

Effect of steam explosion pretreatment of fast-growing poplar (*Populus deltoides* x *maximowiczii*) wood on selected properties of structural substances

Jan Szadkowski

Institute of Wood Sciences and Furniture, Warsaw University of Life Sciences- SGGW

Jan Szadkowski (✉ jan_szadkowski@sggw.edu.pl)

Institute of Wood Sciences and Furniture, Warsaw University of Life Sciences- SGGW

Eva Výbohová

Department of Chemistry and Chemical Technology, Technical University in Zvolen

Viera Kučerová

Department of Chemistry and Chemical Technology, Technical University in Zvolen

Iveta Čabalová

Department of Chemistry and Chemical Technology, Technical University in Zvolen

Andrzej Antczak

Institute of Wood Sciences and Furniture, Warsaw University of Life Sciences- SGGW

Dominika Szadkowska

Institute of Wood Sciences and Furniture, Warsaw University of Life Sciences- SGGW

Michał Drożdżek

Institute of Wood Sciences and Furniture, Warsaw University of Life Sciences- SGGW

Janusz Zawadzki

Institute of Wood Sciences and Furniture, Warsaw University of Life Sciences- SGGW

Research Article

Keywords: polydispersity index, crystallinity index, polymerization degree of cellulose, pretreatment, steam explosion, structural substances

Posted Date: June 14th, 2023

DOI: <https://doi.org/10.21203/rs.3.rs-3053466/v1>

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Additional Declarations: No competing interests reported.

Version of Record: A version of this preprint was published at Wood Science and Technology on February 12th, 2024. See the published version at <https://doi.org/10.1007/s00226-024-01532-7>.

Abstract

The aim of this study was to determine the changes occurring in the wood cellulose of the fast-growing poplar (*Populus deltoides x maximowiczii*) under the influence of steam explosion (SE) pretreatment. Cellulose from native wood and after pretreatment at 160°C and 205°C was isolated. Cellulose polymerization degree by size exclusion chromatography (SEC) and cellulose crystallinity index by Fourier transform infrared spectroscopy-attenuated total reflectance (FTIR-ATR) were determined. The profiles of sugars in the native wood and in the solid fraction after pretreatment (using the acid hydrolysis method) were also determined. In addition, the profile of monosaccharides in the liquid fraction obtained after steam explosion and in the liquid fraction after acid hydrolysis of the oligosaccharides were investigated using high-performance liquid chromatography (HPLC). This allowed to determine the change in the content of hexoses and pentoses in the studied material.

The behavior of cellulose in wood subjected to steam explosion at 160°C and 205°C and isolated by the Kürschner-Hoffer method was studied by determining the absorption bands of FTIR-ATR spectra. The Lateral Order Index (LOI) of cellulose was calculated from the ratio of the intensity of the corresponding absorption bands $A_{1422} \text{ cm}^{-1} / A_{896} \text{ cm}^{-1}$. Total crystallinity index (TCI) of cellulose was calculated from the ratio of the intensity of absorption bands $A_{1372} \text{ cm}^{-1} / A_{2900} \text{ cm}^{-1}$. TCI of Kürschner-Hoffer cellulose isolated from wood subjected to steam explosion at 160°C and 205°C decreased by 5.6% and 5.0% respectively with regard to the applied temperature. LOI increased in cellulose isolated from wood subjected to steam explosion at 160°C (by 0.7%) and at 205°C (by 19.2%) in relation to the index of cellulose isolated from native wood. Kürschner-Hoffer cellulose isolated from wood subjected to steam explosion at 160°C and 205°C exhibited, respectively, a reduced degree of polymerization of about 11% and about 8%. Polydispersity index in Kürschner-Hoffer cellulose was 1% lower after both pretreatments than native sample.

Introduction

The wood biomass is a lignocellulosic complex consisting of glucose-based polysaccharide chain polymers - cellulose, pentose- and hexose-based polymers - hemicelluloses and aromatic polymer-like molecules - lignin (Ralph et al. 2008; Hendriks and Zeeman 2009). Both polysaccharides and lignin are the building materials of cell walls, and besides them there are extractives with different functions and separable using appropriate solvents.

The cellulose molecule is composed of β -D-glucopyranose mers linked by β -1,4-glucosidic bonds (Fengel and Wegener 2003; Klemm et al. 2005). The di-anhydro-D-glucopyranose form a mer repeating n-times in the cellulose molecule, accounting for its degree of polymerization. In the formula of the cellulose molecule, the two extreme glucosidic rings differ from the others, at the fourth carbon atom the -OH group is characterized by reducing properties, and at the first carbon it does not have these properties.

In wood, the degree of polymerization of cellulose is about 15000, and its average degree of polymerization varies from 9000 to 10000 (Goring and Timell 1962; Fengel and Wegener 2003).

Under the influence of physicochemical and mechanical factors acting on wood, the degree of polymerization of cellulose is significantly reduced. The action of high temperatures on cellulose causes a decrease in its degree of polymerization (Kučerová et al. 2016; Zawadzki et al. 2016, Radomski et al. 2010).

If cellulose molecules are arranged parallel to each other at small distances, then they are connected to each other by intermolecular hydrogen bonds. Hydrogen bonds are a form of association between a highly electronegative atom and a hydrogen atom bound by a covalent bond to an electronegative atom. In the cellulose molecule, the electronegative atom is an oxygen atom.

Currently, cellulose is assigned a crystalline- amorphous structure, containing crystalline and amorphous regions. Two cellulose molecules pass through a series of crystalline (oriented) areas, where the cellulose molecules are arranged parallel to each other, and areas of less oriented molecules (amorphous areas). In addition to crystalline and amorphous areas, paracrystalline areas characterized by an intermediate structure between crystalline and amorphous areas may be present in cellulose (Atalla 1993).

Several crystallographic variations of cellulose are known and are numbered with Roman numerals. Cellulose I is found in natural fibers and in this structure the cellulose molecules are arranged in parallel layers. The molecules in each layer are held together by hydrogen bonds, and the cohesion of adjacent layers is maintained by van der Waals forces. Cellulose II is the most thermodynamically stable type of cellulose and is mainly obtained from cellulose I, by mercerization (alkali treatment and regeneration, solubilization and subsequent recrystallization). Cellulose III and Cellulose IV can be formed from cellulose I and II, respectively by treatment with liquid ammonia and reaction is reversible (Hayashi et al. 1975). Cellulose V and cellulose VI can be obtained by heating cellulose III and IV, respectively (Gardiner and Sarko 1985).

Glucose is a simple sugar formed mainly by the hydrolysis of cellulose, although a small amount is associated with the presence of heterogeneous hemicelluloses.

Cellulose, due to its crystalline-amorphous structure, is characterized by the difficult property of being stiff and elastic at the same time. High degree of crystallinity negatively influences its hydrolysis process. In contrast, amorphous cellulose exhibits from 3 to 30 times the efficiency of this process with respect to crystalline cellulose (Lynd et al. 2002). The crystalline areas of cellulose are resistant to chemicals and to the process of hydrolysis, and this occurs in the amorphous areas (Zhu et al. 2008). They are also temperature resistant, with the process in amorphous areas occurring above 150°C and in crystalline areas above 180°C (Yu et al. 2010). In the available literature there is a scarce information about using FTIR-ATR method to crystallinity analysis of cellulose from fast-growing poplars wood in a context of bioethanol production. Hence, in this respect the use of the above method to study the crystallinity can be interesting and noteworthy.

In wood, the polysaccharides accompanying cellulose are hemicelluloses, the molecules of which can be built from monomers of homogeneous and heterogeneous simple sugars. The most common monomers in hemicelluloses molecules are pentoses: β -D-xylopyranose, α -D-arabinofuranose and hexoses: β -D-mannopyranose, β -D-glucopyranose, β -D-galactopyranose (Kačík and Solár 2000, Radomski et al. 2010). In this group of polysaccharides can be distinguished pentosans and hexosans and polyuronides. Among the pentosans, the main polysaccharide is xylan, whose molecule is built from β -D-xylopyranose residues, linked by an oxygen bridge 1,4. On average, seven out of ten D-xylopyranose units have acetyl groups in the C-2 or C-3 position, and every tenth unit is attached by a substituent 4-O-methylglucuronic α -1,2 bond (Fengel and Wegener 2003).

Hemicelluloses show less resistance to physico-chemical agents compared to cellulose. They dissolve in alkaline media and hydrolyze more readily when exposed to dilute acids, and they degrade at range from 160°C to 220°C (Boonstra et al. 2007; Antczak et al. 2019; Antczak et al. 2021).

Steam explosion is a physical and chemical method for pretreatment of lignocellulosic biomass (Brownell et al. 1986; Balan et al. 2020; Mankar et al. 2021). This method combines mechanical treatment with chemical treatment caused by wood autohydrolysis. The autohydrolysis of wood involves acetyl groups present in hemicelluloses and the water environment, which under conditions of elevated temperature and pressure have an action similar to that of acids. Autohydrolysis at elevated temperature results in reduction of hemicelluloses and plasticization of lignin, which forms a complex with hemicelluloses. This results in exposing the cellulose structure and reducing its degree of polymerization (Alvira et al. 2010; Tomas-Pejo et al. 2014, Kučerová et al. 2020). The polymerization degree of cellulose is a key parameter influencing the enzymatic hydrolysis of lignocellulosic biomass (Karimi and Taherzadeh 2016). It is known, that the shorter cellulose chains are more reactive to the enzymes, because the shorter chains contain lower hydrogen bonds and are more accessible to enzymes. Additionally, the reduction of cellulose polymerization degree by a pretreatment process causes a formation of more cellulose ends available to the exoglucanase. Based on literature information (Yang et al. 2011) no clear relation has been observed between polymerization degree of cellulose and enzymatic hydrolysis yield. Hence, the research undertaken in this area is still valid and worth attention.

The aim of this study was investigation of glucose and xylose contents in *Populus deltoides* $\times$ *maximowiczii* wood before and after steam explosion pretreatment. Additionally, the polymerization degree of cellulose, polydispersity and total crystallinity index (TCI) and Lateral Order index (LOI) were studied.

Materials and methods

Materials

Wood of *Populus deltoides* $\times$ *maximowiczii* was obtained from 5-year-old stems cut at the end of the growing season and grown on plantations of the Department of Plant Genetics, Breeding and

Biotechnology, Faculty of Horticulture, Biotechnology and Landscape Architecture, Warsaw University of Life Sciences, located in Wolica (52°08'42"N, 21°04'07"E). The wood was debarked and then chipped on a laboratory cutting mill. Tests were conducted on wood shavings with a fraction of the 0.43 to 1.02 mm fraction. They were dried to a moisture content of 5%, and before being pretreated with a steam explosion they were moistened to remove air from the wood pores. The weighed wood shavings were placed in a beaker and flooded with distilled water to 220 cm³ by placing the beaker on a magnetic stirrer with a heating plate, heating to 90°C for 20 min. Pretreatment by steam explosion was carried out in a high-pressure autoclave with a capacity of 250 cm³, made of acid-resistant steel (Antczak et al. 2021; Gałązka and Szadkowski 2021).

Methods

Acid hydrolysis of native wood and solid fraction obtained after pretreatment

Wood samples before and after steam explosion pretreatment were dried to constant weight at 104°C ± 1°C. Then, 0.1 g of the sample was placed in a 50 cm³ flask and flooded with 1 cm³ of 72% sulfuric acid (VI) (H₂SO₄) (Chempur, Gliwice, Poland). The samples were heated for 1 h at 30°C. After adding 28 cm³ of distilled water, they were heated for 2 h at 120°C in an oil bath (Glycerin, Agroekolab, Zvolen, Slovakia). Then were cooled to room temperature. The hydrolysates were quantitatively poured into 50 cm³ volumetric flasks and made up to the mark with distilled water. From the solutions obtained, 5 cm³ of each were taken and transferred to beaker and neutralized with barium carbonate (BaCO₃) to neutral pH, added 1 cm³ of 5% cellobiose solution (an internal standard), it was filtered and washed with distilled water. The prepared samples were evaporated on a vacuum evaporator (Laborota 4000, Schwabach, Germany). Then the precipitate obtained was dissolved in 1 cm³ of ultrapure water and filtered through a nylon filter with a pore diameter of 0.25 µm and subjected to HPLC (high-performance liquid chromatography) analysis.

Direct analysis of liquid fraction obtained after pretreatment

The determination of free monosaccharides (glucose and xylose) in the liquid fraction obtained after steam explosion at 160°C and 205°C, was carried out by taking 5 cm³ of the solution, which was transferred to a conical flask, adding 1 cm³ of 5% cellobiose solution as an internal standard. After neutralization with BaCO₃ to neutral pH, the solution was evaporated on a vacuum evaporator (Laborota 4000, Schwabach, Germany) to obtain a precipitate. The precipitate was dissolved in 1 cm³ of ultrapure water and filtered through a 0.25 µm pore diameter nylon filter and analyzed by HPLC.

Acid hydrolysis of liquid fraction obtained after pretreatment

In order to analyze the total yield of glucose and xylose in the liquid fraction obtained after pretreatment of *Populus deltoides x maximowiczii* wood subjected to steam explosion at 160°C and 205°C, assuming the presence of oligosaccharides in this liquid, their hydrolysis was performed. For this purpose, 20 cm³ of solution was taken by placing in 50 cm³ spherical flasks. 0.42 cm³ of 96% sulfuric acid (VI) was added to the solution and placed under a reflux condenser above the hob for 4 hours (the liquid boils). Then the solution was quantitatively transferred into 25 cm³ volumetric flasks and made up to the mark with distilled water. From the solutions obtained, 5 cm³ of each were taken and transferred to beaker and neutralized with BaCO₃ to neutral pH, added 1 cm³ of 5% cellobiose solution (an internal standard), it was filtered and washed with distilled water. The prepared samples were evaporated on a vacuum evaporator (Laborota 4000, Schwabach, Germany). Then the precipitate obtained was dissolved in 1 cm³ of ultrapure water and filtered through a nylon filter with a pore diameter of 0.25 µm and subjected to HPLC analysis.

HPLC analysis of monosaccharides

Determination of glucose and xylose yield was carried out with an Agilent 1200 HPLC (Agilent Technologies, CA, USA) with a Benson BP-800 Pb** column (Benson Polymeric Inc., Reno, NV, USA) at 80°C, with flow rate of 0.6 cm³/min and with using refractive index detector. The mobile phase was ultrapure water. All analysis was performed four times per sample and single standard deviations were calculated.

SEC analysis of cellulose isolated from native wood and solid fraction obtained after pretreatment

The degree of cellulose polymerization was measured by size exclusion chromatography (SEC) method. Cellulose samples were isolated using the Kürschner-Hoffer method (Saeman et al. 1954; Krutul 2002,) from native wood and solid fraction obtained after SE pretreatment. Before cellulose isolation, the samples were previously extracted with a chloroform-ethanol (93:7)_{w/w} mixture (Antczak et al. 2006). Molecular weight distribution analysis of the cellulose samples was performed after their conversion into cellulose tricarbanilates (Kačík et al. 2009; Čabalová et al. 2009). Cellulose tricarbanilates were dissolved in tetrahydrofuran and filtered through a Puradisc 25 NYL filter (Whatman International) with a pore size of 0.45 µm. SEC analysis using a DAD detector was performed at 35°C with tetrahydrofuran at a flow rate of 1 cm³/min on a two connected PLgel, 10 µm, 7.5 × 300 mm, MIXED-B columns (Agilent Technologies) preceded by a PLgel, 10 µm, 7.5 × 50 mm, Guard-column (Agilent Technologies). Data acquisitions were carried out with ChemStation software (Agilent Technologies) and calculations were performed with the Clarity GPC module (Data Apex). Numerical outputs were obtained for M_n (the number average molar mass), M_w (the weight average molar mass) and the values were recalculated to underivatized cellulose using the coefficient $k = 162 \text{ g mol}^{-1} / 519 \text{ g mol}^{-1}$.

$$M_w(\text{cellulose}) = x \bullet k$$

x - retention time

$$k = \frac{162 \text{ g mol}^{-1}}{519 \text{ g mol}^{-1}}$$

Degree of polymerization (DP_w) values were calculated by dividing the weight average molar mass by the monomer equivalent mass of anhydroglucose (2)

$$DP_w = \frac{M_w}{162}$$

2

($DP_w = M_w/162$). Polydispersity index (PDI) of cellulose was calculated as the ratio (3),

$$PDI = \frac{M_w}{M_n}$$

3

M_w/M_n . Measurements were performed four times per sample and single standard deviations were calculated.

FTIR-ATR analysis

Fourier transform infrared spectroscopy-attenuated total reflectance (FTIR-ATR) spectra of native wood, solid fractions obtained after SE pretreatment and cellulose isolated from these materials were recorded on Nicolet iS10 FTIR spectrometer equipped with Smart iTR attenuated total reflectance (ATR) sampling accessory with diamond crystal (Thermo Fisher Scientific, Madison, WI, USA). Spectra were measured in the wavenumber range from 4000 to 650 cm^{-1} . A resolution of 4 cm^{-1} and 32 scans per sample were used. Measurements were performed on six replicates per sample and average spectra were created and evaluated. The OMNIC 8.0 software (Thermo Fisher Scientific, Madison, WI, USA) was used to evaluate the spectra.

Results and discussion

The glucose and xylose yield obtained after acid hydrolysis of native wood and SE pretreated solid fractions is presented in Fig. 1a. On the basis of the data presented in Fig. 1a it can be concluded that the steam explosion process of *Populus deltoides x maximowiczii* wood, compared to native wood, increased the glucose yield from 434.2 mg/g to 455.5 mg/g (160°C) and 501.8 mg/g (205°C) respectively. However, the xylose yield decreased after pretreatment from 135.7 mg/g to 130.7 mg/g (160°C) and 110.0 mg/g (205°C).

Glucose is a simple sugar formed mainly by the hydrolysis of cellulose, although a small amount of glucose content is related to its presence in heterogeneous hemicelluloses, i.e., glucomannan. During the pretreatment process of *Populus deltoides x maximowiczii* wood, some saccharides pass into the post-reaction liquid. Figure 1b shows the data of glucose and xylose yield in the liquid fraction obtained after SE of wood at 160°C and 205°C. From the data shown in Fig. 1b, it can be seen that the glucose yield in the post-reaction liquid obtained at 205°C was over three times higher with respect to its yield in the post-reaction liquid obtained at 160°C. In contrast, the xylose yield in the post-reaction liquid obtained at 205°C was about eighteen times higher with respect to its yield in the post-reaction liquid obtained at 160°C.

The total glucose and xylose yield was obtained by concentrating the post-reaction liquid and then hydrolyzing it with 72% H₂SO₄. The data presented in Fig. 1c show that the total yield of glucose in the liquid fraction obtained after wood pretreatment did not differ significantly from each other and was 31.6 mg/g (160°C) and 29.3 mg/g (205°C) respectively. On the other hand, the total xylose yield increased almost seven times when wood was pretreated at 205°C with respect to its total yield (16.0 mg/g) in the liquid fraction obtained at 160°C. The data obtained are consistent with the results of Carvalho et al. (2008).

The data of Marchwicka et al. (2015) showed that the glucose content of 3-year-old *Populus deltoides x maximowiczii* subjected to enzymatic hydrolysis at 24 h, 72 h, 150 h, 216 h and 360 h without pretreatment increased gradually from 11.1–14.6%. On the other hand, if the wood was pretreated followed by enzymatic hydrolysis using the above-mentioned times, the glucose content also increased gradually and was from 15.6–24.1%. The increase in glucose content in wood subjected to steam explosion followed by enzymatic hydrolysis ranged from 28–40% compared to the control wood. From the data of Antczak et al. (2014), the glucose content of the wood of *Populus deltoides x maximowiczii* after extraction and acid hydrolysis was 40.9%. From the above data, it can be concluded that the glucose yield from wood depends on the conditions and processes to which it is subjected.

The xylose content after treatment of *Populus deltoides x maximowiczii* wood is related to its xylan content, although it also occurs as a component of e.g., glucuronoxylan. Hemicelluloses under steam explosion treatment at 205°C are degraded and transformed to liquid and gaseous products.

Analyzing the results presented in Figs. 1b and 1c it can be seen that the SE pretreatment produces oligosaccharides which are not detectable under the applied conditions by HPLC analysis without additional acid hydrolysis. The total glucose yield after acid hydrolysis in the liquid fraction obtained at 160°C was almost eight times higher and at 205°C about twice higher than the yield of this sugar in the liquid fraction without additional acid hydrolysis. On the other hand, the total yield of xylose in the post-reaction liquid obtained at 160°C after acid hydrolysis was over fourteen times higher and at 205°C was over five times higher than the yield of this sugar in the liquid fraction without acid hydrolysis. Comparing the result shown in Figs. 1a, 1b and 1c, it can be observed that hemicelluloses are less heat resistant than cellulose (Nethercot 2005; Gawron et al. 2014; Antczak et al. 2021).

One of the methods allowing tracing the changes occurring in the wood of *Populus deltoides x maximowiczii* before and after steam explosion at 160°C and 205°C is the analysis of FTIR-ATR (Figs. 2 and 3).

During the thermal treatment of wood, several processes take place simultaneously with different effects on the intensity of the absorption band with a maximum in the wavenumber about 1736 cm⁻¹. All components of the wood contribute to the change in the intensity of this band, but in most cases lignin and hemicellulose are the most important.

Based on the data of changes in the intensity of the absorbance band at the wavenumber 1736 cm⁻¹ it can be concluded that in the wood subjected to steam explosion, there was a decrease in the intensity of the absorbance band by 3.6% (160°C) and 60% (205°C) (Fig. 3.) compared to the wood not subjected to this process, which indicates that at 205°C xylan degrades more and goes into solution. On the other hand, the data of Výbohová et al. (2018) show that during the process of the heat treatment of ash wood there was an increase in the absorbance of this peak.

For aromatic skeletal vibrations plus C = O stretch; 1507 cm⁻¹ for aromatic skeletal C = C vibrations and amounts to 160°C and 205°C, respectively, to 7% and approx. 30% regardless of the process temperature used. The action on ash wood temperature 160°C, 180°C and 200°C increased the intensity of the peak 1593 cm⁻¹, which may be due to the relative increase of lignin content in treated wood (Chen et al. 2012; Výbohová et al. 2018).

From the above data, it is evident that in *Populus deltoides x maximowiczii* wood during high temperature pretreatment, lignin plasticizes and condenses with cellulose and hemicelluloses according to Fengel and Wegener (2003), Hill (2006), Nuopponen et al. (2004), Sun et al. (2016) and Antczak et al. (2018).

The decrease in the intensity of the absorption band 1462 cm⁻¹, for aromatic C-H deformation and asymmetric bending of groups -CH₃ and -CH₂- saccharides at process temperatures of 160°C and 205°C was about 60% and 20%, respectively, with respect to native wood, indicating transformation of lignin and polysaccharides.

At the wavenumber of 1235 cm⁻¹ for syringyl ring and C-O stretching in lignin and xylan the reduction in intensity of this band in relation to native wood, respectively to the applied temperature of 160°C and 205°C in the wood of *Populus deltoides x maximowiczii* subjected to the steam explosion was 7% and 53% (Fig. 3).

Based on the above data, it can be concluded that xylan was degraded and passed into solution after pretreatment of wood at 205°C, while structural changes occurred in lignin. With increasing temperature, degradation and hydrolysis as well as dissolution of hemicelluloses occur, most of which goes into solution, and at the same time some of them are transformed into gaseous products (Jönsson and Martín 2016; Brethauer et al. 2020; Antczak et al. 2021).

Changes occurring in the cellulose isolated from *Populus deltoides x maximowiczii* wood by Kürschner-Hoffer method, subjected to a steam explosion at 160°C and 205°C, were investigated by analyzing the absorption bands of FTIR-ATR spectra. The cellulose crystallinity index was calculated from absorption bands TCI $A_{1372} \text{ cm}^{-1} / A_{2900} \text{ cm}^{-1}$ and LOI $A_{1423} \text{ cm}^{-1} / A_{896} \text{ cm}^{-1}$ (Figs. 4a and 4b). Band 2900 cm^{-1} corresponds to stretching vibrations of -CH in -CH₂- and -CH₃ groups, while 1372 cm^{-1} to deformation vibration of C-H in cellulose (Poletto et al. 2012; Geffert et al. 2017). An empirical crystallinity index also known as lateral order index (LOI) was defined as ratio between intensity of the bands at 1422 cm^{-1} (C-H bending vibrations) and at 897 cm^{-1} (β -(1-4)-glycosidic bond and C-H deformation vibrations in cellulose) (Nelson and O'Connor 1964; Kumar et al. 2014).

The TCI is proportional to the degree of crystallinity of cellulose in wood and the LOI is correlated to the overall degree of order in the cellulose (O'Connor et al. 1958; Carrilo et al. 2004; Kumar et al. 2014).

On the basis of the data presented in Fig. 4b, it can be concluded that the total crystalline index of cellulose isolated from wood by the Kürschner-Hoffer method, before and after steam explosion pretreatment at 160°C and 205°C, decreased by 6% and 5.3% respectively. On the other hand, the data of Geffert et al. (2017) showed that the total crystalline index investigated in beech wood increased by 13.2% compared to native wood. This is probably due to the partial degradation of hemicelluloses and amorphous cellulose regions by the influence of high temperature.

Yildiz and Gümüşkaya (2007) found that cellulose isolated from spruce and beech wood samples treated by thermal modification at 150°C, 180°C and 200°C by 6 h and 10 h was characterized by an increase of TCI and LOI. As a results of these modifications, it was concluded that hemicelluloses and less ordered cellulose deteriorated and as a consequence the degree of cellulose crystallinity increased.

According to the literature and the analyzed data, the impact of a steam explosion on wood at 160°C and 205°C resulted in the degradation of hemicelluloses, cellulose with a lower degree of polymerization, and the degradation and change in the structure of lignin. As a result, the observed change in the total crystallinity index was low.

Figure 4a shows data as a LOI $A_{1422} \text{ cm}^{-1} / A_{896} \text{ cm}^{-1}$ of Kürschner-Hoffer cellulose, from which it can be concluded that cellulose isolated from *Populus deltoides x maximowiczii* wood subjected to steam explosion at 205°C was characterized by a 16.1% increase in LOI with respect to the cellulose index from native wood. On the other hand, the cellulose isolated from wood subjected to steam explosion at 160°C characterized by a 0.6% increase in LOI with respect to the cellulose isolated from native wood. Based on the results, it can be concluded that the steam explosion pretreatment at 205°C of *Populus deltoides x maximowiczii* wood affected the increase of crystalline regions in cellulose isolated by the Kürschner-Hoffer method.

According to Ausenfans et al. (2017), a native poplar wood exhibited an LOI of 1.77 ± 0.4 and decreased by about 18% after steam explosion of the biomass. From the data presented in Figs. 4a and 4b, it can be

concluded that the changes in cellulose crystallinity index TCI and LOI in *Populus deltoides x maximowiczii* wood subjected to steam explosion depended on the applied temperature of the biomass pretreatment process.

One of the important characteristics of cellulose is its degree of polymerization. The cellulose with degree of polymerization above 200 (i.e., α -cellulose) is characterized by higher resistance to acids, alkalis in comparison to β -cellulose (degree of polymerization around 200) and γ -cellulose (degree of polymerization below 100). Figure 5a shows the polydispersity index data of K rschner-Hoffer cellulose isolated from *Populus deltoides x maximowiczii* wood before and after steam explosion at 160 C and 205 C.

Based on the data presented, it can be concluded that after taking into account the standard deviation, the cellulose isolated from pretreated wood had a similar polydispersity index with respect to the cellulose isolated from native wood. The data obtained are consistent with the results relating to slight changes in the degree of polymerization of cellulose (Alvira et al. 2010; Tom s-Pejo et al. 2011; Sun et al. 2016).

In summary, it can be stated that for cellulose isolated from wood before and after pretreatment by steam explosion at 160 C and 205 C by the K rschner-Hoffer method, a slight decrease in TCI was observed for cellulose isolated from wood after pretreatment, while for LOI it increased with increasing treatment temperature. In the case of polydispersity index and degree of cellulose polymerization, a decrease in these parameters was observed for pretreated cellulose relative to the native material. The cellulose isolated from wood after pretreatment at 205 C had higher polymerization and polydispersity index than the cellulose obtained from wood after treatment at 160 C.

Conclusion

Based on the data presented, it can be concluded that:

1. Cellulose was resistant to 160 C and 205 C applied for steam explosion of *Populus deltoides x maximowiczii* wood.
2. Pentosans (xylan) were not resistant to the temperature of 160 C and 205 C used in the steam explosion of wood, hence the xylose content in the liquid fraction was three times higher than the glucose content.
3. TCI ($A_{1372} \text{ cm}^{-1} / A_{1900} \text{ cm}^{-1}$) of K rschner-Hoffer cellulose isolated from *Populus deltoides x maximowiczii* wood subjected to steam explosion at 160 C and 205 C decreased by 5.6% and 5%, respectively, to the applied temperature relative to the crystallinity index of K rschner-Hoffer cellulose isolated from native wood.
4. LOI ($A_{1429} \text{ cm}^{-1} / A_{896} \text{ cm}^{-1}$) of K rschner-Hoffer cellulose isolated from *Populus deltoides x maximowiczii* wood subjected to steam explosion at 205 C increased by 19% with respect to the index of cellulose isolated from native wood, whereas K rschner-Hoffer cellulose isolated from wood

subjected to steam explosion at 160°C did not differ highly in index (LOI) from cellulose isolated from native wood.

5. Kürschner-Hoffer cellulose isolated from *Populus deltoides x maximowiczii* wood subjected to steam explosion at 160°C and 205°C had a reduced degree of polymerization of about 11% and about 8%, respectively, according to the temperature used.
6. Kürschner-Hoffer cellulose isolated from *Populus deltoides x maximowiczii* wood subjected to steam explosion at 160°C and 205°C had a similar polydispersity index with respect to cellulose isolated from native wood.

Declarations

Acknowledgements This work was financed by a research project from the National Center for Research and Development, which was “Intelligent systems for breeding and cultivation of wheat, maize, and poplar for optimized biomass production, biofuels, and modified wood” (BIOSTRATEG2/298241/10/NCBR/2016). Poplar material used in presented work was obtained in Welcome 2008/1 project of the

Foundation for Polish Science given to Prof. Stanisław Karpiński.

Author contributions The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript.

Conflict of interest The article does not contain any conflict of interest

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Figures

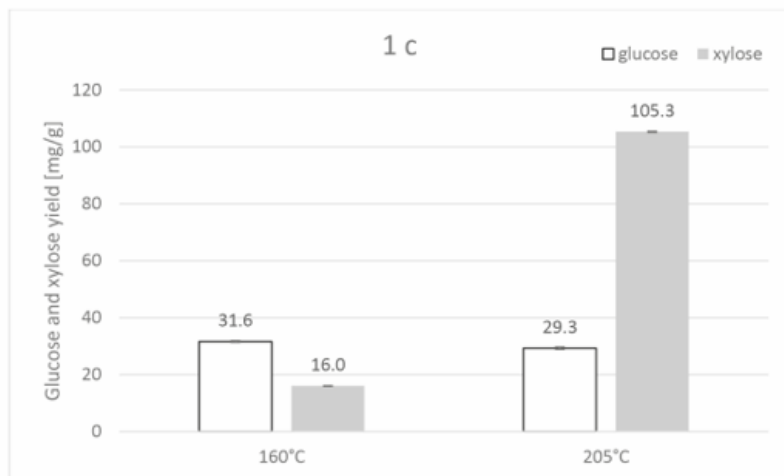
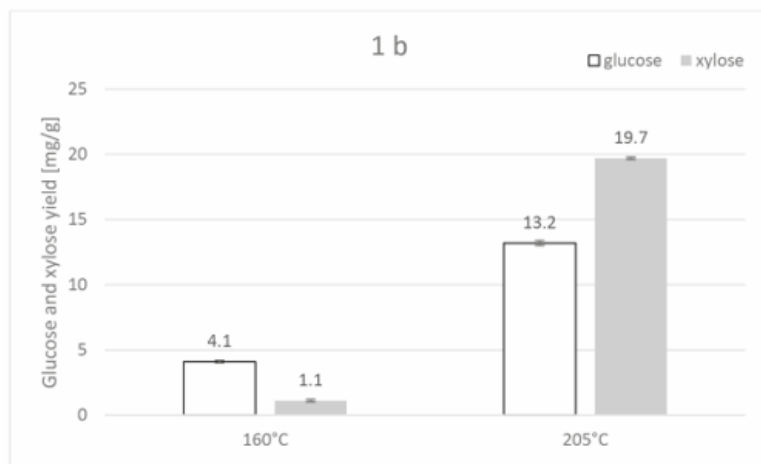
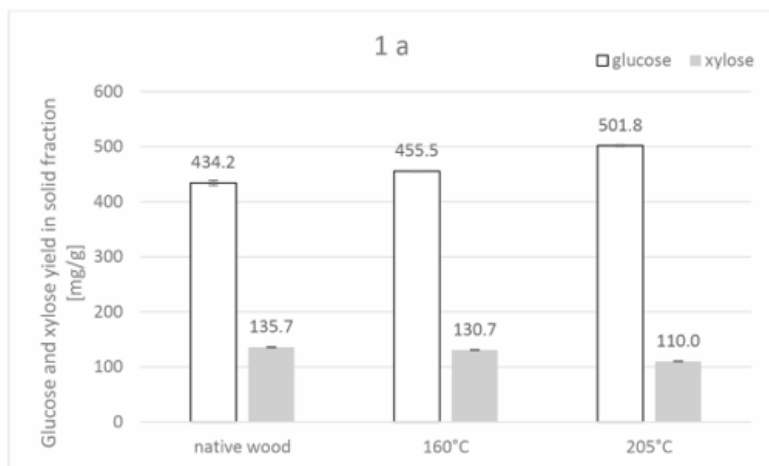


Figure 1

- Glucose and xylose content of the solid fraction,
- Glucose and xylose yield in the postreaction liquid,
- Glucose and xylose content after pretreatment and acid hydrolysis

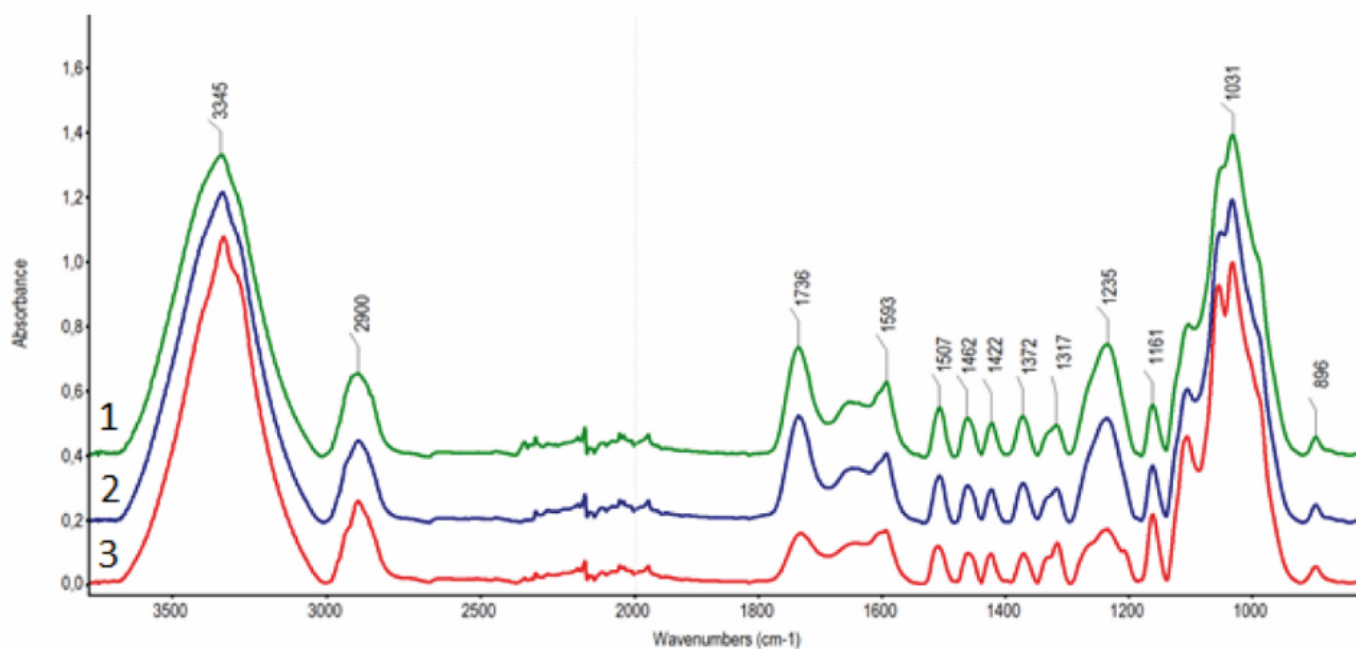


Figure 2

1- native wood, 2 - wood extracted from wood after treatment at 160 °C and 3 -wood extracted from wood after treatment at 205 °C.

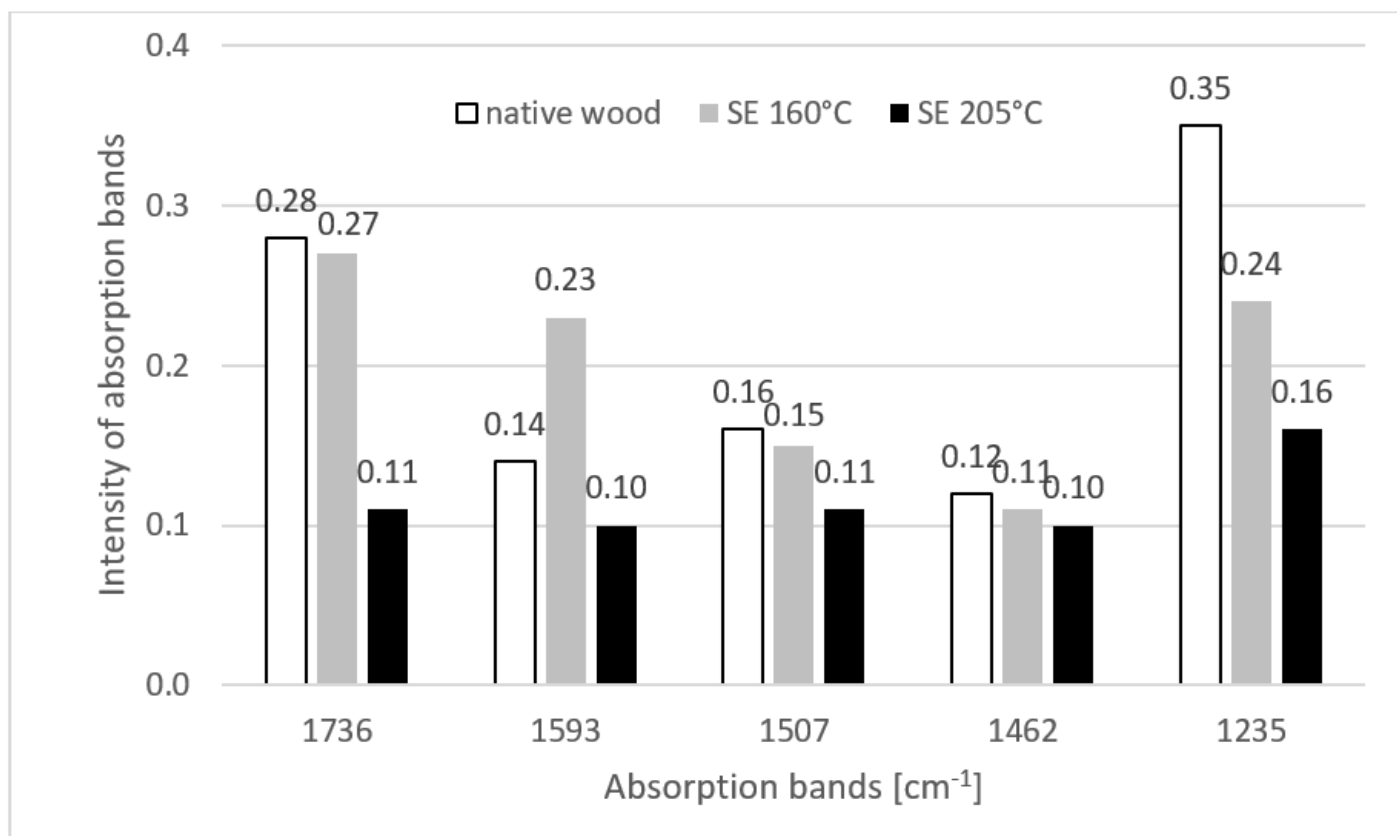


Figure 3

Relative intensity of selected absorption bands in FTIR spectra of wood samples

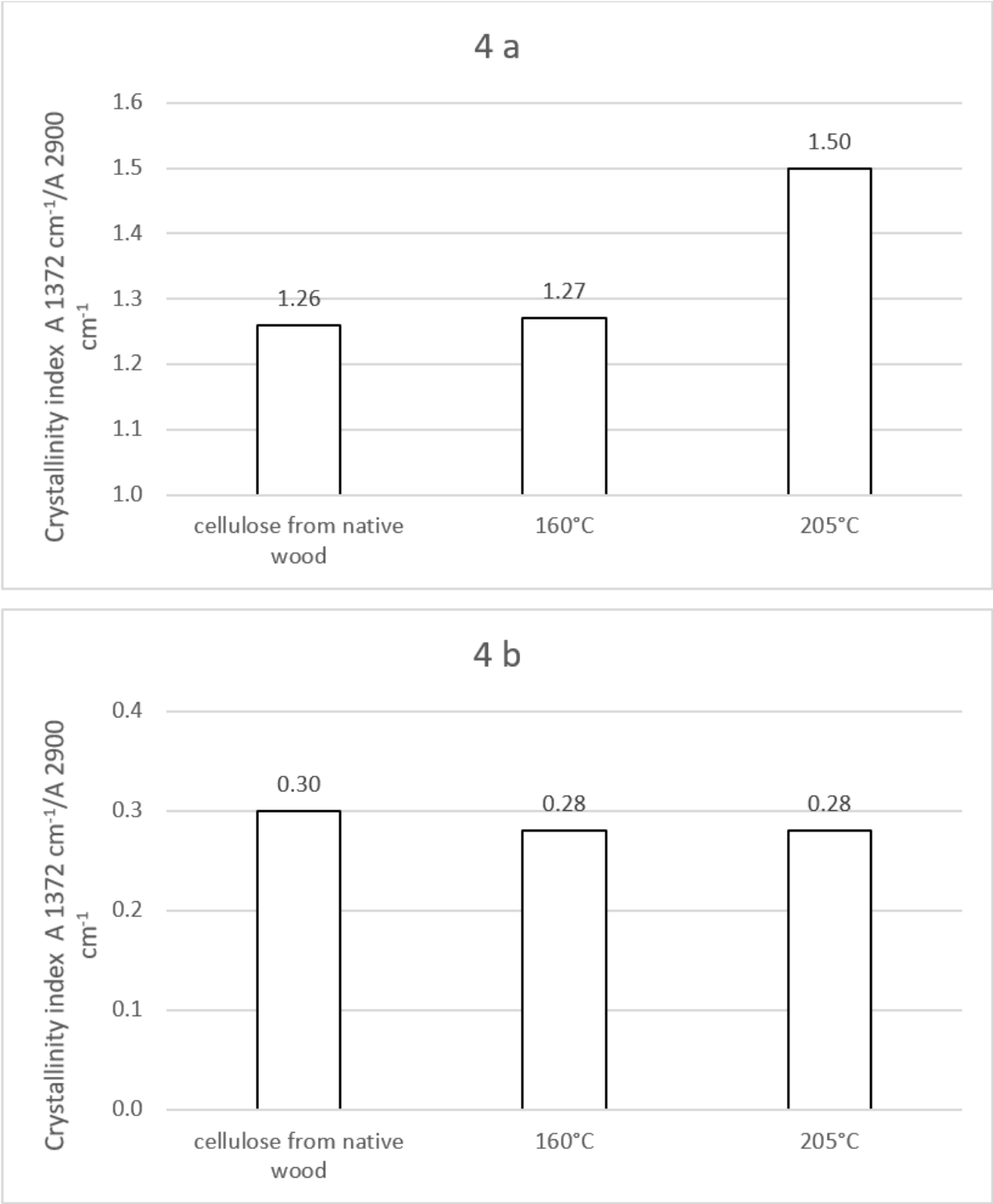


Figure 4

a. Lateral Order Index (LOI) A1422 cm⁻¹/A896 cm⁻¹,

b. Total Crystallinity Index (TCI) A_{1372} / A_{2900}

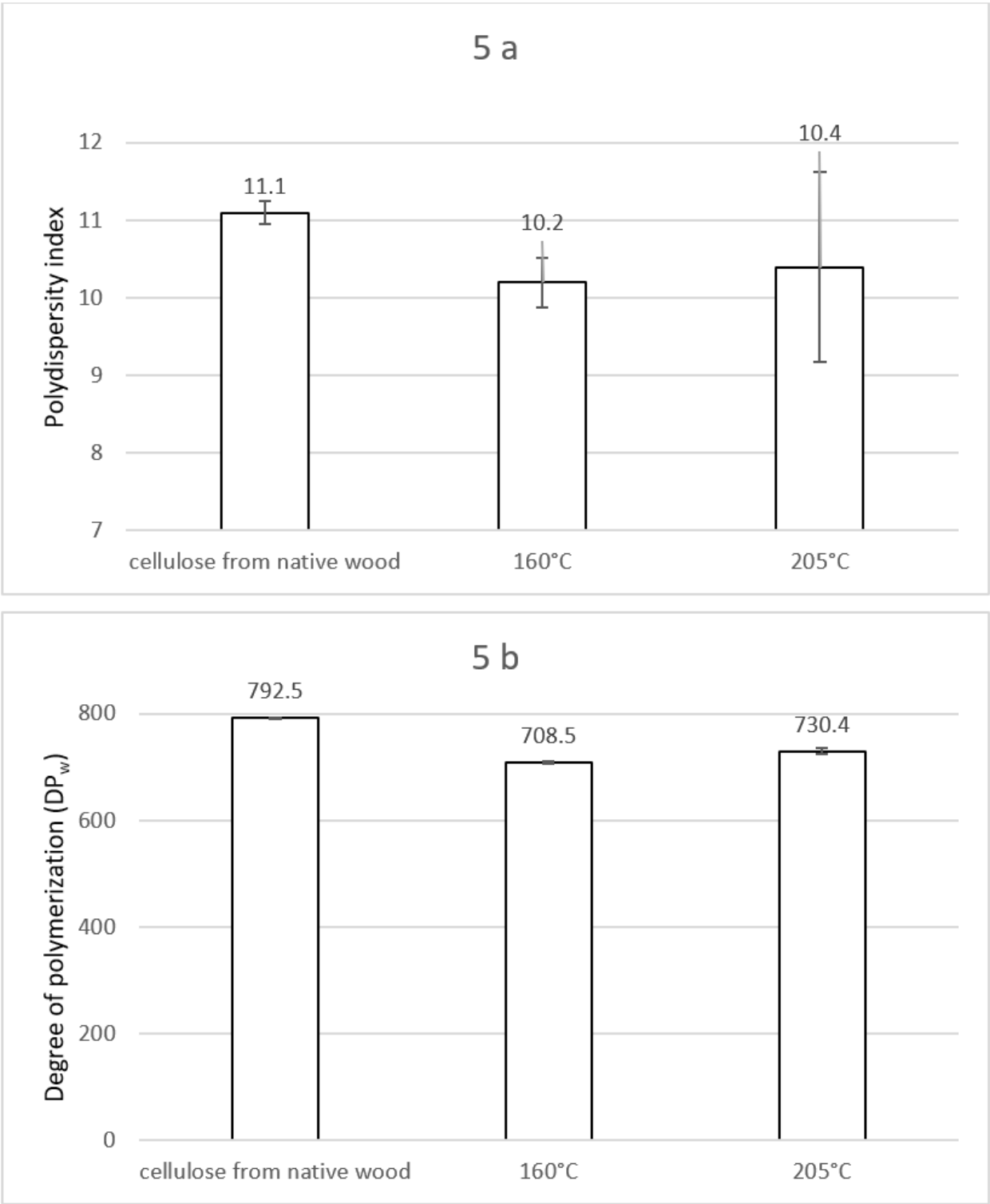


Figure 5

a. Polydispersity index of cellulose,

b. Degree of polymerization of cellulose