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Application of NIR spectroscopy for cellulose determination in flax

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Abstract

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Comparative analysis of the cellulose measurement in fiber and sheave of different flax varieties was conducted by near-infrared spectroscopy and classical chemical method. Samples of fibers and sheaves have been obtained in field tests with plant growth regulators. Discovered difference between results of two compared methods was 0.1-0.8%. Applying of Apin-extra, Floravit and humic-fulvate complex (HFC) resulted in increase of cellulose content in the fiber and sheave at 5.0-11.8%. Floravit was the most effective biostimulator among others. It increased the cellulose content in fiber at 8.1-11.8% compared to control variant, 1.4-5.5% compared to variant with HFC and 0.5-3.3% compared to Apin-Extra. The method of near-infrared spectroscopy recommended as a reliable alternative to classical chemical methods for cellulose determination in flax fiber and sheave.

Keywords: fiber flax; plant growth regulators; cellulose determination; fiber; sheave; NIRS

Introduction

The importance of fiber flax *(Linum usitatissimum*) considerably increased for many industries due to flax became the only source of raw materials for the textile industry in Russia. Flax is a culture of great significance because of its unique properties such as hygienic and technical qualities of its fiber, special chemical and medical qualities of its oil. Flax raw materials can be used for needs of military, textile, construction and chemical industries and the other sectors of the economy. Application of flax for food and medical purposes is a rapidly developing trend in technology of feedstock production and its processing (Belopukhov et al., 2015, 2017).

Raise of flax productivity and its quality is still an actual problem. One way of solving this problem is using of highly effective plant growth regulators during the growing season. Nowadays environmentally friendly bioregulators produced from natural raw materials have become widespread. When used in combination with modern agricultural technologies, these plant growth regulators can improve productivity and quality of the obtained production (Sorlino, 2005; Shtabel and Popelyaeva, 2013).

In our study we use 3 bioregulators: Apin-extra (NEST M, Russia), Floravit (Gella-Farma, Russia) and humic-fulvate complex (Timiryazev State Agrarian University, Russia).

The active substance of Apin-extra is a 24-epibrassinolide (24-BRs). Epibrassinolide can be synthesized in the laboratory and under the industrial conditions. Its structure is identical to that of brassinosteroids present in plants in very small amounts, about 10^{-9}-10^{-6}%. Brassinosteroids are natural plant steroid hormones with growth stimulating effect. Application doses of 24-epibrassinolide are similar to natural brassinosteroids content in plants. 24-BRs promotes activation of phytohormones in plants – gibberellins, cytokinins, auxins.

Floravit contains natural composition of secondary metabolits (but not fungal cells themselves) produced by filamentous fungus *Fusarium sambusinum* Fuckel F-3051D, organic acids (0.1-0.2%), polysaccharides (0.04-0.05%) and sodium benzoate (0.1%) as a stabilizer. Beyond that Floravit also contains proteins with a low molecular weight to ensure synergy of the plants physiological systems. Bioregulator Floravit in low concentrations at 10^{-5}-10^{-11}% acts as a mediator between plant cells and causes auxin action (Dmitrevskaya et al., 2014).

HFC is a high-tech and (ballastless) humic fertilizer, it contains trace elements in chelated form. HFC has properties of a growth stimulant and antistress agent. HFC contains humic acids (HS) (80%), fulvic acids (20%) and mineral elements (Belopukhov et al., 2013; Belopukhov et al., 2016). Nitrogen-containing components of HS demonstrate hormone-like activity with an effect similar to auxin and polyamines. Humic substances act as auxin mimetics which promote root growth through the stimulation of H^+-ATPase activity (Zandonadi, 2013).

Fiber quality is determined by physicochemical and mechanical properties of raw material: yield of long and short fiber, sheave content, flexibility, tensile strength, shrinkage after the heat treatment, air and vapor permeability, content of cellulose in fiber, lignin, pectin, hemicellulose, nitrogenous compounds and ash (Titok et al., 2010).

High quality flax fiber should contain more cellulose and less lignin and pectin. Flax fiber contains more than 30 chemical elements, which give this fiber a unique health and hygiene properties: high strength, ability to oppress a pathogenic flora, antimicrobial activity as well as high thermal conductivity, which is higher by 20-30% in comparison with cotton fiber, and high thermal resistance, this is why the linen garments are comfortable to wear under a wide range of ambient temperatures. Linen yarn and fabrics are widely used in medicine (Dmitrevskaya et al., 2010; Meshram and Palit, 2013).

Value of chemical elements of the ash should also be considered, because the composition and concentration of macro- and microelements in ash define biomedical properties of flax, linen fabrics and their products (Maity, 2014).

There are a lot of methods for cellulose content determination today. Traditionally, in laboratories for this parameter determination wet chemistry methods were used – slow and laborious procedures with low productivity.

Near-infrared spectroscopy (NIRS) used in our study provides a quick and fairly accurate analysis of the main quality parameters of agricultural products (grain, forage, fodder, fiber, feed raw materials, fish meal, etc.) and food (meat, semi-finished products, sausages, cheese, butter, cookies, etc.). The analysis in the near-infrared region requires minimal sample preparation and allows the evaluation of several parameters simultaneously (Barton et al., 2002).

Materials and Methods

In our study we used fibers obtained from four flax varieties: Antay, TOST 5, Voshod (Russia), Marilyn (Netherlands). Our field experiment was conducted in the Central Non-Black Zone of Russia, on the field of experimental station of Russian Timiryazev State Agrarian University, Moscow, in 2010-2013. Flax cultivation technology was spring harrowing, applying of complex fertilizer Kemira field-10 before sowing, sprinkling of herbicide Kortes and plant growth regulators. For Apin-extra the application rate was $2{,}5.10^{-4}$ gl^{-1}, for Floravit – 10^{-4} gl^{-1} and for HFC – 10^{-4} gl^{-1}. Every studied growth regulator was applied during the early phase of the growing season (Belopukhov, 2015).

Content of cellulose in a flax fiber and sheave was measured by near-infrared spectroscopy (NIRS) and wet chemical method for purposes of comparative analysis. The essence of wet chemistry method for determining of cellulose is a treatment of the test sample with a 17.5% sodium hydroxide solution, rinsing the sample with 9.5% sodium hydroxide and distilled water with drying to constant weight. This approach refers to the classic cellulose gravimetric analytical method, which takes a significant amount of time.

In our study we used a near-infrared analyzer SpectraStar XL 2500XL-R. This instrument has a Top Window design (the optical window is positioned at the top of the device). This feature allows to analyze not only solid, but also liquid and pasty objects. Using large cuvettes in conjunction mode of sample rotation makes it possible to analyze coarse milled samples and samples without preceding milling. The device works in a spectral range of 680-2500 nm, which allows performing an analysis of complex components. Monochromator is pre-dispersive and based on a diffraction grating; detector is Indium Gallium Arsenide (InGaAs), thermo-stabilized. Mode spectrum measurement: reflection or transmission. Light source: halogen precalibrated lamp. The interval between the points of the spectrum is 1 nm. The number of points of the spectrum: 1820. Bandwidth (resolution): 10 +/- 0.3 nm. Photometric range: 3.0 AU. Scan time: less than 0.8 seconds per scan point. Analysis time: 10-60 seconds. Wavelength accuracy: less than 0.1 nm. This instrument equipped with touch-screen, its proprietary software InfoStartm works under Windows 7 operating system.

Nowadays NIRS is officially accepted by many countries for analysis of grain, oil, feed, cotton, wool, synthetic fiber and other agricultural products. During the last twenty years a large amount of guidelines for NIRS quality control of agricultural production were developed (Faughey and Sharma, 2000; Workman et al., 2007).

Results and Discussions

According to the previous researches, high quality flax fiber should contain 70-80% of cellulose, low content of pectin, hemicellulose, nitrogen-containing substances and ash. Therefore, these properties can serve as objective quantitative characteristics of fiber's quality (Grishina and Belopukhov, 2013; Grishina et al., 2013).

Comparative analysis of cellulose determination in fiber by NIRS and classical wet chemistry method is shown in Table 1 and Fig. 1. For the purposes of comparison, we added to the scheme of experiment a variant without applying of growth regulators (control).

Data of analyzes demonstrates a high content of cellulose fiber, 70-86% for all combinations of factors. All plant growth regulators applied during the growing season had a positive impact on increasing cellulose content for all studied varieties. Apin-extra (the active ingredient of which is 24-epibrassinolide) can be a benchmark for other bioregulators and controls. Apin-extra increased cellulose content in the fiber to 6.6-10% compared to control and 0.5-3.7% compared to HFC. The most effective growth regulator was Floravit, it increased content of cellulose to 8.1-11.8% com-

Table 1
Determination of cellulose in flax fiber, % abs. dry substance

Methods	Variety of fiber flax	Plant growth regulators			
		Control	HFC	Floravit	Apin-extra
Chemical analysis	Antaeus	70.3 ± 2.7	76.6 ± 3.1	81.7 ± 3.4	79.8 ± 3.1
NIRS		70.1 ± 2.9	76.4 ± 3.0	81.9 ± 3.4	80.1 ± 3.1
Chemical analysis	Voshod	70.8 ± 2.7	77.9 ± 2.9	80.5 ± 3.0	79.4 ± 2.9
NIRS		70.7 ± 2.7	78.3 ± 2.9	80.1 ± 3.1	79.1 ± 3.0
Chemical analysis	Marilyn	76.9 ± 2.8	83.6 ± 3.3	85.8 ± 3.4	86.1 ± 3.4
NIRS		77.2 ± 2.8	83.9 ± 3.4	85.3 ± 3.5	86.3 ± 3.1
Chemical analysis	TOST 5	73.5 ± 2. 8	79.9 ± 2.9	83.3 ± 3.4	80.1 ± 3.0
NIRS		73.7 ± 2.8	80.2 ± 3.0	83.8 ± 3.5	80.5 ± 2.9

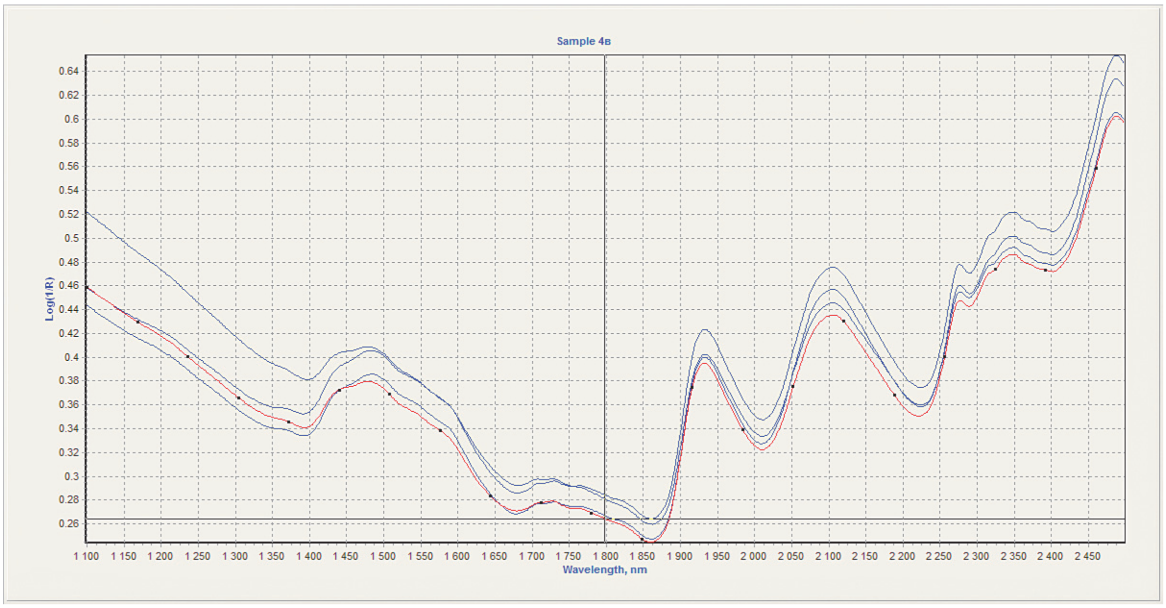

Fig. 1. NIR spectrum of flax fiber in TOST 5 variety (from up to down Floravit, Apin-extra, HFC, Control)

Table 2
Determination of cellulose in the flax sheave, % abs. dry substance

Methods	Variety of fiber flax	Plant growth regulators			
		Control	HFC	Floravit	Apin-extra
Chemical analysis	Antaeus	55.2 ±1.9	60.1 ± 2.1	60.1 ± 2.1	54.2 ± 1.8
NIRS		55.8 ± 1.8	60.4 ± 2.0	59.3 ± 2.0	54.8 ±1.9
Chemical analysis	Voshod	53.2 ± 1.8	55.4 ± 1.9	59.1 ± 2.1	54.1 ± 1.7
NIRS		53.5 ± 1.8	56.1 ± 1.9	59.2 ± 2.0	53.9 ± 1.7
Chemical analysis	Marilyn	60.7 ± 2.2	62.3 ± 2.3	64.5 ± 2.3	62.3 ± 2.1
NIRS		61.1 ± 2.2	63.1 ± 2.0	64.1 ± 2.4	62.7 ± 2.0
Chemical analysis	TOST 5	57.9 ± 2.0	58.9 ± 2.1	60.3 ± 2.2	59.5 ± 2.0
NIRS		57.1 ± 2.0	59.4 ± 2.1	59.8 ± 2.1	60.1 ± 2.2

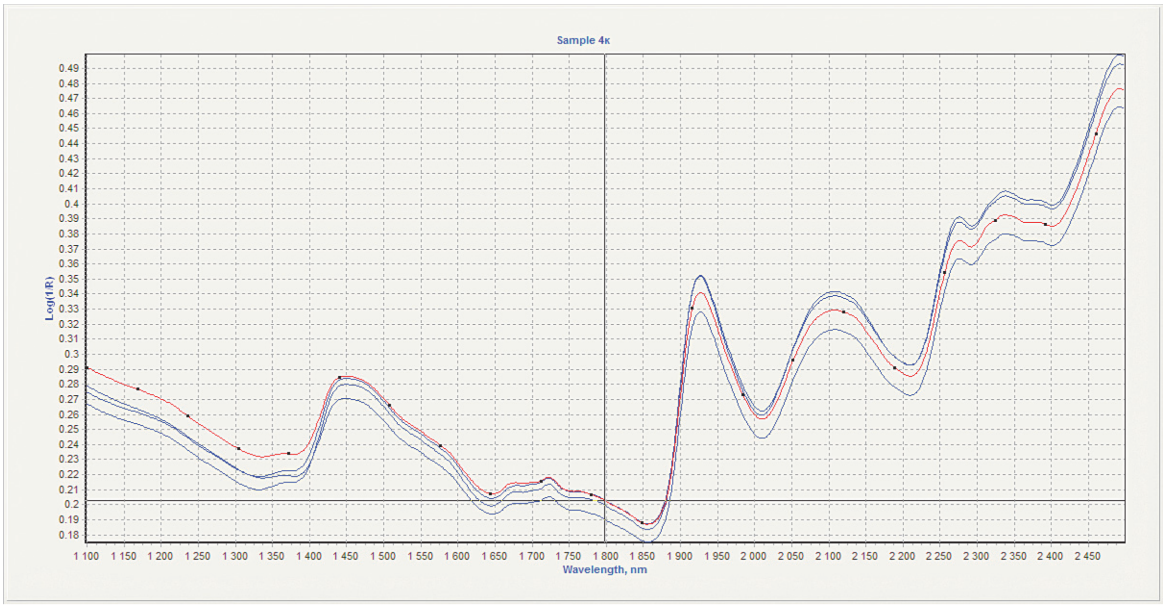

Fig. 2. NIR spectrum of flax sheave in TOST 5 variety (from up to down Floravit, Apin-extra, HFC, Control)

pared to control, 1.4-5.5% compared to HFC and 0.5-3.3% compared to Apin-extra.

Comparative analysis of the cellulose percentage measured by wet chemistry method and by NIRS showed that the difference between results of these two methods is 0.1-0.5%. We claim this as a reason why near-infrared spectroscopy can be a good alternative to time-consuming classical analytical method.

The application of residues after sheave stripping and rippling is a matter of a special interest in different industries. Sheave can be used for manufacturing of a wide variety of building insulation, wallpaper, animal bedding, etc. Sheave is composed of 50-70% cellulose, 30% of lignin, pectin, nitrogen and waxy substances, ash components. Sheave component composition determines a further use of this material. The results of cellulose determination in the flax sheave are shown in Table 2 and Fig. 2. According to this data the flax sheave of all variants contained 55-65% cellulose, difference of the results of two analytical methods is 0.1-0.8%.

The plant growth regulators had a similar effect on the cellulose content in a sheave, as well as in a flax fiber for all examined varieties. An increase of cellulose in sheave under a treatment with Apin-extra was 0.5-3.0%, HFC – 1.6-5.0%, Floravit – 2.4-6.0 % in comparison with control. The significant increase of cellulose content (0.5-6.0%) was discovered for a variant with Floravit application compared to Apin-extra and HFC. Thus the use of plant growth regulators Apin-extra, Floravit and HFC during the growing season increases content of cellulose in sheave up to 0.5-6.0%.

Conclusions

Comparative analysis of cellulose determination by chemical method and NIRS analysis showed that the difference between results of these methods was in the range of 0.1-0.8%. Therefore, the near-infrared spectroscopy is a good alternative to time-consuming classical analytical method.

The application of plant growth regulators Apin-extra, HFC amd Floravit during the growing season resulted in an increase of cellulose content for all studied varieties. The most effective biostimulator was Floravit, as it increased the content of cellulose in fiber at 8.1-11.8% compared to control, 1.4-5.5% compared to HFC and 0.5-3.3% compared to Apin-extra.

The increase of sheave cellulose content observed during treatment with Apin-extra was 0.5-3.0%, HFC – 1.6-5.0%, Floravit – 2.4-6.0%, compared to control. A significant increase (0.5-6.0%) of the cellulose content was demonstrated by Floravit compared both to Apin-extra and HFC.

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