching of bagasse soda pulp applying the $OQOPQP_{1.5\%}$ and $OQOPQP_{4\%}$ sequences resulted in a final ISO brightness of 79.3 and 82.6%, respectively (Table 3). Khristova et al. [28] reported that the application of the TCF bleaching Q_1O/PQ_2P sequence to bagasse soda, soda-AQ and AS-AQ pulp produced pulps resulted in pulps with good final ISO brightness of 73, 76, and 77% respectively, and acceptable strength properties, especially with bagasse AS-AQ pulp. Galal Eldin Karar [29] also applied the TCF bleaching sequence Q_1O/PQ_2P for bagasse soda, soda-AQ and AS-AQ pulps, yielding a final ISO brightness of 71.6, 74.1 and 76.9% respectively and a viscosity in the range of 790 -990 ml/g.

3.1.3 Comparison of unbleached, ECF and TCF bleached bagasse soda pulps

The ECF-bleached bagasse soda pulp exhibited higher brightness and lower HexA than the TCF-bleached analogues (Table 3). In a study, Beltramino et al. [30] also reported that the ECF pulps were brighter than the TCF ones. According to the results, the application of a final P stage in the ECF sequences resulted in better values in the brightness than after a final D sequence, which was consistent with prior study on the effect of ECF bleaching on optical and mechanical properties of bagasse soda pulp [31].

The HexA (hexenuronic acid) contents of the bleached pulps were considerably lower than those of the unbleached pulp (Table 3). It is known that neither alkaline peroxide (H_2O_2) treatment nor oxygen delignification can degrade HexA and the amount of hexenuronic acid is not significantly changed during these sequences. HexA reacts with electrophilic bleaching chemicals such as chlorine dioxide or ozone under acidic conditions where the intermediates are unstable. Furthermore, a hot A-treatment (hot acid washing stage) selectively removed HexA from the pulp [1]. Loureiro et al. [32] compared two industrial ECF bleaching sequences, $D_0(EOP)D_1(EP)D_2$ and OQ(PO)DP, for *Eucalyptus globulus* kraft pulps, demonstrating that HexA is not considerably affected by the oxygen stage, unlike D_0, and the slight reduction in HexA content after the oxygen delignification may be attributed to dissolution of xylan fragments in the alkaline medium.

The molar mass distributions of pulps after the different bleaching sequences were compared (Fig. 1). The peak in the higher molecular mass region represented the molar mass distribution of the cellulose fractions and the peak in the lower molecular mass region represented the xylan fractions. No severe degradation was observed in the high molecular fraction (cellulose) of all bleached pulps, except after OA/DEpP treatment, where the molar mass distribution shifted significantly to the lower molecular weight range, as confirmed by the reduction of intrinsic viscosity of more than 300 mL/g. The degradation of the cellulose chain narrowed the MMD (lower PDI), which was most pronounced in the OA/DEpP-bleached pulp. The xylan fraction (the peak in the logM of 4 – 5) was virtually unaffected by any bleaching sequence.

Fig. 1

The DTR test explains disparate forms of the pulp reactivities under dissolution. The unbleached pulp showed a low initial dissolution rate followed by a long dissolution time while the bleached pulp exhibited the opposite behaviour (Table 3). The initial dissolution rate (IDR) for the bleached pulps (beside OA/DEpP) was 7–11 times higher than for the unbleached pulp. The initial dissolution rate is related to the accessibility of the solvent towards the pulp. Higher surface area and pulp swelling facilitated the initial dissolution. The relatively short dissolution time of the bleached pulps was likely attributed to the lower Mw in comparison to the unbleached pulp [33].

The $ODEpQP_{1.5\%}$ sequence produced the optimum bleached pulp in terms of physical and chemical properties and bleaching chemicals consumption. Therefore, this pulp was selected for further xylan removal treatments to produce dissolving pulp.

3.2 Hemicellulose extraction from unbleached bagasse soda pulp

The IONCELL-P treatment effectively dissolved the hemicelluloses together with certain short-chain cellulose fractions (Table 4 and ESI 2). IL-water extraction increased the proportion of higher molecular weight fractions (DP> 2000) in the pulp with a simultaneous decrease in the polydispersity indices [7]. The addition of water to the IL promotes the formation of hydrogen bonds between the water and IL molecules creating a competitive environment for dissolution of the cellulose, resulting in a reduced dissolution capacity of the IL, and as the water content increases, the amount of dissolved cellulose in the IL was reduced to very small amounts [18]. The reduction of the water content in the IL solution resulted in an increase in the proportion of higher molecular weight fractions (DP> 2000) in the dissolved fraction (Table 4).

Table 4

The MMD of the extracted pulp (mainly containing residual cellulose) and the precipitated xylan were measured (Fig. 2). The effective isolation of the xylan fraction significantly narrowed the MMD (the PDI dropped from 14.8 to 3.4 – 4.4, Table 4).

Fig. 2

At a lower water content, the cellulose fraction with lower DP was dissolved, thus increasing the viscosity of the solution and converting the dissolved portion into cellulose-II. The phase separation by filtration and washing may be incomplete as a result, which can lead to an incorrect yield as well as a too low molar mass determination of the cellulose residue [18].

Roselli et al. [22] employed different ionic liquids, known as good cellulose solvents in water-free form, as well as NMMO to identify common features of the IONCELL-P process using bleached birch kraft pulp as a substrate, in terms of fractionation behavior as a function of water content. All tested solvent systems exhibited selective dissolution of hemicelluloses. However, the xylan removal efficiency and selectivity at the respective optimal water content varied for different solvent. The use of the solvent systems comprising [emim][OAc], [emim][DMP], [emim]Cl, [mDBN][DMP] and NMMO yielded a hemicellulose content below 5%. Among all the tested solvent systems, [Emim][DMP] was the most effective and selective (1.3% residual xylan, 1.4% dissolved cellulose). Using the example of [emim][OAc] at optimally selected water content, GPC measurements to determine the MMD of the undissolved fraction showed that the selective dissolution of hemicelluloses is molar mass dependent. Indeed, polymers with low molecular weight were found to dissolve at higher molar water content, and when the water content in the solvent system is reduced, the size of the dissolved polymers gradually increases. Accordingly, the selectivity and efficiency of xylan removal increases with the increase in the difference of the mean molar mass of cellulose and xylan.

The IONCELL-P extraction was reported to virtually yield an intact cellulose fraction [7]. However, the GPC peaks of the cellulose fraction isolated from unbleached bagasse pulp shifted towards lower molar masses (Table 4 and Fig. 2). This phenomenon could be explained that the dissolution of the unbleached pulp sample into DMAc/LiCl, particularly of the higher molecular mass fraction, was incomplete, thus resulting in the DMAc/LiCl dissolved portion of the samples might not fully represent the original high-molar-mass cellulose present in the unbleached samples.

The molar mass of hemicelluloses, such as xylan, can be shifted to lower molar masses by enzymatic treatment using xylanase, amplifying the molar mass difference between cellulose and xylan, thus boosting the efficiency of xylan removal [7]. Based on such findings, the unbleached bagasse soda pulp was subjected to a combined enzymatic and IONCELL-P treatment, yielding a pulp with only 3.3% xylan, which is the lowest among the investigated treatments (Table 5). The removal of low-DP hemicelluloses resulted in an increase in intrinsic viscosity of the unbleached pulp.

The removal of short-chain carbohydrates, significantly narrowed the MMD of all pulps (ESI 4).

(Table 5)

Yuan et al. [34] assessed the effect of two treatment methods, xylanase and CCE on the residual hemicellulose content of unbleached alkaline pre-extracted kraft bamboo pulp. The residual hemicellulose content reduced with increasing xylanase dosage. With increasing time up to 12 h, the residual hemicellulose content of the pulp reduced only slightly, probably due to molecular interactions between hemicellulose and the lignocellulosic matrix which cannot be hydrolyzed by the used xylanase system, and the limited accessibility of the enzyme to the inner cell wall

layers. In contrast, the pure CCE treatment with a 12% NaOH concentration was able to reduce the hemicellulose content to 4.66%, which was much more effective than the endoxylanase treatment.

3.3 Hemicellulose extraction from ECF-bleached bagasse soda pulp

The bagasse soda pulp bleached by the ODEpQP$_{1.5\%}$ sequence was chosen for various xylan removal treatments to produce dissolving pulp. This bleaching sequence limited the dosage of chlorine-containing bleaching agents while reaching high brightness with lowest possible DP degradation. The D-stage stabilized the cellulose after an O-stage so that the subsequent Ep-stage could develop the highest possible bleaching performance with simultaneous low DP degradation. The Q-stage before the final P-stage also provided DP-stabilizing effect due to the masking of any catalytically active transition metal ions.

In addition to effective extraction of xylan, CCE also resulted in slight pulp brightening (to 89.7% for CCE and 90.2% for X-CCE, Table 6) due to further removal of chromophore residues from the pulp. Brightening effect of alkaline extraction was previously reported by Das et al. [6] and Friebel et al. [35]. The CCE treatment at a very high NaOH concentration of 12% was more effective in the purification, i.e., removal of xylan, of bleached bagasse pulp compared to IONCELL-P treatment (3.5% residual xylan vs 5.9%, Table 6). However, CCE treatment at such high NaOH concentration induces extensive conversion to the cellulose-II structure (over 80% in the case of wood pulp) [36].

Table 6

A xylanase treatment preceding the CCE treatment further improved the pulp purity to 3.1% residual xylan content.

Cao and Tan [37] and Le Moigne [38] assigned the improvement of pulp reactivity by enzymatic treatments to various factors, such as the breakage of hydrogen bonds of OH groups, the removal of the outer cell wall layers and the destruction of fibers at the macrostructural level. Ceccherini and Maloney [14] reported higher pulp reactivity with increasing pulp surface area due to enhanced accessibility of reactive groups towards solvents and reactants, therefore enzymatic hydrolysis resulted in an improved pulp reactivity indicated by an increase in the initial dissolution rate and decrease in the dissolution time. Combined X-CCE treatment on bleached bagasse soda pulp evidently enhanced the pulp reactivity in comparison with the individual treatments of xylanase and CCE, as indicated by a 40% decrease of dissolution time and more than two-fold initial dissolution rate in the CED pulp reactivity test (Table 6). This improvement in reactivity could be explained by the swelling and the molecular mass reduction of the xylan that occurred during CCE and enzymatic treatment, which mainly affected the outer layers of the cell wall, leading to a larger available surface area.

Bleached paper-grade bagasse pulp was successfully converted to dissolving pulp, potentially suitable for textile applications (as viscose fibers) or cellulose ether (as carboxymethyl-, methyl-hydroxyethyl- or methyl-hydroxypropyl-cellulose, etc.) for food and medical applications. CCE and IONCELL-P successfully extracted xylan from the ODEpQP$_{1.5\%}$-bleached bagasse soda pulp to the dissolving pulp level (4 – 6% odp) with high intrinsic viscosity (> 1100 mL/g) and limited brightness (<87%), which are suitable for high-viscosity cellulose ether applications after an extra brightening stage to 87% (especially for the IONCELL-P pulp). For viscose fibers, beside the pulp brightness (target >90%), the cellulose DP (target 400 – 500 mL/g) shall also be adjusted to an optimal molar mass distribution for fiber spinning. Lyocell fiber application might not be feasible because of the process sensitivity (direct pulp dissolution) to impurities in the CCE and IONCELL-P pulps (>0.2%), which would require further purification, probably rendering the process uneconomical. [36]

A xylanase treatment prior to CCE (X-CCE) yielded a more purified (3.1% xylan) and brighter (90%) pulp which is potentially directly suitable for high-viscosity cellulose ether for medical or food applications. X-IONCELL-P of bleached pulp is expected to yield lower brightness (similar to the CCE vs IONCELL-P), thus requiring an extra brightening to reach the 87% target for cellulose ether pulp. The xylan content of X-CCE or X-IONCELL-P (*ca.* 3%) was still too high for cellulose acetate application (target xylan <1.5%). Further treatment to extend the xylan removal would be too economically unfavorable, thus, not recommended. [36]

4 Conclusion

The fast growth rate and low cost of sugarcane bagasse is raising interest in the production of dissolving pulp from bagasse soda pulp in paper quality. In this study, TCF and ECF bleaching with different sequences were applied to unbleached bagasse soda pulp. ECF bleaching with the sequence ODEpQP$_{1.5\%}$ was selected as the optimum bleaching sequence among the ECF and TCF bleaching sequences studied, due to its suitable properties, such as a

final ISO brightness of 87% at viscosity of 966 ml/g. The focus of this work was on the extraction and recovery of hemicelluloses from bagasse soda pulp by both CCE and IONCELL-P extraction processes, again both with or without an endoxylanase (X) pretreatment.

In the IONCELL-P process, a mixture of the cellulose dissolving IL [emim][OAc] with a 13−20% water content could selectively extract xylan when mixed with a unbleached bagasse soda paper pulp for 3 h at 60 °C. A water content of 15% was chosen due to the lower viscosity of the IL-water mixture, favourable yield and macromolecular properties of the resulting dissolving pulp. All the tested treatments on unbleached and ECF-bleached pulps were able to dissolve xylan, but there were differences in the efficiency and selectivity of xylan removal. The lowest xylan content was achieved at 3.3% after X-IL treatment of an unbleached pulp and at 3.1% after X-CCE treatment of a bleached pulp, but at a NaOH concentration in the CCE stage of 12%. The reactivity of the X-CCE treated pulp was comparable to that of a commercial dissolving pulp.

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Declarations

Ethical approval:
This declaration is not applicable as this study does not involve human and/or animal studies.

Competing interests:
The authors declare no competing interests regarding this article.

Author contributions:
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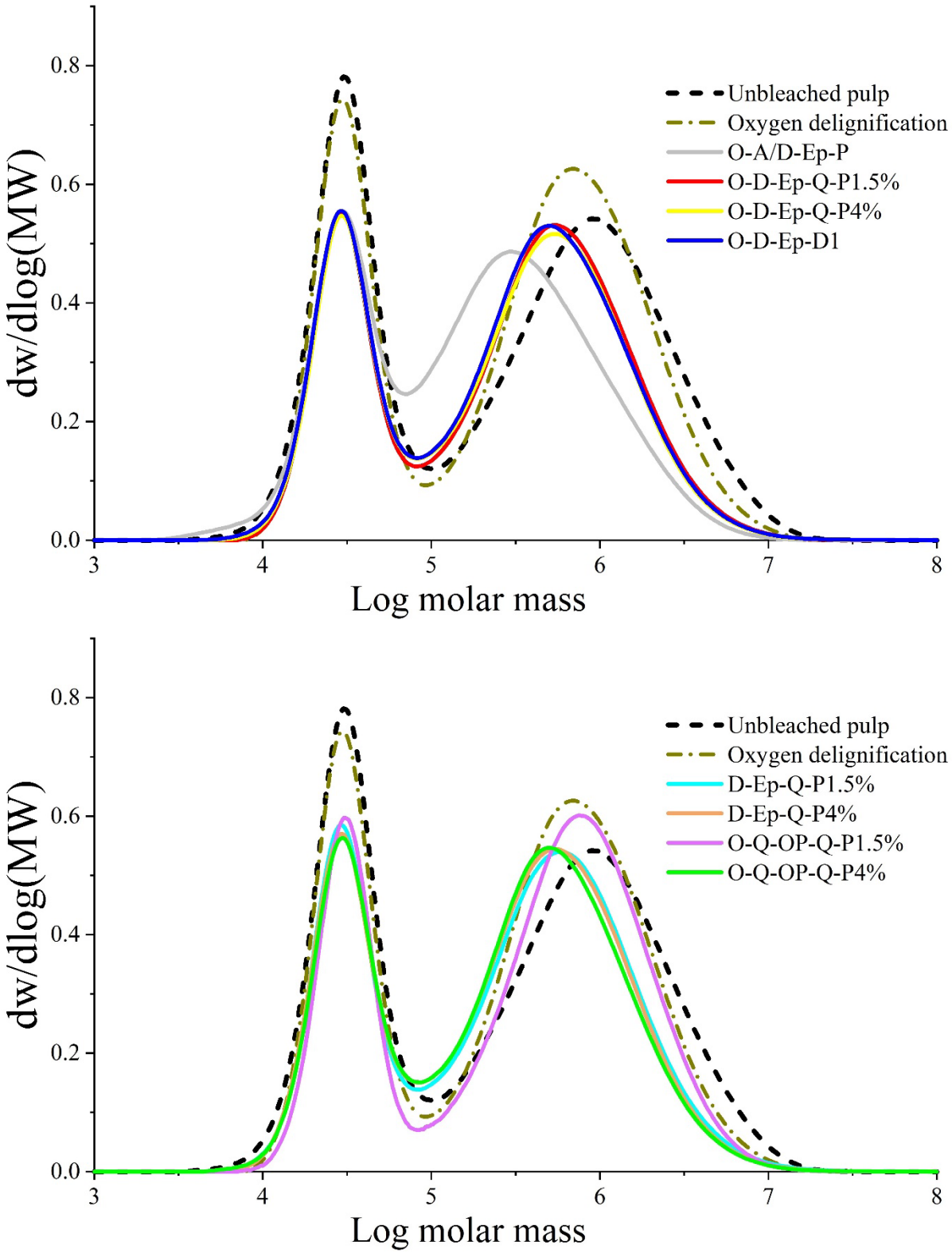

Fig. 1 Molar mass distributions of paper pulps obtained from different ECF and TCF bleaching sequences of the original bagasse soda pulps (Top: ECF with O sequences; Bottom: TCF and ECF without O sequences).

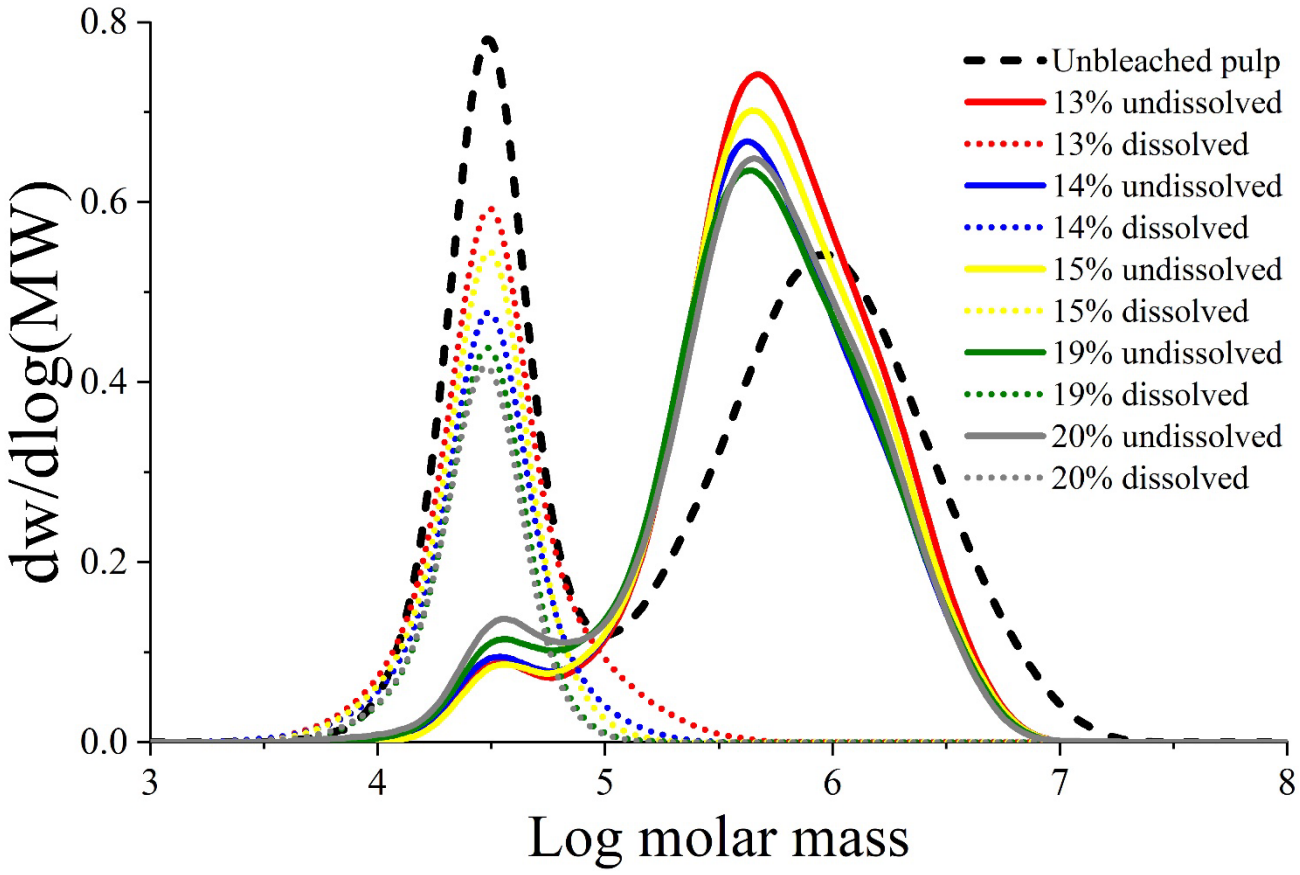

Fig. 2 Molar mass distribution of the selected undissolved (cellulose) and the dissolved (xylan) fractions after IONCELL-P extractions of unbleached bagasse soda pulp.

Table 1. Characteristics of the unbleached bagasse soda pulp. (%odp: percent on oven-dried pulp)

Properties	**Amount**
Kappa number	14.3
Total Lignin [%odp]	3.0
Cellulose [%odp]	63.1
Xylan [%odp]	30.5
Ash content [%odp]	2.2
Silica [mg/g]	1.4
ISO-brightness [%]	44.0
Intrinsic viscosity [mL/g]	968
Mw [kDa]	988
PDI	14.8
DP>2000 [%]	52.1
HexA [mmol/kg]	15.1

%odp: percentage on oven-dried pulp

Table 2. General conditions used for ECF and TCF bleaching of unbleached bagasse soda pulp. (%odp: percent on oven-dried pulp)

Parameters	ECF bleaching										TCF bleaching				
	ODEpQP, DEpQP					ODEpD$_1$	OA/DEpP				OQOPQP				
	O	D	Ep	Q	P	D$_1$	O	A/D	Ep	P	O	Q	OP	Q	P
Consistency [%]	10	10	10	10	10	10	10	10	10	10	10	10	10	10	10
Temperature [°C]	90	50	70	90	70	75	90	90	70	70	90	90	90	90	70
Time [min]	90	60	60	30	120	80	90	110/10	60	120	90	30	90	30	120
ClO_2 [κ(kappa) factor]	-	0.25	-	-	-	0.25	-	0.25	-	-	-	-	-	-	-
O_2 pressure [bar]	10	-	-	-	-	-	10	-	-	-	10	-	3	-	-
Initial pH	-	-	-	5-6	-	4.5	-	2.9	-	-	-	5-6	-	5-6	-
End pH	-	3.3	-	-	-	3.2	-	2.1	-	-	-	-	-	-	-
H_2O_2 [%odp]	-	-	0.5	-	1.5/ 4	-	-	-	0.5	1.5	-	-	1.5	-	1.5/4
NaOH [%odp]	2	-	1.5	-	1/3	-	2		1.5	1	2	-	1	-	1/3
$MgSO_4$ [%odp]	0.5	-	-	-	0.2	-	0.5	-	-	0.2	0.5		0.2	-	0.2
EDTA [%odp]	-	-	-	0.3	-	-	-	-	-	-	-	0.3	-	0.3	-
Na_2SiO_3 [%odp]	-	-		-	1	-	-	-	-	1	-	-	1	-	1

Table 3. Properties of ECF- and TCF-bleached bagasse soda pulps. (%odp: percent on oven-dried pulp)

Pulp properties	Unbleached pulp	Oxygen delignification	ECF bleaching						TCF bleaching	
			$DEpQP_{1.5\%}$	$DEpQP_{4\%}$	$ODEpQP_{1.5\%}$	$ODEpQP_{4\%}$	OA/DEpP	$ODEpD_1$	$OQOPQP_{1.5\%}$	$OQOPQP_{4\%}$
Lignin [%odp]	3.0	1.2	0.7	0.7	0.6	0.5	0.4	0.6	0.6	1.0
Cellulose [%odp]	63.2	65.1	67.4	67.6	67.6	67.7	68.3	67.8	67.6	67.5
Xylan[a] [%odp]	30.5	30.8	30.5	30.5	30.4	30.4	29.8	30.4	30.2	30
Ash [%odp]	2.2	1.8	0.2	0.1	0.2	0.3	0.3	0.1	0.4	0.4
ISO-brightness [%]	44.0	52.0	86.2	87.4	87.0	88.1	86.8	85.5	79.3	82.6
η [b] [mL/g]	968	1027	960	941	966	900	627	923	920	933
Mw [kDa]	988.0	821.2	723.0	681.0	678.0	653.0	467.0	665.0	848.0	662.0
PDI	14.8	10.9	9.7	9.1	8.9	8.8	7.8	9.1	9.9	8.9
DP>2000 [%]	52.1	54.4	50.2	49.4	50.5	49.0	36.1	48.5	58.3	48.6
DP<100 [%]	3.4	2.1	2.5	2.5	2.2	2.5	4.1	2.7	1.5	2.6
DP<50 [%]	0.3	0.0	0.1	0.1	0.0	0.1	0.9	0.1	0.0	0.1
HexA[c] [mmol/kg]	15.1	11.8	4.9	3.1	3.4	3.1	3.6	2.4	8.1	7.7
T0.1[d] [s]	3038.3	-	256.2	253.3	275.0	235.0	208.3	256.7	203.3	240.0
IDR[e] [μNm/s]	0.21	-	1.74	1.74	1.96	1.60	0.70	1.43	2.20	1.75

[a] Xylan content of pulp, expressed in percentage on oven-dried pulp.
[b] Intrinsic viscosity of pulp;
[c]Hexuronic acid;
[d] Overall dissolution time;
[e] Initial dissolution rate.

Table 4. Yields and macromolecular properties of the extracted pulps (undissolved fractions) and the precipitated xylan (dissolved fractions) obtained from the Ioncell-P treatment of the unbleached bagasse soda pulp (Total yields of undissolved and dissolved fractions that are less than 100 % are due to handling losses and deviations in yield determination; the latter is caused by non-precipitated short-chain xylan with high water content and too high yield of the cellulose residue due to partial cellulose dissolution, whereby the filter residue after washing is higher due to the increased solution viscosity and part of the dissolved cellulose precipitates again on the residue).

Water content In IL solution [%]	Undissolved fraction						Dissolved fraction					
	Yield [%]	Mw [kDa]	PDI	DP>2000 [%]	DP<100 [%]	DP<50 [%]	Yield [%]	Mw [kDa]	PDI	DP>2000 [%]	DP<100 [%]	DP<50 [%]
Unbleached pulp	100.0	988.1	14.8	52.1	3.4	0.3	-	-	-	-	-	-
IONCELL-P												
13	76.0	877.0	3.5	70.8	0.2	0.0	25.0	45.8	1.37	0.4	3.4	0.3
14	71.3	812.3	3.7	66.7	0.3	0.0	23.9	41.5	1.31	0.1	4.0	0.6
15	75.0	852.9	3.4	69.3	0.1	0.0	26.0	40.1	1.26	0.1	3.8	0.4
16	73.0	832.1	3.6	67.7	0.3	0.0	23.0	40.9	1.24	0.0	3.0	0.2
17	73.4	867.6	3.8	69.5	0.3	0.0	24.6	38.8	1.19	0.0	2.9	0.2
18	74.9	875.9	3.6	70.1	0.1	0.0	23.2	40.1	1.24	0.0	3.4	0.3
19	72.0	801.6	4.0	64.8	0.4	0.0	22.3	39	1.21	0.0	3.2	0.3
20	73.4	799.2	4.4	65.3	0.7	0.1	21.2	39.3	1.22	0.0	3.4	0.3

Table 5. The properties of the extracted pulps and isolated xylan fractions obtained from different hemicellulose-removal treatments of unbleached bagasse soda pulp. (%odp: percent on oven-dried pulp)

Parameters	Unbleached pulp	Ioncell-P (15%)	X	X-Ioncell-P
Cellulose fraction				
Yield [%]	-	74.9	83.0	75.0
Lignin [%odp]	3.0	2.8	2.9	2.1
Cellulose [%odp]	63.2	89.7	71.8	92.9
Xylan[a] [%odp]	30.5	5.5	23.6	3.3
Ash [%odp]	2.2	0.9	0.6	0.6
η [a] [mL/g]	968	1244	1136	- *
Kappa number	14.3	12.2	11.2	8.0
Hemicellulose fraction				
Yield (%)	-	24.0	-	23.8
Lignin [%odp]	-	4.8	-	4.0
Cellulose [%odp]	-	6.1	-	16.6
Xylan [%odp]	-	89.1	-	77.6

* The intrinsic viscosity of the X-IP pulp could not be reliably determined due to the lack of pulp samples.
[a] Xylan content of pulp, expressed in percentage on oven-dried pulp;
[b] Intrinsic viscosity of pulp.

Table 6. The properties of the extracted pulps and isolated xylan fractions obtained from different xylan-removal treatments ODEpQP1.5%-bleached bagasse soda pulp. (%odp: percent on oven-dried pulp)

Parameters	Bleached pulp	CCE(12%)	Ioncell-P (13%)	X	X-CCE(12%)
Cellulose fraction					
Yield [%]	88.4	71.5	71.4	92.8	77.0
Lignin [%odp]	0.8	0.6	0.7	0.7	0.6
Cellulose [%odp]	67.6	95.9	92.8	74.4	96.3
Xylan[a] [%odp]	30.2	3.5	5.9	24.6	3.1
Ash [%odp]	0.2	0.2	0.6	0.2	0.2
ISO-brightness [%]	87.0	89.7	82. 8	86.0	90.2
η [b] [mL/g]	958	1155	1183	977	1314
Reactivity measurement					
T0.1[c] [s]	273.0	260.0	-	272.0	162.0
IDR[d] [μNm/s]	1.9	2.0	-	2.5	5.7
BET surface Area [m^2/g]	2.0	2.3	-	2.6	2.1
Hemicellulose fraction					
Yield [%]	-	26.0	28.9	-	22.3
Lignin [%odp]	-	1.7	2.2	-	1.5
Cellulose [%odp]	-	2.9	12.9	-	5.6
Xylan [%odp]	-	94.3	83.6	-	91.7

[a] Xylan content of pulp, expressed in percentage on oven-dried pulp.
[b] Intrinsic viscosity of pulp;
[c] Overall dissolution time;
[d] Initial dissolution rate.

The potential of different hemicelluloses extraction methods in conversion of environmentally friendly ECF and TCF bleached paper-grade bagasse soda pulp to dissolving-grade pulp

ELECTRONIC SUPPORTING INFORMATION (ESI)

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ESI 1 Schematic representation of the four consecutive phases from the resulting torque vs time rheogram (from the injection of CED till the end of the dissolution) (Ceccherini & Maloney, 2017).

ESI 2 The sugar analysis of the undissolved (cellulose) fraction after IONCELL-P extractions of unbleached bagasse soda pulp in 1 g scale.

ESI 3 Characteristics of the extracted pulps and the precipitation from the extraction filtrate after different treatments of bleached and unbleached- soda bagasse pulp.

ESI 4 Molar mass distributions of the pulps obtained from different treatments of unbleached bagasse soda pulps, confirming the successful hemicellulose removal in each treatment.

ESI 5 Characteristics of the Commercial dissolving pulps.

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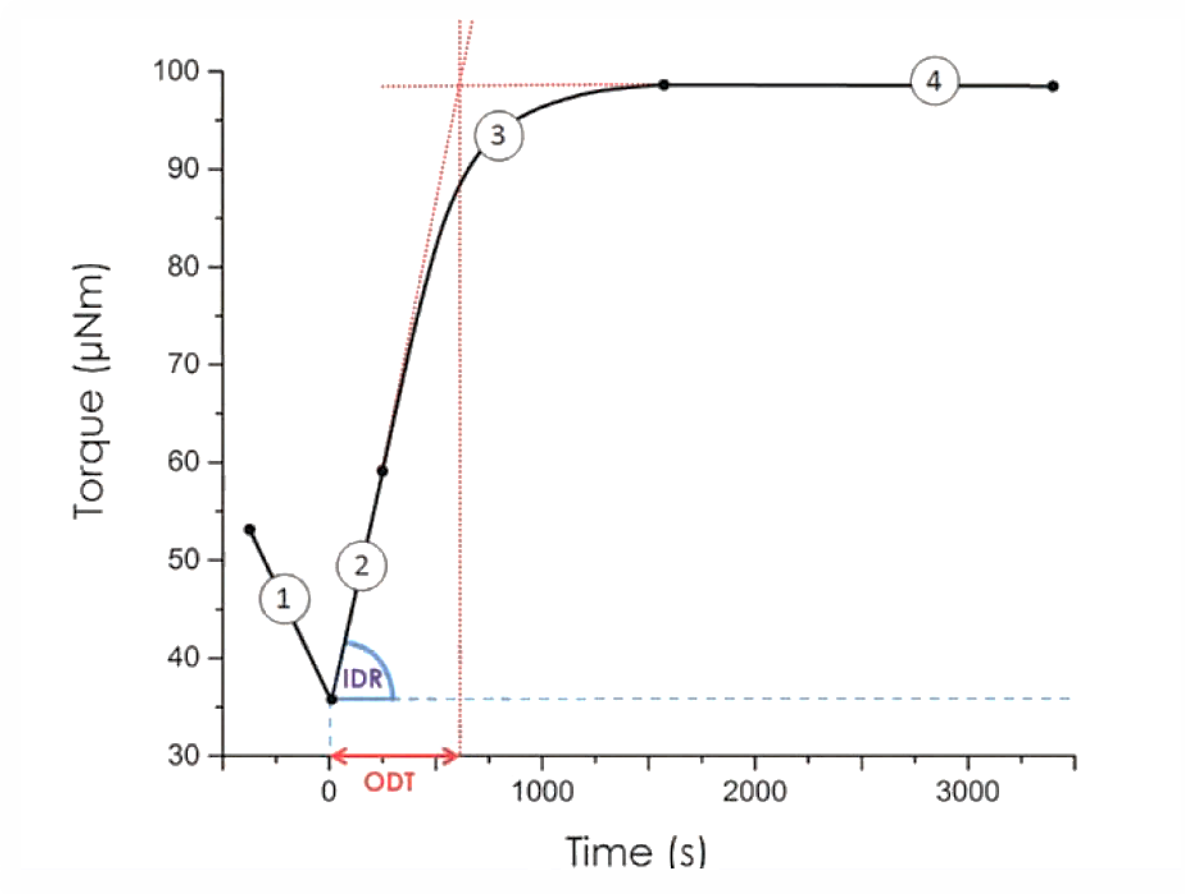


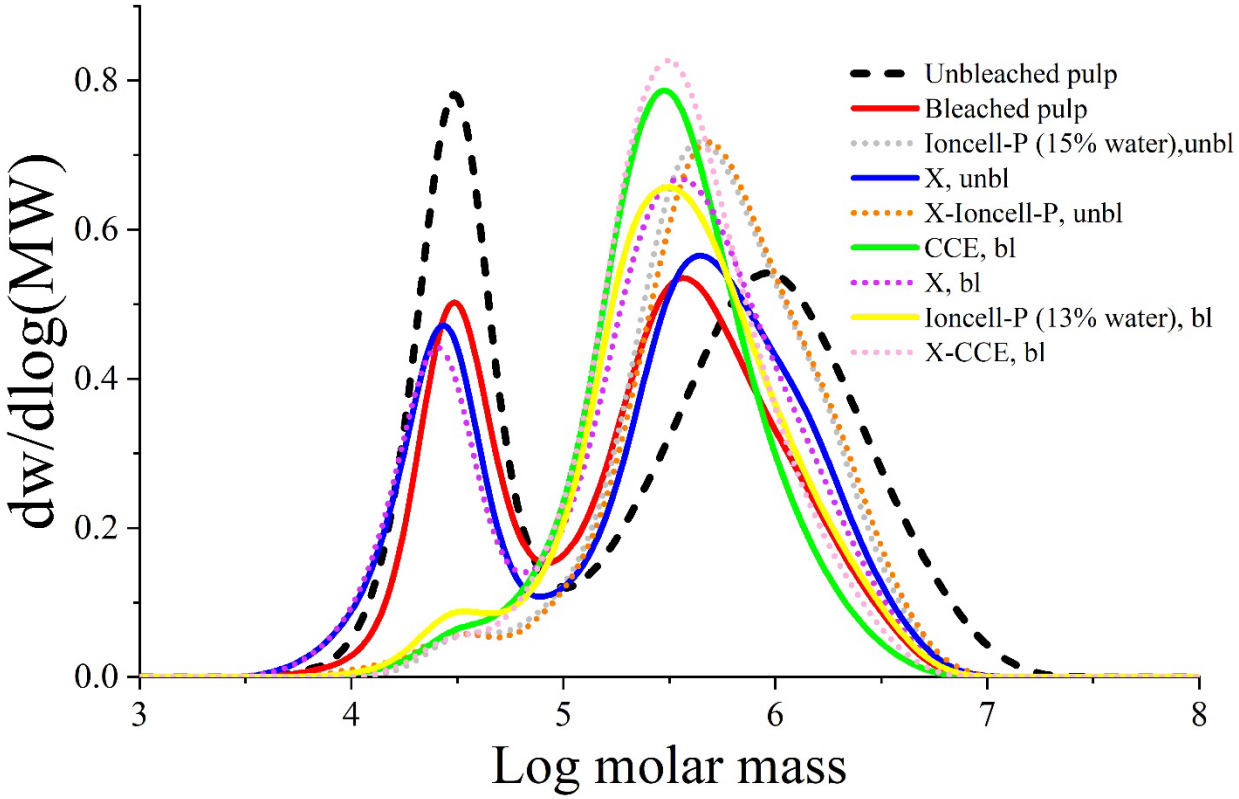
ESI 2 The sugar analysis of the undissolved (cellulose) fraction after IONCELL-P extractions of unbleached bagasse soda pulp in 1 g scale.

Water content	Undissolved fraction [% odp]			
[wt%]	Cellulose	Hemicellulose	Lignin	Ash
Unbleached pulp -	63.2	30.5	3.0	2.2
IONCELL-P				
13	88.6	7.4	2.0	0.9
14	86.7	8.7	2.4	1.1
15	88.6	7.0	2.2	1.1
16	88.4	7.1	2.3	1.1
17	87.8	7.7	2.3	1.1
18	87.8	7.9	2.2	1.0
19	86.2	9.3	2.4	1.0
20	85.8	9.5	2.3	1.3

ESI 3 Characteristics of the extracted pulps and the precipitation from the extraction filtrate after different treatments of bleached and unbleached- soda bagasse pulp.

Parameters	Unbleached pulp	Ioncell-P (15%)	X	X-Ioncell-P	Bleached pulp	CCE(12%)	Ioncell-P (13%)	X	X-CCE(12%)
Cellulose fraction									
Mw [kDa]	988	840	625	883	519	500	631	540	550
PDI	14.8	3.0	9.9	3.6	7.0	2.6	3.4	8.0	2.6
DP>2000 [%]	52.1	69.6	50.4	71.1	43.9	49.2	54.2	45.9	52.7
DP<100 [%]	3.4	0.1	5.8	0.6	2.5	0.3	0.5	5.5	0.1
Hemicellulose fraction									
Mw [kDa]	-	52	-	61	-	40	67	-	30
PDI	-	1.5	-	2.3	-	1.3	1.9	-	1.3
DP>2000 [%]	-	1.4	-	3.1	-	0.1	2.7	-	0.0
DP<100 [%]	-	3.3	-	11.2	-	0.4	0.7	-	2.4

ESI 4 Molar mass distributions of the pulps obtained from different treatments of unbleached bagasse soda pulps, confirming the successful hemicellulose removal in each treatment



unbl: unbleached pulp
bl: bleached pulp

ESI 5 Characteristics of the commercial dissolving pulps

Parameters	Wood	Southern Pine	Eucalyptus grandis	Birch
	Process	PHK-CCE	Acid Sulfite	PHK
Mannan [%]		0.6	0,2	1.1
Xylan[a] [%]		0.7	1.9	7.0
η [b] [mL/ g]		572	372	502
ISO-brightness [%]		86.8	91.9	-
Reactivity measurement				
T0.1[c] [s]		482	68	178
IDR[d] [μNm/s]		0.130	0.164	0.226

[a] Xylan content of pulp, expressed in percentage on oven-dried pulp.
[b] Intrinsic viscosity of pulp;
[c] Overall dissolution time;
[d] Initial dissolution rate.