### Potential of Jerusalem Artichoke Stem for Cellulose Production

A.N. Prusov<sup>1</sup>, S.M. Prusova<sup>1</sup>, A.G. Zakharov<sup>1</sup>, A.V. Bazanov<sup>1\*</sup>, V.K. Ivanov<sup>2</sup>

<sup>1</sup>Krestov Institute of Solution Chemistry of the Russian Academy of Sciences, Academicheskaya, 1,

Ivanovo, 153045, Russia

<sup>2</sup>Kurnakov Institute of General and Inorganic Chemistry of the Russian Academy of Sciences,

Leninsky Prosp., 31, Moscow, 119991, Russia

Article info	Abstract
Received:	There is a potential opportunity to convert almost any type of biomass into
30 November 2018	biofuel and bio- nanomaterials, if the appropriate biotechnological and chemical processing methods are used. The preference for this or that bioresource is due
<i>Received in revised form:</i> 15 January 2019	to the stability of the raw material base and the prospect of its use. Jerusalem artichoke stem ( <i>Helianthus tuberosus</i> L.) (JA) is widely known as a potential non-
<i>Accepted:</i> 16 February 2019	food raw material for biofuels due to high biomass extraction (36–49 t/ha (tons per hectare)) and limited cultivation requirements. But little attention is given to study the possibility of using the stems to produce various kinds of cellulose. This article
<i>Keywords</i> Jerusalem artichoke ( <i>Helianthus tuberosus L.</i> ), Cellulose, Chemical properties, Chemical processing, Biofuel.	presents samples of cellulose that were obtained from the Jerusalem artichoke stem using mechanical and chemical methods. Cellulose yield from the stem was: cortex 51.1%, pith 65.2% with the $\alpha$ -cellulose content 96–98%. Methods of electron microscopy, atomic absorption, IR spectroscopy, X-ray diffraction, BET for nitrogen adsorption, thermogravimetry were used to study the cortex and the pith of the Jerusalem artichoke stem. Analysis of the cellulose samples confirmed the possibility of obtaining high-quality cellulose.

### 1. Introduction

Lignocellulose biomass of annual plants is a potential source not only for biofuels [1, 2], catalyst carriers or adsorbents [3], precursors for activated carbons [4], hybrid nanocomposites [5, 6], but also for the production of cellulose and its derivatives [7, 8]. In this connection, the attention of many researchers is drawn to Jerusalem artichoke. Successful cultivation of this crop is possible from the tropics to the northern agricultural regions on various soils, except strongly acidic. It produces a high yield of biomass in any year even unfavorable for climatic conditions.

Jerusalem artichoke is valuable for the high content of sugars in green mass and tubers, among which inulin, known to treat diabetes [9], is most appreciated. However, only tubers are fully processed industrially. The leafy stem vegetative part of Jerusalem artichoke has no industrial application, although it is well known as a promising raw material for the production of biofuels [10, 11], as well as natural fibers for the reinforcement of composite materials, instead of synthetic ones [12].

The purpose of this paper is to study the macrostructure, microstructure, chemical composition, changes in the composition and physical properties of the Jerusalem artichoke stem as a result of mechanical and chemical treatments. Analyses of the Jerusalem artichoke potential as bioresource for cellulose production.

### 2. Materials and methods

#### 2.1. Jerusalem artichoke stem

In this study, we used the stems of Jerusalem artichoke (*Helianthus tuberosus* L.), grown in Russia. The duration of plant growth was 130–150 days. After drying in air, some Jerusalem artichoke cortex samples (JA1) were crushed in a centrifugal mill to a particle size of ~0.5 mm (JA2), others in a twin-screw extruder to particles of less than ~0.42 mm (JA3), and washed with water to remove dust and other contaminants. Then the samples

© 2019 Eurasian Chemico-Technological Journal.

<sup>\*</sup>Corresponding author. E-mail: aisetnt@mail.ru

This is an open access article under the CC BY license (http://creativecommons.org/licenses/by/4.0/).

were dried in an oven at 50 °C for 48 h. The pith extracted from the stem (JA4) was dried at 40 °C for 48 h.

The content of trace elements in the samples was determined using the MGA-915 atomic absorption spectrometer. Ash content was determined by the method described in paper [13].

### 2.2. The microstructure and morphology

Jerusalem artichoke stem samples morphology was studied using a scanning electron microscope Carl Zeiss NVision 40 (SEM) at an accelerating voltage of 1 kV using a secondary electron detector (SE2).

The amorphous-crystalline state of Jerusalem artichoke stem and cellulose was studied by X-ray diffraction using a D8 Advance X-ray diffractometer (CuK<sub>a</sub> radiation). The crystallinity index (CrI) of the cellulose was calculated using generally adopted equation [1]:

$$CrI = (I_{200} - I_{am}/I_{200}) \cdot 100\%$$

where  $I_{200}$  is the diffraction intensity at  $2\theta = 22.5^{\circ}$ ,  $I_{am}$  is the diffraction intensity at  $2\theta = 18^{\circ}$ , corresponding to the amorphous part of the sample [14]. The mean square error of the three CrI measurements was  $\pm 0.15$ .

The spectra of the samples in the form of tablets were obtained using a VERTEX 80v IR-Fourier spectrometer with a resolution of 0.2 cm<sup>-1</sup> in the range 4000–400 cm<sup>-1</sup>. To prepare the tablets, the pre-crushed cellulose-containing materials were thoroughly grinded with potassium bromide (KBr) and tableted. The weight of the tablets, their thickness and the content of the analyte are constant.

### 2.3. Degree of polymerization

The degree of polymerization (DP) of cellulose was determined by a method based on the determination of the cellulose solutions viscosity. Using the following equation:  $DP^{0.905} = 0.75[\eta]$  [15].

All experiments were performed using an Ubbelohde viscometer at  $25\pm0.5$  °C. As a solvent for cellulose the copper (II) ethylene diamine hydroxide was used. The intrinsic viscosity was calculated by interpolation using the USP Table [16], which shows the intrinsic viscosity and concentration ([ $\eta$ ], C) values for cellulose solutions with relative viscosity ( $\eta_{re}$ ) between 1.1 and 9.9. Viscosity ( $\eta_{re}$ ) was calculated using the ratio:  $\eta_{re} = t/t_0$ , where t and  $t_0$  are the outflow times for the cellulose solution and the solvent, respectively.

### 2.4. Thermogravimetric analysis

The process of thermal destruction of modified cellulose samples was studied by the thermogravimetric method. Thermogravimetric studies were performed on a differential scanning heat flux calorimeter DSC 204 F1 Phoenix and a TG 209 F1 Iris thermal analyzer. The heat treatment of the samples was carried out in a nitrogen flow of 10 ml/min in the temperature range 25–950 °C at a heating rate of 10 °C/min. Data processing was carried out using the Thermokinetics Professional SW/KIN software.

## 2.5. Characteristics of the porous structure of the samples

The porous structure characteristics of the samples (the specific surface area  $S_{BET}$ , the total pore volume  $V_{por}$  and the average pore diameter) were determined on a Quantchrome Nova Series 1200e device using the low temperature nitrogen adsorption at 77 K.

Specific (apparent) density is calculated as the ratio of the mass of a substance to its volume, including pores. The mass of the stem core was weighed to an accuracy of 0.0001 g, the mass volume was determined using a pycnometer filled with mercury.

# 2.6. Isolation of cellulose from the cortex and the pith of Jerusalem artichoke

JA2, JA3 and JA4 samples (5 g) were each supplemented with 50 ml of a 2% (w/w) solution of NaOH and mixed for 4 h (30 °C) with subsequent centrifugation. The samples were then treated with a 50 ml alkaline solution of  $H_2O_2$  containing 1% (w/w) NaOH and 3% (v/v)  $H_2O_2$ , respectively, for 2 h (50 °C). Then samples were washed with distilled water to  $pH \approx 7$  and centrifuged. The resulting samples were treated with an aqueous solution of HNO<sub>3</sub> (50 ml) at a concentration of 0.05 (mol/L) with stirring for 1 h (70 °C). Finally, the samples were washed with distilled water to  $pH \approx 7$  and centrifuged at 5000 rpm for 10 min, then dried at 80 °C. As a result of the chemical treatment of samples JA2, JA3 and JA4, pure cellulose JA5, JA6 and JA7 samples were obtained, respectively.

A batch of cortex (JA1, JA2) and pith (JA4) was treated with 18% sodium hydroxide (NaOH) solution for 2 h (20 °C), washed until neutral, centrifuged and dried (60 °C). Thus, samples JA8, JA9 and JA10 were obtained, respectively.

A 5 g batch of cortex (JA1) was treated with an 18% NaOH solution, washed with water and treated for 2 h (50 °C) with a 50 ml alkaline solution of  $H_2O_2$  containing 1% (w/w) NaOH and 3% (v/v)  $H_2O_2$ , respectively. After washing, the sample was separated into fibers and dried (60 °C). Thus, JA11 sample was obtained. Thus, JA11 sample was obtained.

### 3. Results and discussion

### 3.1. The microstructure and morphology

Figure 1 shows SEM image of the longitudinal section of the dry stem, the stem is formed by several layers from various tissues. The outer part of the stem is covered with epidermis, which protects the plant from adverse external factors the next thin layer of tissue, which serves for gas exchange with the atmosphere and evaporation of water. Beneath these layers there is a solid layer of cortex and pith, the main component of which is cellulose. This layer also contains hemicellulose (a mixture of low molecular polysaccharides with monosaccharides), lignin, pectin form connective tissues (Fig. 1, Table 1). The cortex is a close-fitting cellulose fiber covered with ligneous connective tissue (Fig. 1). The pith of the stem is a thin-walled tissue, which also consists mainly of cellulose I.

In Table 1 the results of the analysis of the chemical composition and specific surface area of the cortex and core of the stem of Jerusalem artichoke. The cellulose content in the core (67.7%) is higher than in the cortex (54.1%). Note that JA4 contains much less lignin, pectin and ash than JA1.

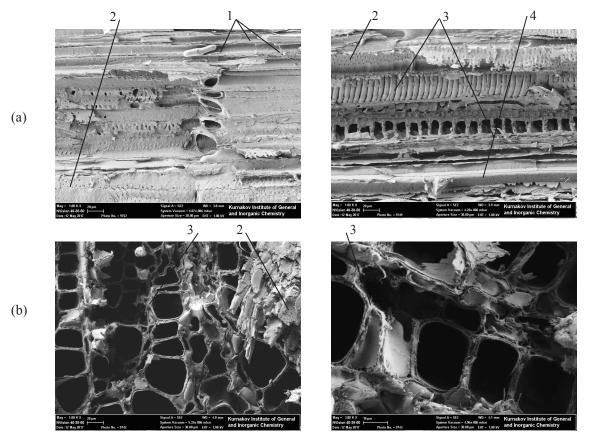


Fig. 1. SEM image of the longitudinal section of the bark of the stem (a) and transverse (b): 1 - fibers (sclerenchyma cells); 2 - phloem; 3 - xylem; 4 - pith.

Table 1						
The chemical composition of the absolutely dry Jerusalem artichoke stems in mass percentages,						
specific surface area $(m^2/g)$						

Jerusalem artichoke stem	α-cellulose	hemicellulose	lignin	Ash	Specific surface area
JA1	54.1±0.9	16.3±0.4	12.5±0.6	$1.8 \pm 0.06$	6.5
JA4	67.7±1.0	4.6±0.3	7.6±0.6	1.3±0.06	37.6

Eurasian Chemico-Technological Journal 21 (2019) 173-182

SEM images of the stem pith (JA4) are shown in Fig. 2. The cellulose structure of the pith resembles a honeycomb with closed cells. The transverse section of the pith of the Jerusalem artichoke stem is a closely fitting polygon (Fig. 2). The walls of the cells are multilayered (Fig. 2). The transverse dimensions of the cells are 40–120  $\mu$ m (Fig. 2) and the longitudinal dimensions of the cells are about 200 µm (Fig. 2). Such a structure of the Jerusalem artichoke pith conditions the specific density (apparent) (0.03 g/cm<sup>3</sup>) and the high specific surface area of the JA4 sample. The average pore diameter of the pith of Jerusalem artichoke is about 4.8 nm, and the total pore volume is 0.045  $\text{cm}^3/\text{g}$  for the pores which diameter of less than 214 nm. The specific surface area of the sample JA4 is 37.6 m<sup>2</sup>/g (Table 1). The cortex of Jerusalem artichoke sample (JA1) has a smaller specific surface area (6.5  $m^2/g$ ) (Table 1). Accordingly, the average pore diameter of the cortex is 11 nm, the total pore volume being 0.02 cm<sup>3</sup>/g for the pores which diameter is less then 178 nm.

The pith of Jerusalem artichoke has the necessary indicators of heavy metal biosorbents (porous structure, high specific surface, presence of hydroxyl groups). The use of artichoke stem powder to remove heavy metals (Zn, Pb and Cd) is known [17].

 Table 2

 Microelement composition Jerusalem artichoke stem, mg/kg (dry)

Jerusalem artichoke stem	Со	Са	Ni	Fe	Cu	Mn
JA1	0.056	13	3	245	5	16
JA4	0.43	31	5	261	10	36

The stem of Jerusalem artichoke contains more metals in the pith and less in the cortex (Table 2).

Aqueous solution of NaOH effectively removes from the stem of Jerusalem artichoke hemicellulose, lignin, pectin, trace elements soluble in aqueous alkaline solutions, namely, potassium, sodium, etc. However, most of the trace elements form insoluble metal hydroxides. When drying the stem, metal hydroxides lose water and form oxides, for example, oxides of iron, cobalt, Nickel, silicon, etc. in Fig. 3 these mineral accumulations are visible. It is known that the treatment of cellulose in 18% aqueous NaOH occurs swelling of the crystalline regions of the polymer. On Figs. 2 and 3 it is clearly seen that the walls of the core cell are formed by closely adjacent lamellar cellulose layers.

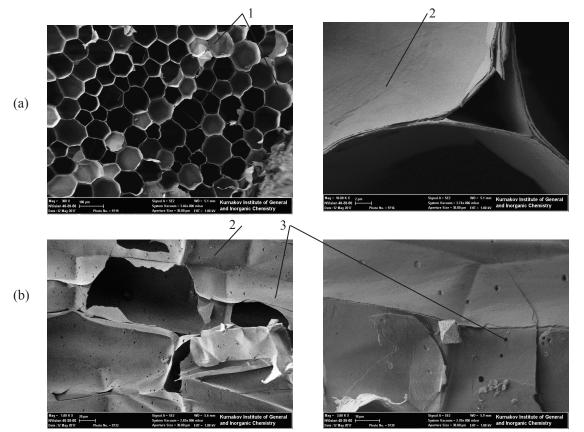


Fig. 2. SAM image of topinambur stem cut (JA4): (a) – longitudinal, (b) – transverse. 1 - cell; 2 - cellulose tissue of the cell wall; 3 - the pores of the cells wall.

Eurasian Chemico-Technological Journal 21 (2019) 173-182

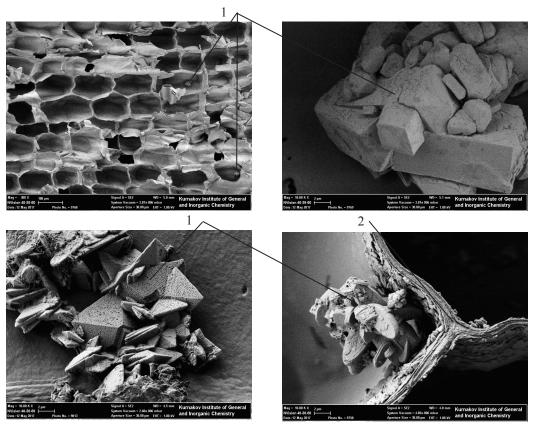


Fig. 3. SEM image of the Jerusalem artichoke stems pith cut treated with 18% NaOH (JA10) solution: (a) – longitudinal; (b) – transverse. 1 – mineral substances; 2 – swollen cellulose tissue cells wall.

The morphological and structural changes that occurred during the chemical processing of the Jerusalem artichoke stem are important for the further processing of cellulosic precursors, for example, microcrystalline cellulose [18], biofuel [15] etc. To obtain biofuels, the process of saccharification of biomass is carried out, i.e. the production of hexose, for example, by enzymatic hydrolysis [15]. The intensity of the process of biomass hydrolysis is determined by the availability of reagents in the cellulose matrix. The amorphous part of cellulose is most accessible for chemical reagents. Thus, the intensity of the biomass processing process depends on the CrI of cellulose, which characterizes the ratio of crystalline and amorphous regions in the polymer.

The obtained X-ray diffraction patterns of Jerusalem artichoke samples are typical for the I<sub> $\beta$ </sub> modification of cellulose, with characteristic diffraction maxima at 2 $\theta$  = 15.0, 16.6 and 22.5°, which can be assigned to 1 $\overline{10}$ , 110 and 200 reflections [15].

As can be seen from Table 3, the lowest CrI index is HT1 (44%) and the highest is HT7 (56%). Consequently, the treatment of HT1 and HT4 sample with chemical solutions increases the CrI of cortex and core cellulose and adversely affects the efficiency of biomass hydrolysis.

Samples of Jerusalem artichoke cellulose obtained by chemical method are presented on Fig. 4: JA7-stem core cellulose; JA5 and JA6 – cortex cellulose crushed in a centrifugal mill and in a twinscrew extruder respectively; JA11 – cortex fibrous.

Jerusalem artichoke stem	CrI	$D_{3400}/D_{1320}$	D <sub>1372</sub> /D <sub>2900</sub> (TCI)	D <sub>1429</sub> /D <sub>897</sub> (LOI)
JA2	44.1	1.46	0.94	2.52
JA4	56.3	1.49	1.23	2.84
JA5	50.3	1.80	1.02	2.83
JA7	63.8	1.51	1.27	2.98

 Table 3

 Infrared crystallinity ratio and hydrogen bond intensity of the studied samples



Fig. 4. SEM image Jerusalem artichoke cellulose: (a) - sample JA7; (b) - sample JA5; (c) - sample JA6; (d) - sample JA11.

The main indicators of cellulose quality are the content of  $\alpha$ -cellulose and hemicellulose, degree of polymerization and ash content. We additionally processed the Jerusalem artichoke stems with HNO<sub>3</sub> solution and deeply removed lignin and trace elements from the cellulose.

According to the works [18, 19], reduction of lignin and DP, ash content and increase of  $\alpha$ -cellulose content is a positive factor in the process of enzymatic biomass splitting.

As follows from Table 4, all samples of cellulose have a high content of  $\alpha$ -cellulose and small ash content. Lignin is absent in the sample JA6 and the sample JA7 contains a negligible amount of it. The lignin content in the samples JA5, JA9 and JA10 is cortex and pith within the range of 1.5–2.7%.

It should be expected that cellulose with DP = 800–1400 and ash content less than 0.3%, lignin less than 0.6%,  $\alpha$ -cellulose 96–98%, specific surface 6.5 and 158.2 m<sup>2</sup>/g (Table 4) will be in demand for catalytic conversion to ethylene glycol [19], enzymatic hydrolysis [20], for the production of cellulose ethers, for example nitrocellulose, as well as microcrystalline and nanocellulose [21].

It should be noted that cellulose of the highest quality is obtained from the pith and the cortex of Jerusalem artichoke stem which was pretreated in a twin-screw extruder.

### 3.2. Fourier transform infrared spectroscopy (FTIR) of the cortex and pith of Jerusalem artichoke stem before and after chemical treatments

The Fig. 5 shows the FTIR spectra of samples JA2, JA4, JA9, JA10. The peak at 3342 cm<sup>-1</sup> can be caused by the O-H stretching vibration and hydrogen bond of the hydroxyl groups [22]. The peak at 2900 cm<sup>-1</sup> is due to stretching vibration of CH and CH<sub>2</sub> groups in cellulose and hemicellulose components. The peak at 1739 cm<sup>-1</sup>, which is more intense for the JA2 sample, can be attributed to the stretching vibrations of the carbonyl group of lignin or the ester group in hemicellulose. The peak at 1650 cm<sup>-1</sup> and 1597 cm<sup>-1</sup> occurs when there is water in the samples. A little peak at 1506 cm<sup>-1</sup> is attributed to C=C stretching of aromatic ring of the lignin. The absorbance at 1429 cm<sup>-1</sup> is associated to the CH symmetric bending present in cellulose. The two peaks observed at 1374 cm<sup>-1</sup> and 1318 cm<sup>-1</sup> in the spectrum indicate the bending vibration of C-H and C-O groups of the aromatic ring in polysaccharides. The absorbance peak centered at 1245 cm<sup>-1</sup> is due to the C-O stretching vibration of the acetyl group in lignin [23].

Two peaks at 1108 cm<sup>-1</sup> and 1037 cm<sup>-1</sup> can be associated with stretching vibrations of O–H and C–O cellulose groups [24]. A small peak at 897 cm<sup>-1</sup>

Name	JA5	JA6	JA7	JA9	JA10
α-cellulose	93.2±0.7	96±0.5	97.8±0.6	91.3±0.9	92.5±0.8
lignin	1.5±0.1	no	0.6±0.1	2.7±0.1	$1.8 \pm 0.1$
hemicellulose	no	no	no	$0.80{\pm}0.1$	0.6±0.1
DP	1123	825	1352	1950	-
ash content	< 0.5	< 0.3	< 0.1	< 1	-
Specific surface area, m <sup>2</sup> /g	-	158.2	15.3	-	-

 Table 4

 Cellulose characteristics Jerusalem artichoke stem

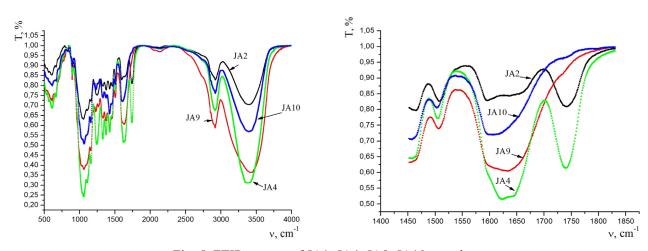


Fig. 5. FTIR spectra of JA1, JA4, JA9, JA10 samples.

is explained by the presence of  $\alpha$ -glycosidic bonds between monosaccharides [25]. The peak at 598 cm<sup>-1</sup> corresponds to the deformation vibrations of C-OH, characteristic of cellulose structural modification I. The absorption band at 535 cm<sup>-1</sup> corresponded to the in-plane bending vibration of C-C=O [26].

These spectral details indicate that the pith and stem of the Jerusalem artichoke contains of a variety of functional groups that can interact with metal ions.

NaOH solution treatment of the samples JA9 and JA10 significantly reduces the amount of hemicellulose, IR spectra are characterized by a small shoulder at 1739 cm<sup>-1</sup> due to stretching vibrations of the ester group in hemicellulose [27] (Fig. 5, Table 4). The small peak in both samples at 1506 cm<sup>-1</sup>, responsible for the vibrations of the aromatic rings of lignin, indicates that it was not completely removed when exposed to 18% NaOH solution (Fig. 5, Table 4).

The absence of a peak in the spectra of the JA5, JA6, and JA7 (Fig. 6) samples at 1739 cm<sup>-1</sup> responsible for stretching vibrations of the ester group in

hemicellulose is due to the complete removal of hemicellulose (Fig. 6, Table 4). A small peak at 1506 cm<sup>-1</sup> in the spectrum of the JA5 sample and a small shoulder in the spectrum of the JA7 sample, responsible for the vibrations of the lignin aromatic rings, indicate that lignin was not completely removed (Fig. 6, Table 4).

A small peak at 1506 cm<sup>-1</sup> in the spectrum of the JA5 sample and a small shoulder in the spectrum of the JA7 sample, responsible for the vibrations of the lignin aromatic rings, indicate that lignin was not completely removed (Fig. 6b, Table 4). The absence of the peak at 1506 cm<sup>-1</sup> in the spectrum of JA5 sample (Fig. 6), which is responsible for fluctuations in aromatic rings of lignin, indicated that it is completely removed (Table 4).

The ratio of the absorption bands intensities at 3400 cm<sup>-1</sup> and 1320 cm<sup>-1</sup> was used to characterize the intensity of the hydrogen bond. The ratio of peak intensities at 1429 cm<sup>-1</sup> and 897 cm<sup>-1</sup> was used as the empirical crystallinity index (LOI). As a total crystalline index the ratio of peak intensities at 1372 cm<sup>-1</sup> and 2900 cm<sup>-1</sup> (TCI) was used [28].

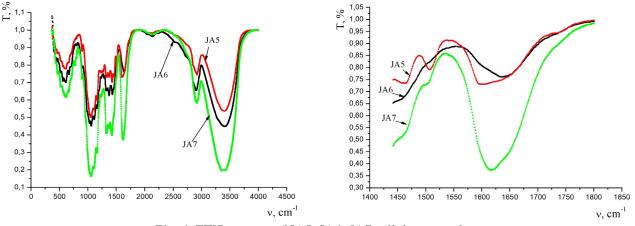


Fig. 6. FTIR spectra of JA5, JA6, JA7 cellulose samples.

The TCI is proportional to the crystallinity degree of cellulose and the LOI is correlated to the cellulose order strengthening. The highest values of TCI and LOI were found for JA4 and JA7 samples, indicating the highest crystallinity index and the cellulose order strengthening (Table 3). The lowest values of TCI and LOI were found for sample JA1. The highest of the hydrogen bond intensity is characteristic for the JA5 sample, while its crystallinity index is lower than for the JA7 sample. It should be noted that the cellulose samples have higher TCI and LOI values than the starting materials. There is a good correlation of crystallinity index determined by IR spectroscopy and X-ray diffraction (Table 3).

#### 3.3. Thermal analysis

To confirm changes in the internal structure of Jerusalem artichoke stem as a result of mechanical and subsequent chemical treatment, a thermogravimetric study was carried out.

Thermal stability of cellulose is an important factor for bioenergetics [29], when used as a filler for reinforcing polymeric nanocomposites [28], synthesis of carbon nanocomposites [30] and activated carbons [31]. The cortex and pith of Jerusalem artichoke stem, as well as samples of the obtained cellulose and its modifications, were studied by using thermal analysis. The thermal degradation (TG) and derivative thermal degradation (DTG) curves showing the mass loss during the pyrolysis process for WPF are presented in Fig. 7 for JA1, JA4 and JA10 samples.

These thermograms demonstrate a typical thermal decomposition behavior of lignocellulosic biomass in an inert medium.

The DTG curve of JA1 sample is characterized by the presence of an initial peak at 71.6 °C between 51.1 and 97.5 °C due to the evaporation of water (loss in weight 6.71%). After this peak, the shape of the DTG curve indicates two stages of decomposition (Fig. 7a). At 198.4 °C the first stage of the decomposition of the stem cortex begins, which ends at 226.7 °C and has a peak at 217.2 °C. The loss of weight (13.86%) is mainly due to depolymerization of hemicelluloses, pectin and glycosidic bonds of cellulose. The second stage of thermal destruction of JA1 sample is in the temperature range 302.8-359.8 °C and has a peak at 329.1 °C (weight loss 58.43%). At this stage, the degradation of cellulose takes place. Among the three components (hemicellulose, cellulose, lignin), lignin was the most difficult one to decompose. Its decomposition happened slowly under the whole temperature range from ambient to 950 °C.

In the temperature range of 360–950 °C, the DTG curve exhibits a low rate of mass loss due to the degradation of lignin.

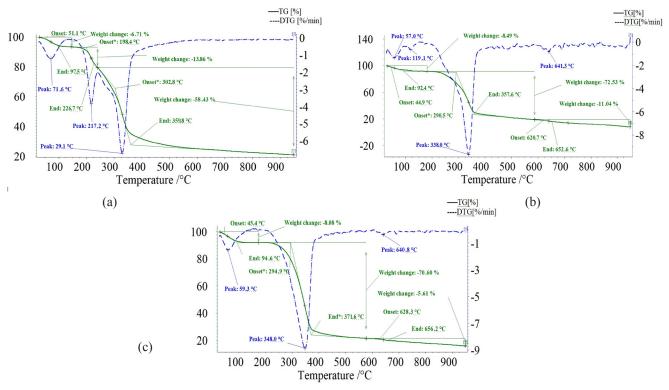


Fig. 7. TG and DTG curves: JA1 (a), JA4 (b), JA10 (c).

Eurasian Chemico-Technological Journal 21 (2019) 173-182

Unlike JA1 sample, the DTG curve of sample JA4 is characterized by the presence of two water removal peaks at 57.0 and 119.1 °C (Fig. 7b) (weight loss 8.49%), which is apparently due to differences in morphology and microstructure (Figs. 1–3). In the interval 290.5–357.6 °C the first stage of stem pith thermal degradation takes place, which is characterized by a peak at 338.0 °C. The weight loss of 72.53% is mainly due to the degradation of cellulose and small amounts of hemicellulose and pectin. The small peak at 641.3 °C (weight loss 11.04%) can be attributed to the degradation of the charred high-molecular lignin.

After processing the Jerusalem artichoke stems with an aqueous solution of 18% NaOH (JA10), washing and drying, the cellulose content in the sample increased, the content of hemicelluloses and lignin decreased (Table 4), and the initial segment of the DTG curve of the sample has only one peak at 59.3 °C between 45.3 and 94.5 °C (weight loss is 8.08%) (Fig. 7c). The second peak at 348.0 °C corresponds to the thermal destruction of cellulose in the temperature range 294.9–371.6 °C, where the weight loss is 70.60%, which is less than in the original sample (JA4). The small third peak at 640.8 °C with a weight loss of 5.61% is possibly associated with the degradation of the charred high-molecular lignin, which is not removed by NaOH (Table 4).

Thermal analysis showed that the cortex and pith of Jerusalem artichoke stem remain stable until 198.4 and 290.5 °C, respectively, which is similar to the thermal stability of other natural fibers, such as jute (205 °C), kenaf (219 °C), hemp (250 °C) [25], bagasse (222 °C) and bamboo (214 °C) [32]. During the thermal decomposition of the cortex and pith (25–950 °C), the carbon residue is 21.0% and 7.94%, respectively.

### 4. Conclusions

The cortex and pith of the Jerusalem artichoke stem have a different structure. The cortex is a dense woody material. The pith of the stem is similar to honeycomb with closed cells. It has a small density and a large specific surface, being an effective sorbent of metal ions. The cortex contains 54.1% of cellulose, the pith – 67.7%. All cellulose samples obtained from cortex and pith are characterized by a high content of  $\alpha$ -cellulose and have small ash content. Chemical treatment of the pith and mechanochemical treatment of the Jerusalem artichoke cortex contribute to an increase in the content of  $\alpha$ -cellulose and its CrI, while reducing the content of non-cellulose components, ash and degree of polymerization. The high quality of the Jerusalem artichoke cellulose makes a possibility of using it for the production of microcrystalline cellulose, nanocellulose, cellulose ethers.

According to IR spectroscopy, the highest values of TCI and LOI have the samples of the initial pith and the cellulose isolated from it. Lignin is nearly absent in the cellulose obtained chemically from the pith and in the cellulose obtained mechanochemically from the cortex of the Jerusalem artichoke stem.

Thermal analyses have shown that the thermal stability of the studied samples depends mainly on the structure and content of non-cellulose components. The results of thermogravimetric studies of the cortex, stem and cellulose of Jerusalem artichoke can be compared with the data for known annual plants (hemp, jute, kenaf, sisal, bagasse, bamboo), which allows using Jerusalem artichoke stems as filler or for reinforcing of polymeric nanocomposites and as the precursors for activated carbons production.

### Acknowledgments

The study was supported by the Russian Science Foundation (project №. 17-13-01240.)

### References

- S. Wang, J. Chen, G. Yang, K. Chen, R. Yang, J. Zeng, *BioResources* 12 (2017) 1031–1040. DOI: 10.15376/biores.12.1.1031-1040
- [2]. R. Maurya, C. Paliwal, T. Ghosh, I. Pancha, K. Chokshi, M. Mitra, A. Ghosh, S. Mishra, *Bioresource Technol.* 214 (2016) 787–796. DOI: 10.1016/j.biortech.2016.04.115
- [3]. A. Jain, R. Balasubramanian, M.P. Srinivasan, *Chem. Eng. J.* 283 (2016) 789–805. DOI: 10.1016/j.cej.2015.08.014
- [4]. M. Sevilla, A. Fuertes, R. Mokaya, *Energy Environ. Sci.* 4 (2011) 1400–1410. DOI: 10.1039/c0ee00347f
- [5]. C. Sanchez, L. Rozes, F. Ribot, C. Laberty-Robert, D. Grosso, C. Sassoye, C. Boissiere, L. Nicole, *CR Chim.* 13 (2010) 3–39. DOI: 10.1016/j.crci.2009.06.001
- [6]. J. Shen, Z. Song, X. Qian, Y. Ni, *Ind. Eng. Chem. Res.* 50 (2011) 661–666. DOI: 10.1021/ ie1021078
- [7]. R. Travaini, J. Martín-Juárez, A. Lorenzo-Hernando, S. Bolado-Rodríguez, *Bioresource*

*Technol.* 199 (2016) 2–12. DOI: 10.1016/j. biortech.2015.08.143

- [8]. M.A. Mehmood, G.Ye, H. Luo, C. Liu, S. Malik, I. Afzal, J. Xu, M.S. Ahmad, *Bioresource Technol.* 228 (2017) 18–24. DOI: 10.1016/j. biortech.2016.12.096
- [9]. W. Li, J. Zhang, C. Yu, Q. Li, Dong, F. G. Wang, G.D. Gu, Z.Y. Guo, *Carbohyd. Polym.* 121 (2015) 315–319. DOI: 10.1016/j. carbpol.2014.12.055
- [10]. X.H. Long, H.B. Shao, L. Liu, L.P. Liu, Z.P. Liu, *Renew. Sust. Energ. Rev.* 54 (2016) 1382–1388. DOI: 10.1016/j.rser.2015.10.063
- [11]. J. Matías, J.M. Encinar, J. González, J.F. González, *Energy Sustain. Dev.* 25 (2015) 34–39. DOI: 10.1016/j.esd.2014.12.009
- [12]. V. Fiore, A. Valenza, G. Di Bella, *Compos. Sci. Technol.* 71 (2011) 1138–1144. DOI: 10.1016/j. compscitech.2011.04.003
- [13]. T211 om-2. Ash in Wood, Pulp, Paper and Paperboard: Combustion at 525 °C. Approved by the Standard Specific Interest Group for this Test Method TAPPI.
- [14]. F. Xu, Y.C. Shi, D. Wang, *Carbohyd. Polym.* 94 (2013) 904–917. DOI: 10.1016/j. carbpol.2013.02.008
- [15]. W. Zhang, Z.L. Yi, J.F. Huang, F.C. Li, B. Hao, M. Li, S.F. Hong, Y.Z. Lv, W. Sun, A. Ragauskas, F. Hu, J.H. Peng, L.C. Peng, *Bioresource Technol.* 130 (2013) 30–37. DOI: 10.1016/j.biortech.2012.12.029
- [16]. USP, 2002. 25/NF 20 (United States Pharmacopeia 25/National Formulary 20). 2010.
  456 Washington, DC, p. 701.
- [17]. T.V Prokopov, N.D. Delchev, D.S. Taneva, Journal of Food and Packaging Science, Technique and Technologies. 3 (2014) 64–68.
- [18]. M. Li, J. Wang, Y. Yang, G. Xie, *Bioresource Technol.* 208 (2016) 31–41. DOI: 10.1016/j. biortech.2016.02.053

- [19]. L. Zhou, J. Pang, A. Wang, T. Zhang, 2013.
   *Chinese J. Catal.* 34 (2013) 2041–2046. DOI: 10.1016/S1872-2067(12)60686-X
- [20]. M. Ioelovich, E. Morag, *BioRes.* 6 (2011) 2818– 2835.
- [21]. M. Ioelovich, BioRes. 3 (2008) 1403-1418.
- [22]. H. Yang, R. Yan, H. Chen, D.H. Lee, C. Zheng, *Fuel* 86 (2007) 1781–1788. DOI: 0.1016/j. fuel.2006.12.013
- [23]. W. Liu, K. Mohanty, L.T. Drzal, P. Askel, M. Misra, J. Mater. Sci. 39 (2004) 1051–1054. DOI: 10.1023/B:JMSC.0000012942.83614.75
- [24]. L. Zhu, H. Qi, M. Lv, Y. Kong, Y. Yu, X. Xu, *Bioresource Technol.* 124 (2012) 455–459. DOI: 10.1016/j.biortech.2012.08.059
- [25]. I.M. De Rosa, J.M. Kenny, D. Puglia, C. Santulli,
   F. Sarasini, *Compos. Sci. Technol.* 70 (2010) 116–122. DOI: 10.1016/j.compscitech.2009.09.013
- [26]. C. Li, J. Lin, G. Zhao, J. Zhang, *BioResources* 11 (2016) 1596–1608. DOI: 10.15376/ biores.11.1.1596-1608
- [27]. A. Alawar, A.M. Hamed, K. Al-Kaabi, *Compos. Part B-Eng.* 40 (2009) 601–606. DOI: 10.1016/j. compositesb.2009.04.018
- [28]. M. Poletto, H.L.O. Júnior, A.J. Zattera, Materials 7 (2014) 6105–6119. DOI: 10.3390/ma7096105
- [29]. A. Mabuda, N. Mamphweli, E. Meyer, *Renew. Sust. Energ. Rev.* 53 (2016) 1656–1664. DOI: 10.1016/j.rser.2015.07.038
- [30]. J. Hoekstra, A.M. Beale, F. Soulimani, M. Versluijs-Helder, Dirk van de Kleut, J.M. Koelewijn, J.W. Geus, L.W. Jenneskens, *Carbon* 107 (2016) 248–260. DOI: 10.1016/j. carbon.2016.05.065
- [31]. M. Sevilla, A.B. Fuertes, *Energy Environ.* Sci. 4 (2011) 1765–1771. DOI: 1 10.1039/ C0EE00784F
- [32]. F. Yao, Q. Wu, Y. Lei, W. Guo, Y. Xu, *Polym. Degrad. Stabil.* 93 (2008) 90–98. DOI: 10.1016/j. polymdegradstab.2007.10.012